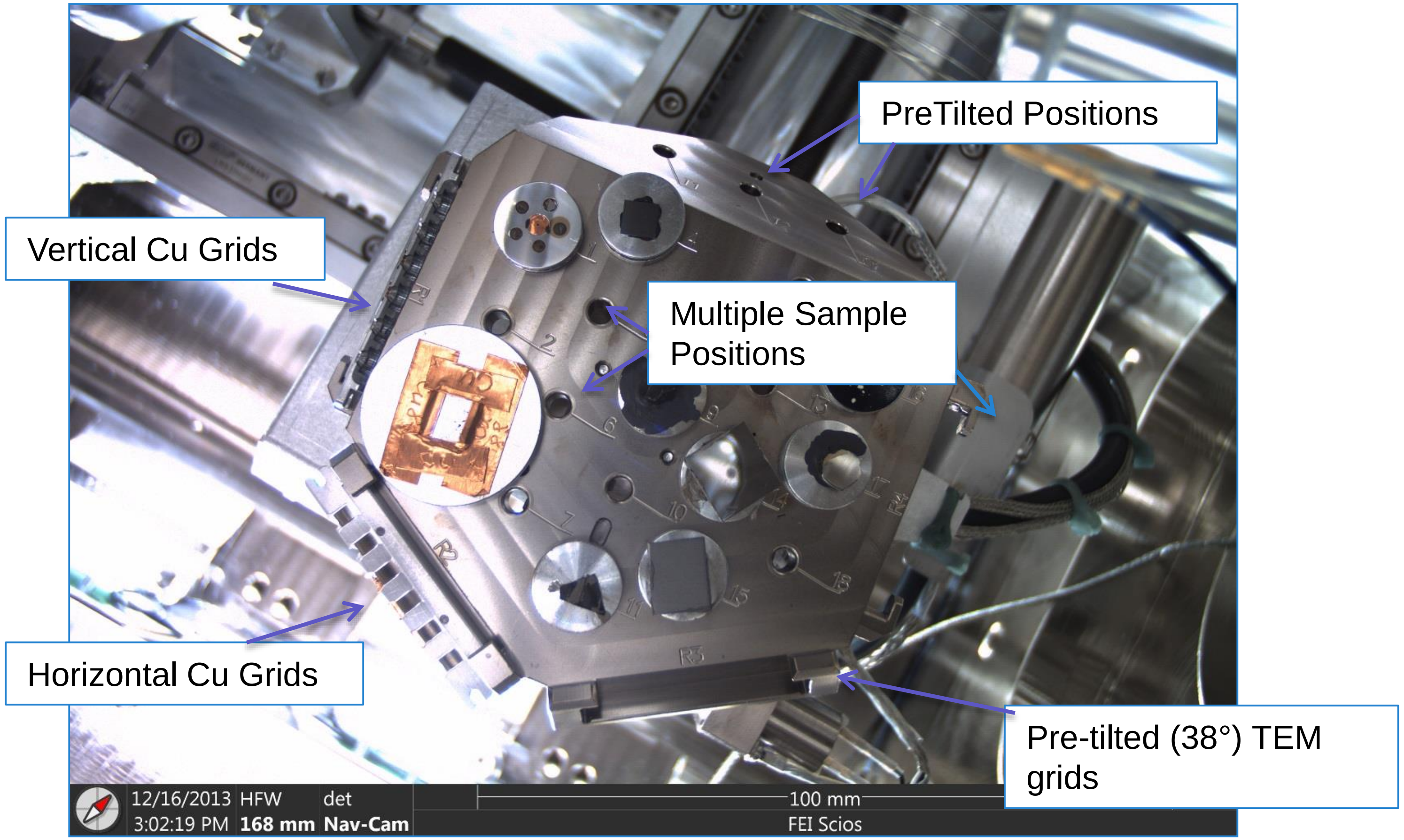
Scios
(S)TEM sample prep

Module 7

The world leader in serving science

Large sample holder and stage, Easy Navigation



- Multiple positions accommodate plenty of samples
- Pre-tilted sample positions for Bulk Samples
- Large Stage movement;
  110mm x 110mm X,Y, 90° tilt
- Vertical, Horizontal and Pre-tilted TEM grid holder positions
- Easy and Fast sample Location with Nav-Cam
- Bulk samples and pre-tilted TEM grids can be loaded simultaneously. Allowing S(TEM) sample preparation and STEM end pointing and imaging without breaking the vacuum

Step by step (S)TEM sample preparation

1. Find area of interest
2. Set coincidence point using e-beam + Z-adjustment
   (start with: zero beam shift and uncheck Z-Y link)
   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 ≥ 1.6nA ->Start

   NOTE: Retract GIS
3. Tilt to 52 and continue with ion beam Pt deposition in quad 2.
   Overlay rectangle on E-beam dep. (increase X) Min. thickness of Pt layer=1.5um.
4. Rough cut/bulk milling using Regular Cross Section + Si multipass new 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 size and material.
   Put pattern not too close to the Pt layer (for high BC Δ>2um) start to mill front side first.
5. Than repeat from back side; rotate pattern 180deg (advanced tab)(or change milling direction to top to bottom) NOTE: end point (where patterning stops) is always the 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. Apply to front and back side. Thickness of lamella 1-1.5um

Step by step (S)TEM sample preparation

7. Cut-out: Tilt to 7 degrees. Create cut-out; place a rectangle at the bottom (3/4 from top).
   Width of bottom cut min. 1um. Draw a rectangle at the EasyLift side (left side); to be cut completely.
   Place 3rd rectangle at right side and leave a part of the lamella attached to the bulk.
   Choose parallel milling (right top patterning page) z=2um, ≥1nA BC.
   RTM (or unpause ion beam) while patterning. Snapshot using e-beam for monitoring.
8. Tilt back to zero.
   NOTE: at this stage the FIB image can be scan rotated 180°
9. Lower ion BC to 50pA and go to EasyLift (=EL) tab

SEM image:
EL correction X + Y

FIB image:
EL correction X + Z

10. Insert EL needle to "park position"; EL visible in SEM image and at lower mag. in FIB image.
    Insert EL further down by using Z ↓ in FIB image.
    Insert Pt-GIS when EL is close to the lamella (few um)
11. Lower EL -> Z ↓ further until gentile touch down (left hand side of lamella) and retract EL 1 step
12. Weld the EL to the lamella using Pt-dep. <u>Wait for the vacuum to recover</u> and cut
    free lamella at other (right hand) side, by increasing ion BC to 1nA (fast).
    NOTE: before starting patterning make i-beam snapshot (or F9)
13. Lower i-beam current to 50pA (Check In SEM image if lamella is free by tilting to 35°)
14. Move out ↑ Easy Lift until lamella is removed from bulk. Bring EL to park position, than fully retract.
    Than retract the GIS from UI.

TEM prep using vertical TEM grids

Rowbar + grids loaded in position R1 or R4

Step by step (S)TEM sample preparation

15. Place halfmoon TEM (R1 or R4) grid under beam.
    Focus on grid link Z to WD set Z to 7mm. xT align grid horizontally. Set beam coincidence point.
16. Modify TEM grid to lamella width @ 0° tilt and by using high BC and CCS (X >2um smaller as lamella width, Y =18um, Z =2um)
17. Lower ion BC to 50pA and unpause image.
    Insert EL to park position. Insert EL further by using Z ↓ in FIB image.
    Insert Pt-GIS when EL is close to TEM grid.
    Lower EL -> Z further until (almost) touch down.
18. Weld lamella to grid using Pt-dep. Wait for the vacuum to recover and cut free EL from lamella by increasing ion BC to 1nA (fast).
19. Move out Easy Lift a few ums. Bring EL to park position than fully retract
    And last step retract GIS from UI.
20. For extra stability, the lamella can be attached by an extra Pt dep from the other side.
21. Or use the Cu redep method. Tilt to 52° and use 1nA + CCS; 4x3x1um. At right hand side rot pattern 90° start milling close to lamella, repeat at left hand side rot pattern 270°.
22. Lamella is now ready for further thinning.

Final thinning example

- 1 step: CCS @ 1nA, +/- 2° tilt
  CCS @ 54° bottom to top and CCS @ 50° bottom to top (or rotate pattern 180°)
- 2 step: CCS 300pA +/- 1.5° tilt (thickness about 300nm)
- 3 step: CCS 100pA +/- 1.2° tilt

For e-beam end-pointing use < 5kV

- finish with back-side milling; mill until uniform contrast

- use Pt layer as reference. Final thickness about 100nm.

- final step:

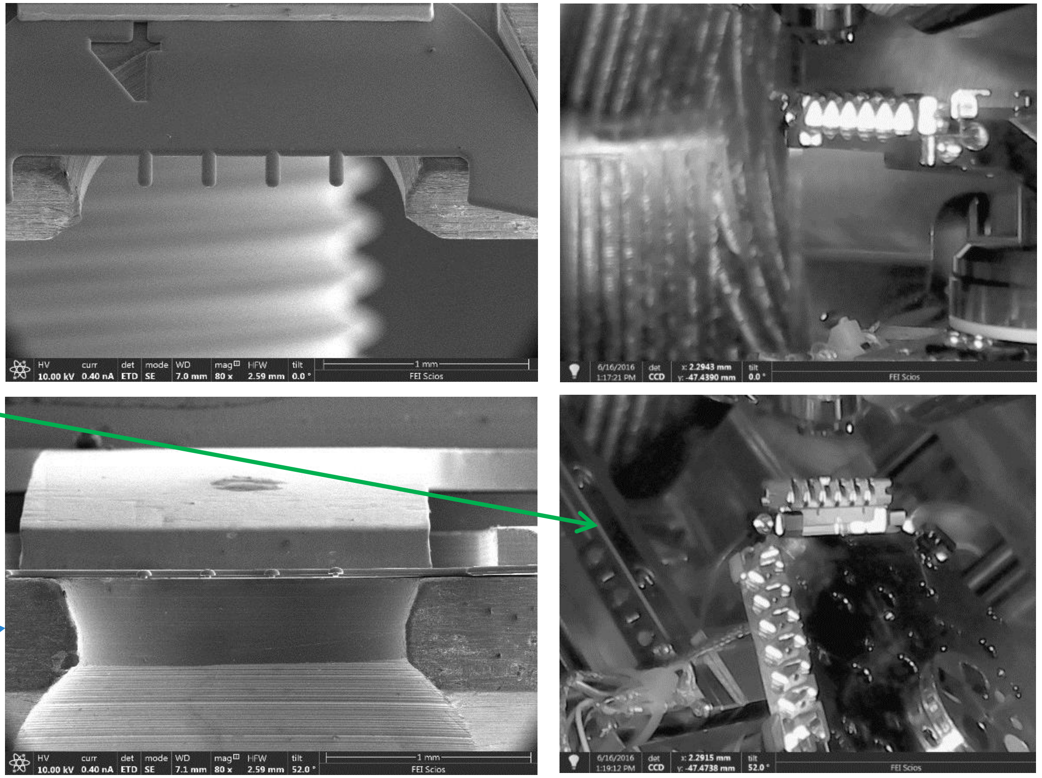
- Low kV clean is always needed for crystalline material: milling @ 30kV: amorphous layer (in Si) = 23nm
  - 5kV+48pA, +/- 5°; using rectangle Z=100nm (1min) @ 5kV: ~6nm
  - 2kV+27pA, +/- 7°; using rectangle Z=30nm (fast) @ 2kV: ~2nm

NOTE: Use live e-beam image for end-pointing
For stability, reduce width (X) by every cleaning step: e.g. start 15 –> 13 –> 11 um

TEM prep using 38° pre-tilted TEM grids

Rowbar + grids loaded in position R3

Step by step (S)TEM sample preparation continued



15. Place halfmoon TEM grid (R3) under SEM beam.
    Focus on grid link Z to WD set Z to 7mm.
    xT align grid to horizontally.
    Set beam coincidence point.
    NOTE: If needed rotate stage 180° (Compucentric) so the large part of the holder will be tilting downwards

16. Modify TEM grid to lamella width @ 0° tilt and by using high BC and CCS (X >2um smaller as lamella width, Y =18um, Z =2um)
17. Tilt stage to 52°

18. Lower ion BC to 50pA, lower magnification and unpause image.
    Insert EL to park position. Insert EL further by using Z ↓ in FIB image.
    Insert Pt-GIS when EL is close to TEM grid.
    Lower EL -> Z further until (almost) touch down.
19. Weld lamella to grid using Pt-dep. <u>Wait for the vacuum to recover</u> and cut free EL from lamella by increasing ion BC to 1nA (fast).

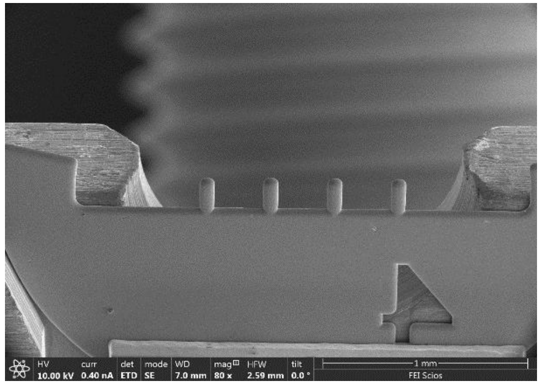
Step by step (S)TEM sample preparation continued

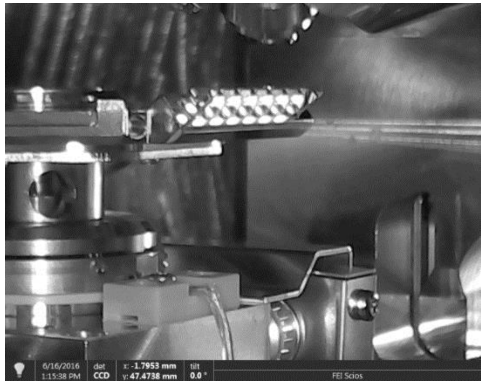
20. To increase the mounting stability of the thick lamella to the grid, some extra PT EBID can be applied;
tilt stage further to 65deg. Use e-beam to deposit extra Pt (2kV, >>1nA) to attach lamella to grid.

21. Retract Pt GIS and tilt back to 0°

22. Rotate stage to milling position = 180° (Compucentric)
NOTE: when a higher sample is loaded, lower stage first !!

23. Or use the Cu redep method. Use 1nA + CCS; 4x3x1um.
At right hand side rot pattern 90° start milling close to
lamella, repeat at left hand side rot pattern 270°.





23. Lamella is ready for further thinning.
NOTE: FIB is now looking perpendicular to the grid+lamella

Final thinning example

- 1 step: CCS @ 1nA, +/- 2° tilt
  CCS @ 54° bottom to top and CCS @ 50° bottom to top (or rotate pattern 180°)
- 2 step: CCS 300pA +/- 1.5° tilt (thickness about 300nm)
- 3 step: CCS 100pA +/- 1.2° tilt

For e-beam end-pointing use < 5kV

- finish with back-side milling; mill until uniform contrast

- use Pt layer as reference. Final thickness about 100nm.

- final step:

- Low kV clean is always needed for crystalline material: milling @ 30kV: amorphous layer (in Si) = 23nm
  - 5kV+48pA, +/- 5°; using rectangle Z=100nm (1min) @ 5kV: ~6nm
  - 2kV+27pA, +/- 7°; using rectangle Z=30nm (fast) @ 2kV: ~2nm

NOTE: Use live e-beam image for end-pointing
For stability, reduce width (X) by every cleaning step: e.g. start 15 –> 13 –> 11 um

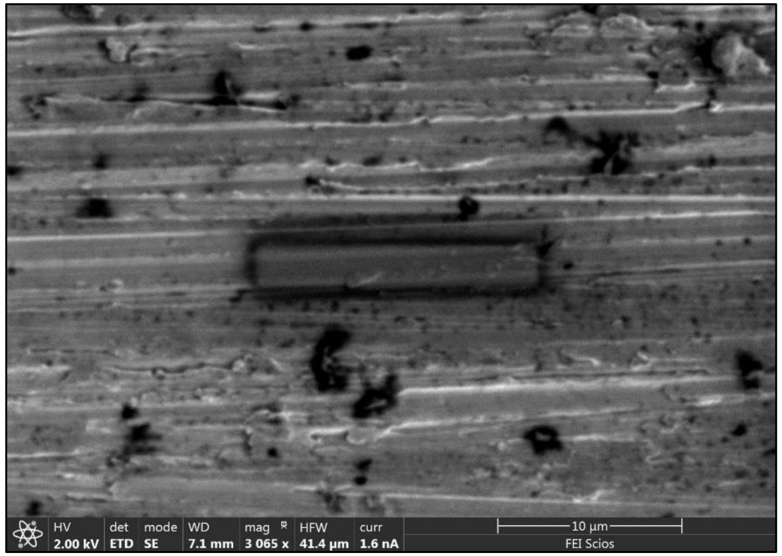
Tilt correction is function of BC and material

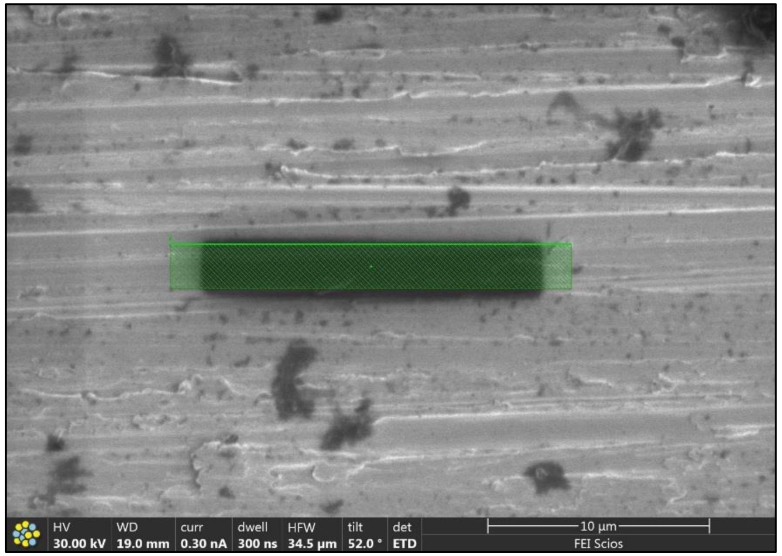
I-beam current	tilt Si	tilt GaAs	tilt Fe	polymers
50pA	1			0.3
100pA	1.2	0.8	2	0.5
300pA	1.6	1.2	2.5	0.8
500pA	1.8		2.8	1
1nA	2	1.8	3.5	1.2
3nA	3		4	

This table is in indication of the correction angles (extra tilt angles)
that need to be applied during the final milling steps:
When the sample is thinning too fast at the bottom, the extra tilt angle is too high
When the Pt is disappearing, the extra tilt angle is too low.

Protective layers of Platinum

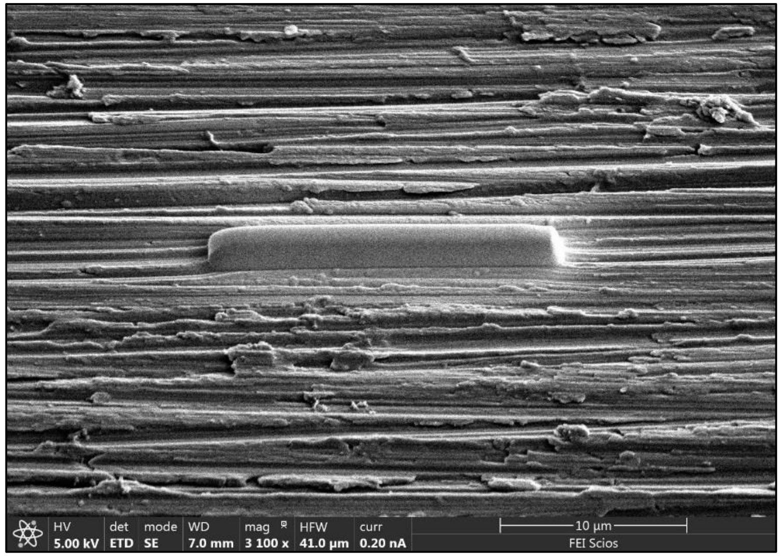
e-beam induced
(ebid) Pt deposition

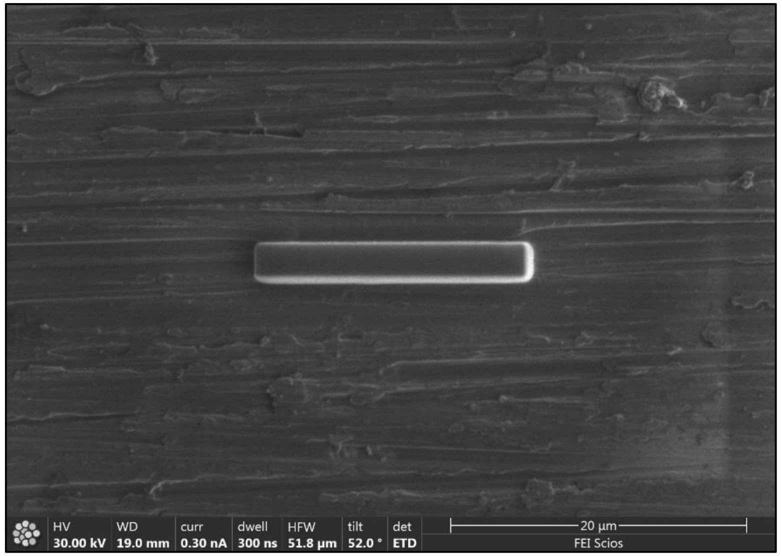




i-beam induced (ibid)
Pt deposition;
pattern placement over ebid-Pt

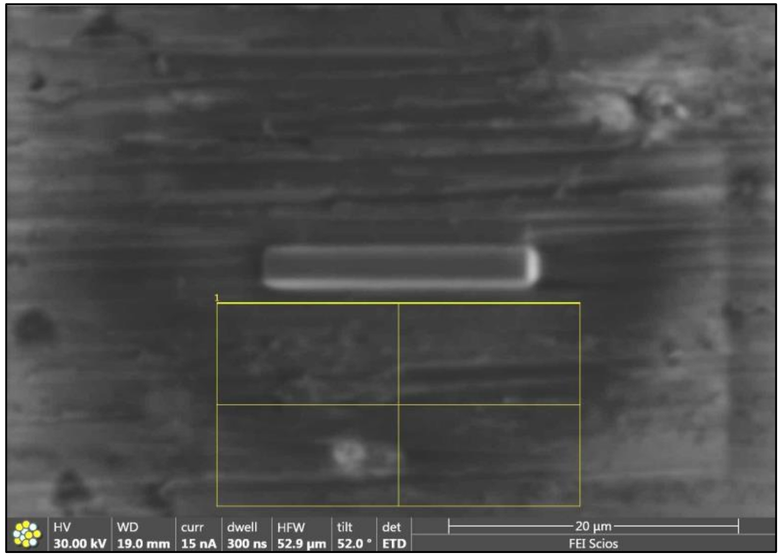
ibid-Pt deposition
On top of ebid-Pt



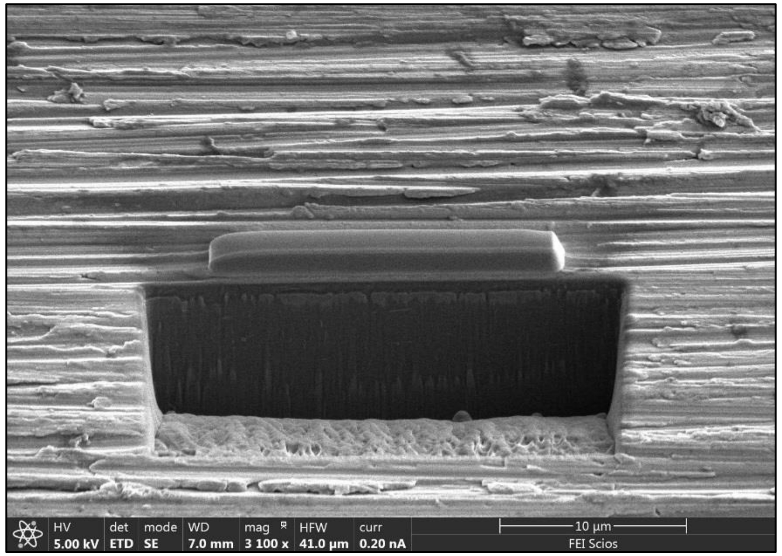


ibid-Pt deposition on
top of ebid-Pt

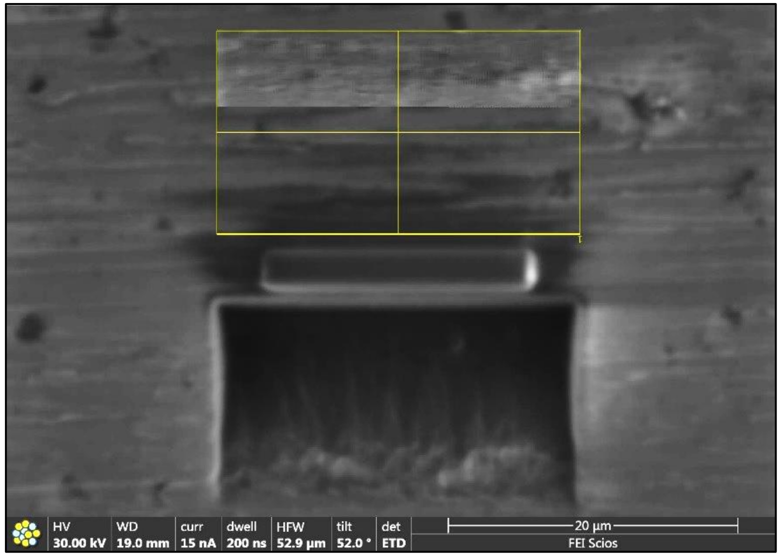
Regular Cross Sectioning/bulk milling; Si-multipass new



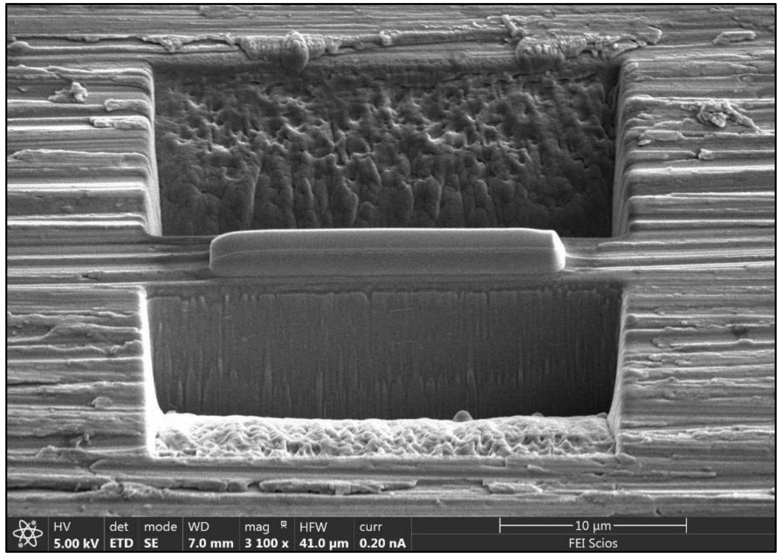
RCS pattern placed
at front side (BC=15nA)



RCS front side finished

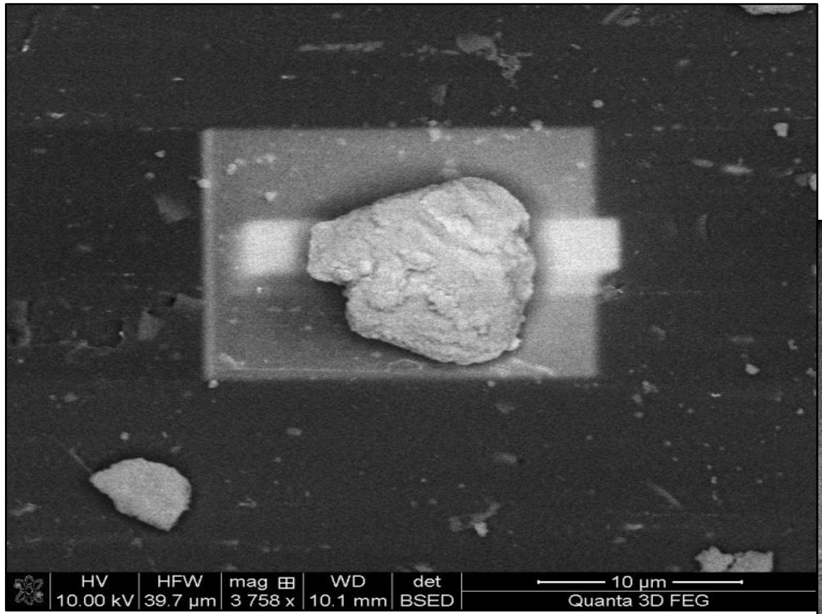


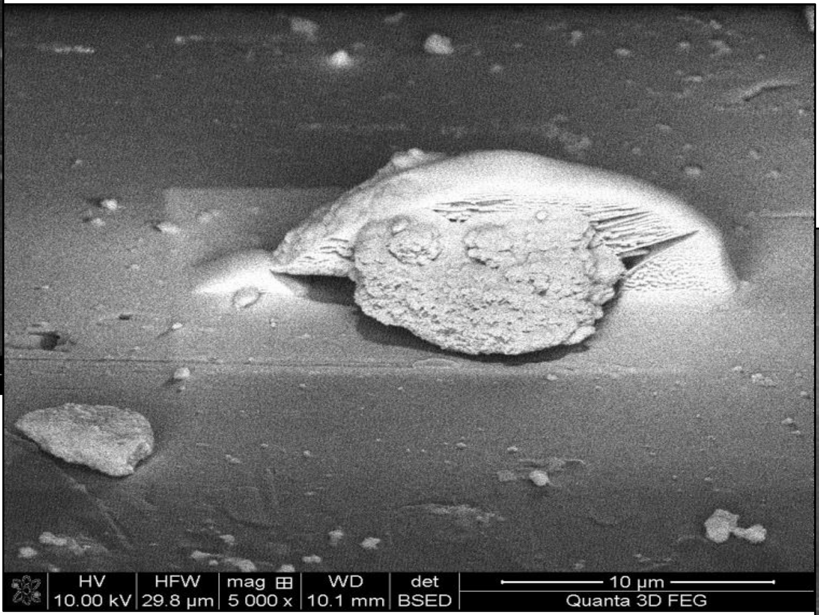
RCS pattern placed
at back side;
pattern rotated

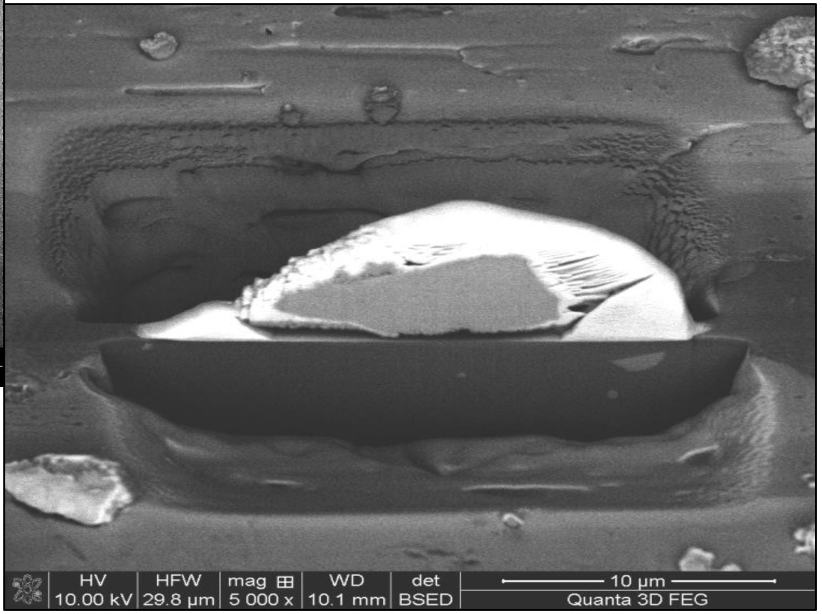


RCS back side finished

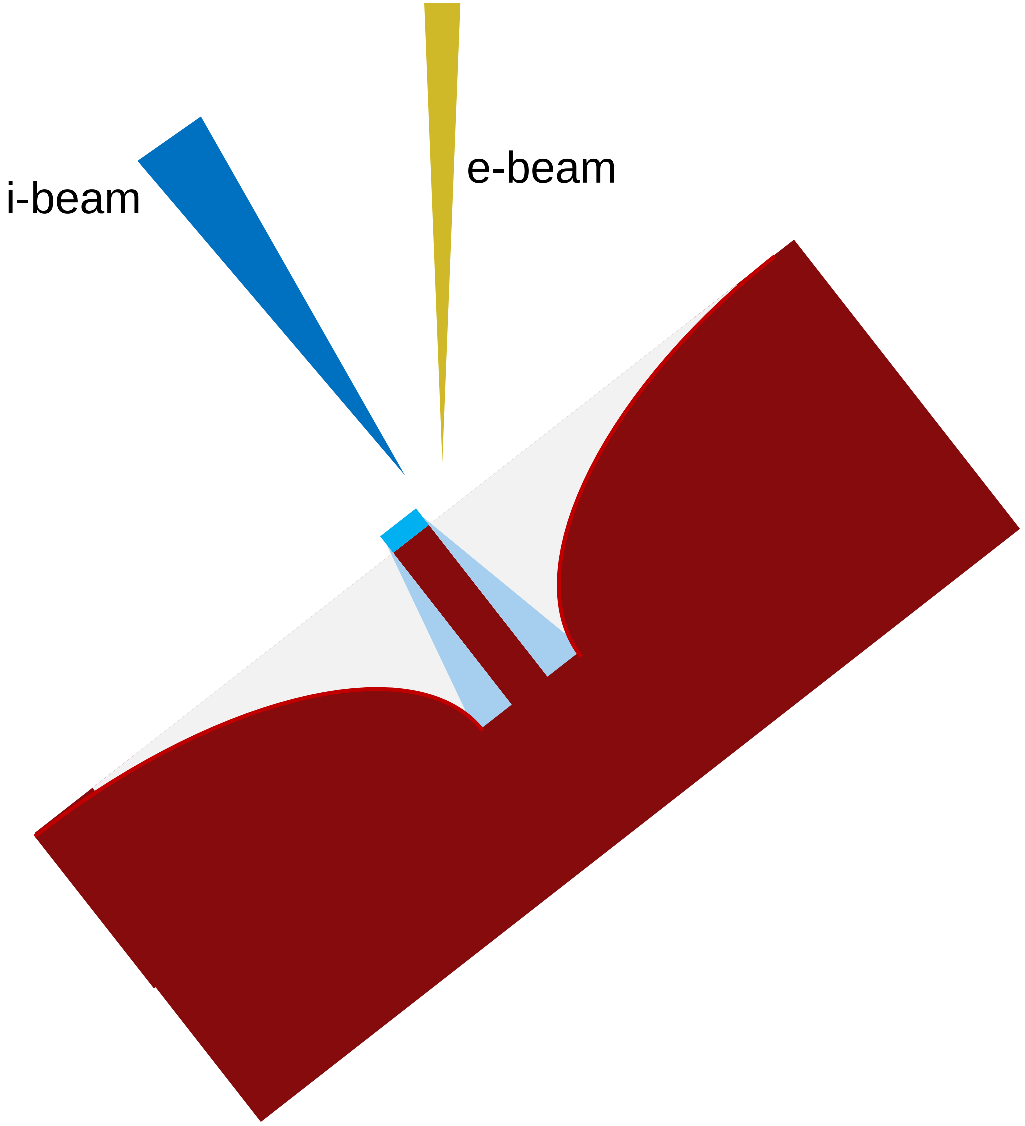
Site specific; e.g. powder sample







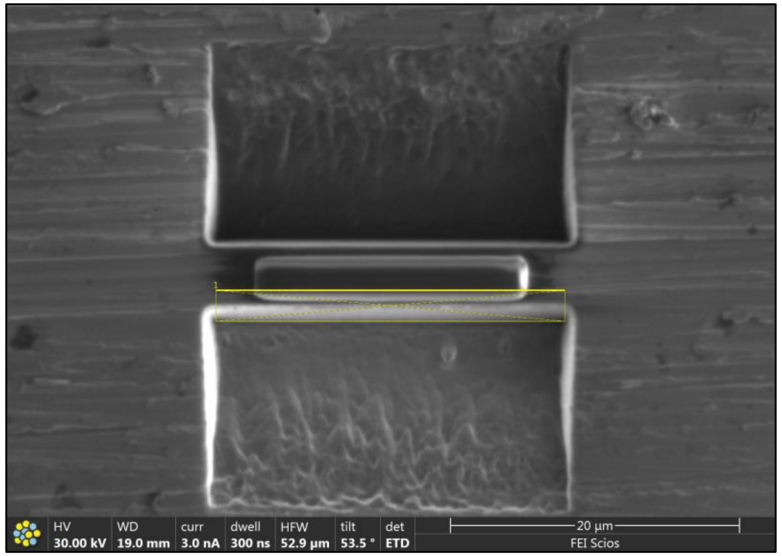
RCS: Si-multipass new (both sides)

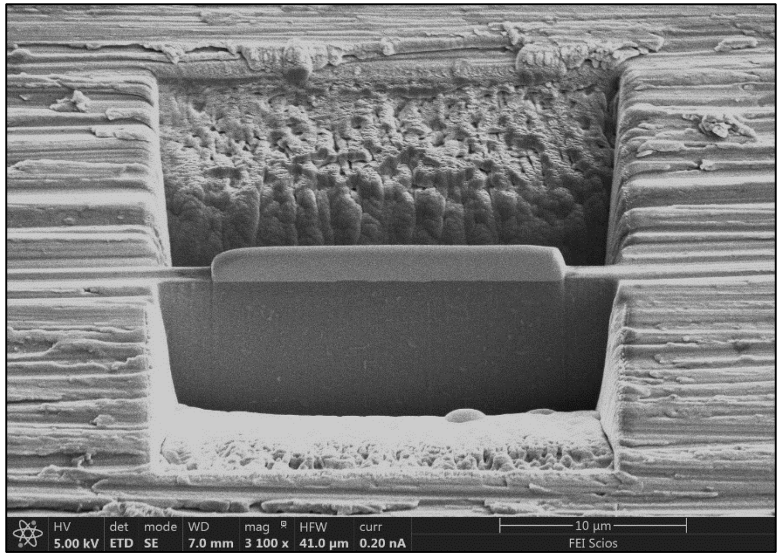


Side view @ 52°tilt; after removing material at both sides of the Pt layer using **Si-multipass new** with high i-beam current

Cleaning Cross Sectioning; Si-application file

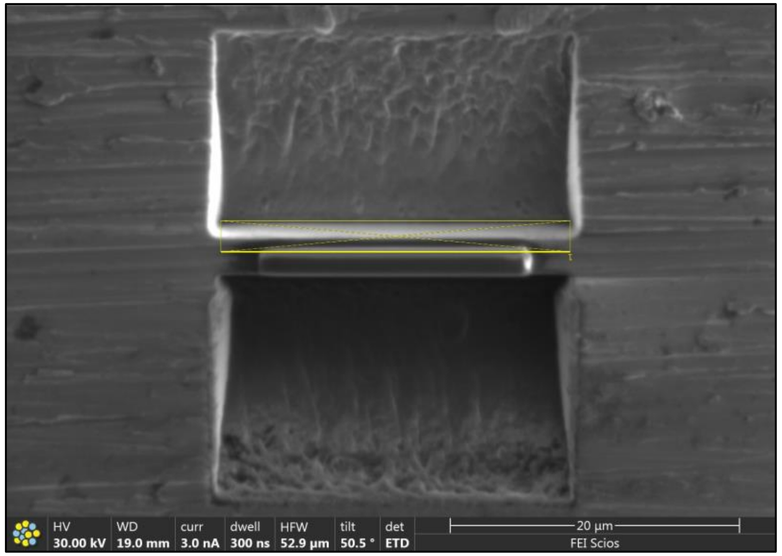
CCS pattern placed
at front side (+ extra tilt);
BC=3nA

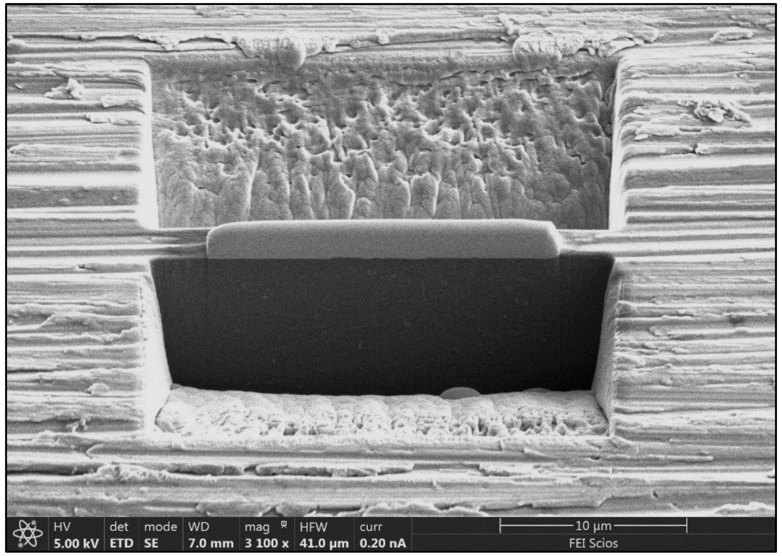




CCS front side finished

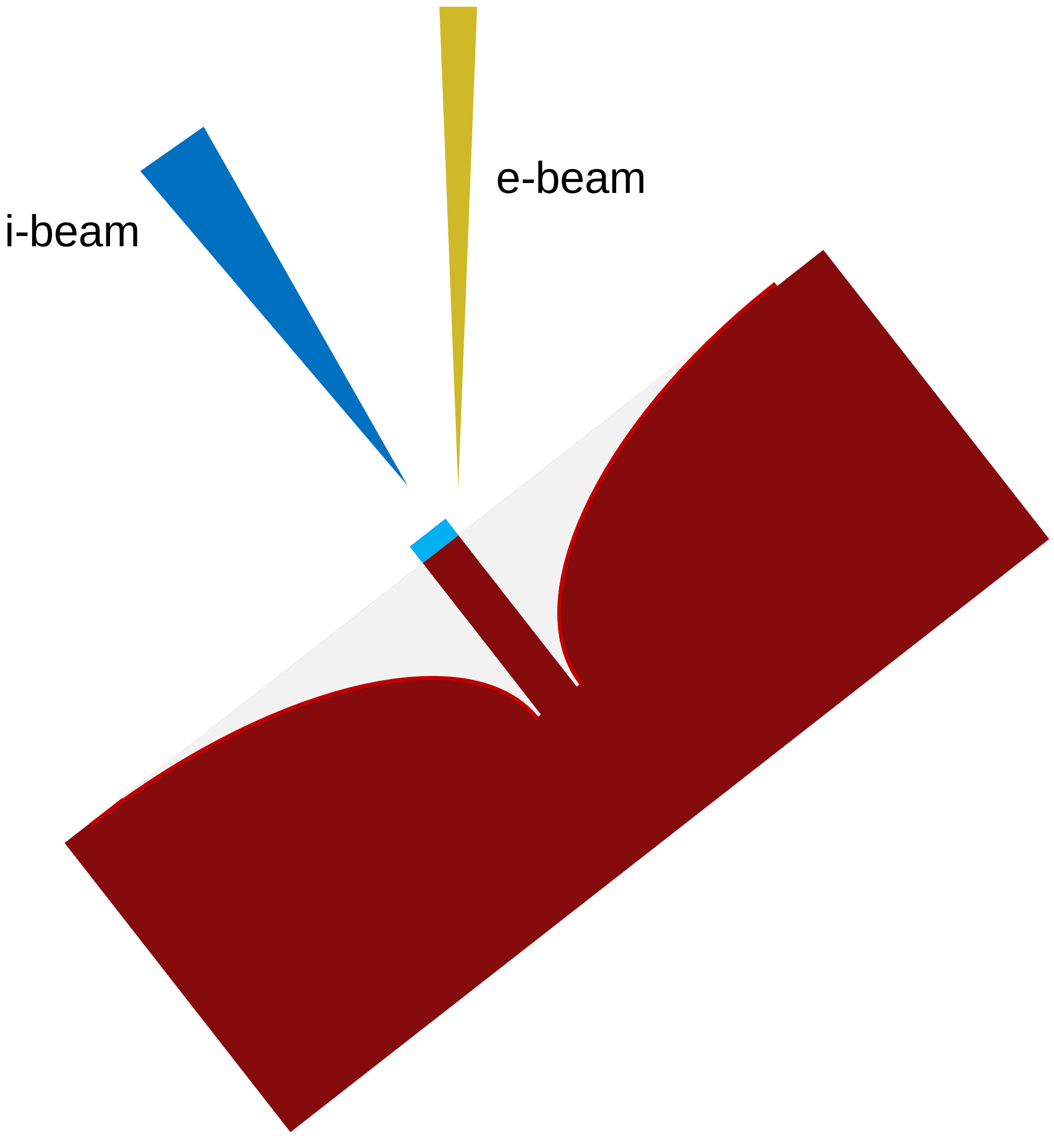
CCS pattern placed
at back side (- extra tilt);
pattern rotated





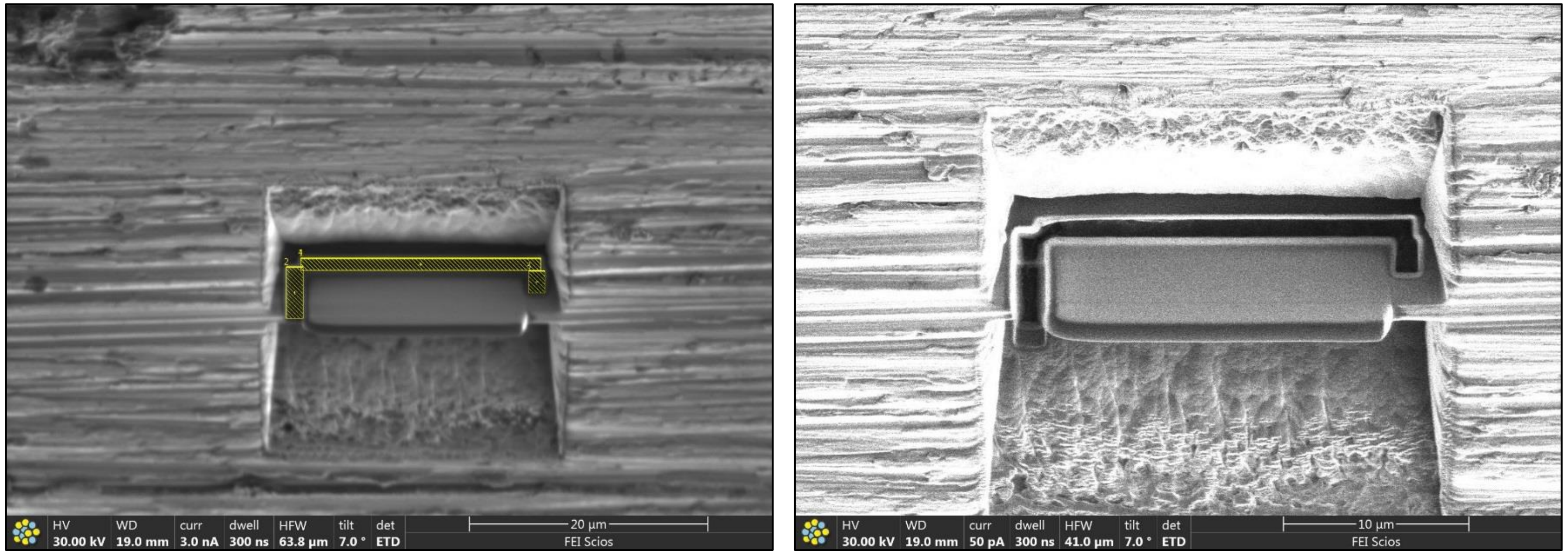
CCS back side finished

Cleaning Cross Section (both sides)



Side view @ 52°tilt; after removing material at both sides of the Pt layer using **CCS +** Si application file with medium i-beam current

Cutting free lamella



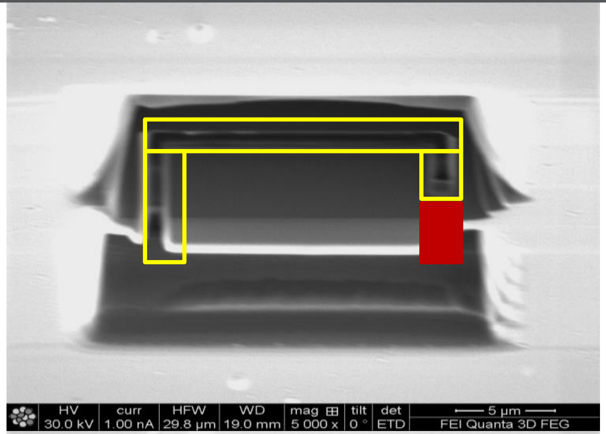
FIB images @ 7° tilt; before and after milling

-normal rectangles: 1-2-3 parallel milling (to prevent redepo)

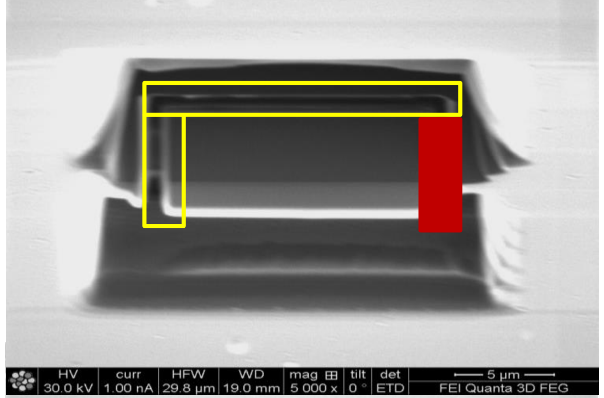
-width about 1um

-right-hand-side still attached

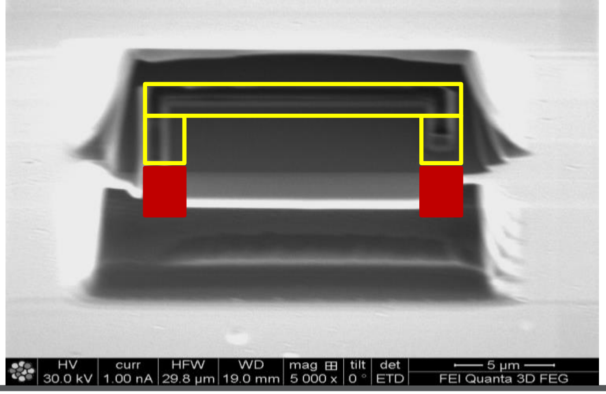
-placement of bottom rectangle not too deep (= at 2/3)



Normal sample size: Red is still attached



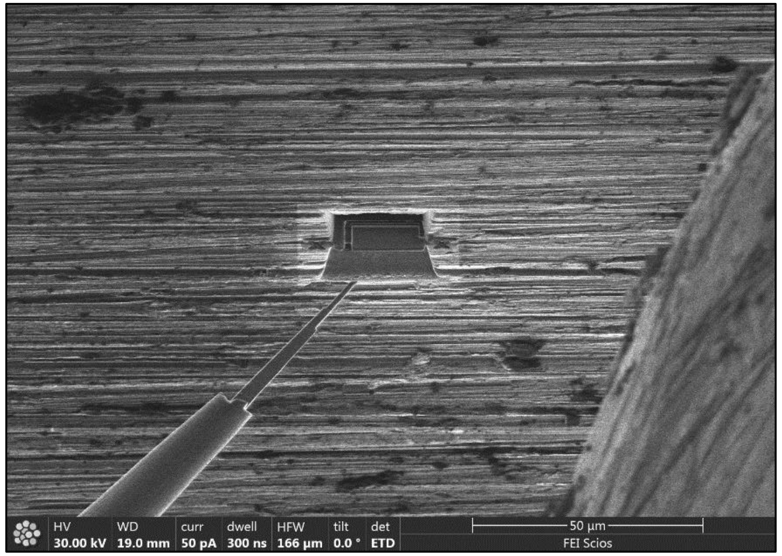
Small lamella

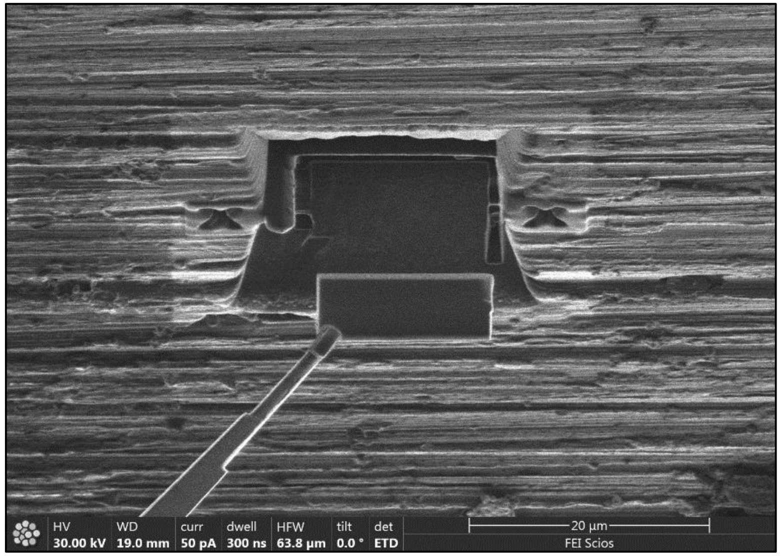


Stress in sample

In-situ thick lift out using EasyLift

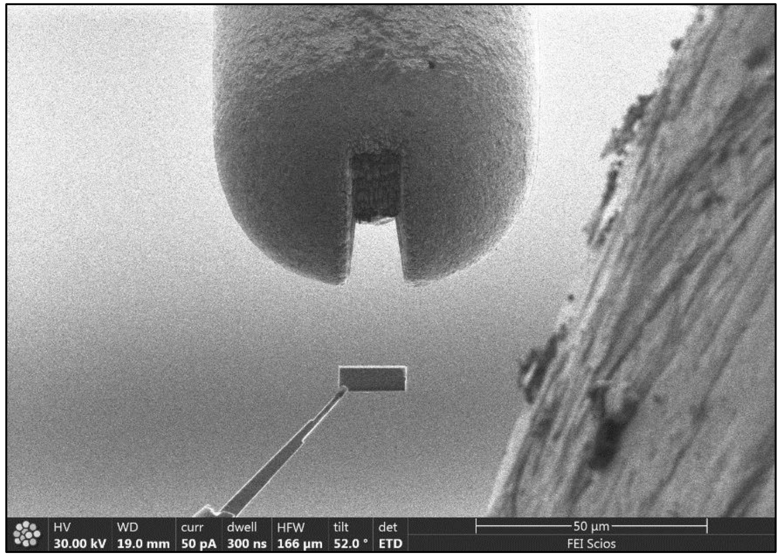
EasyLift + Pt GIS inserted;
sample @ 0° tilt

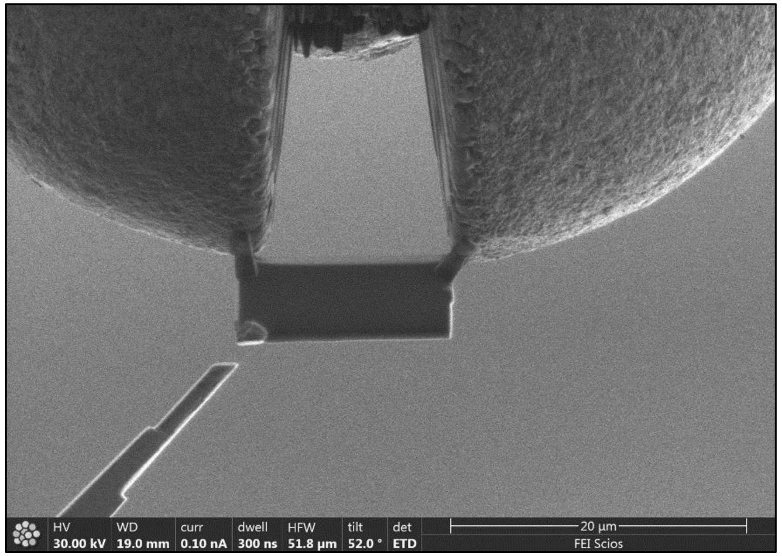




Thick lamella attached to EasyLift, cut free and removed from bulk

EasyLift + lamella approaching modified TEM grid

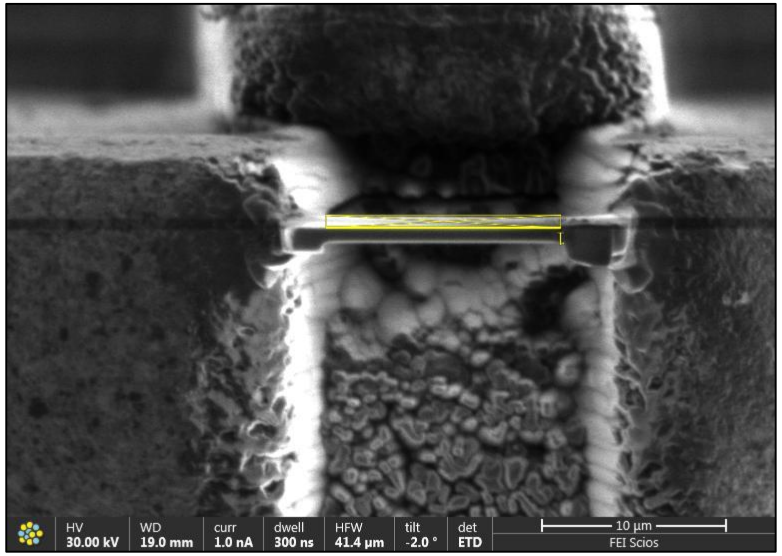




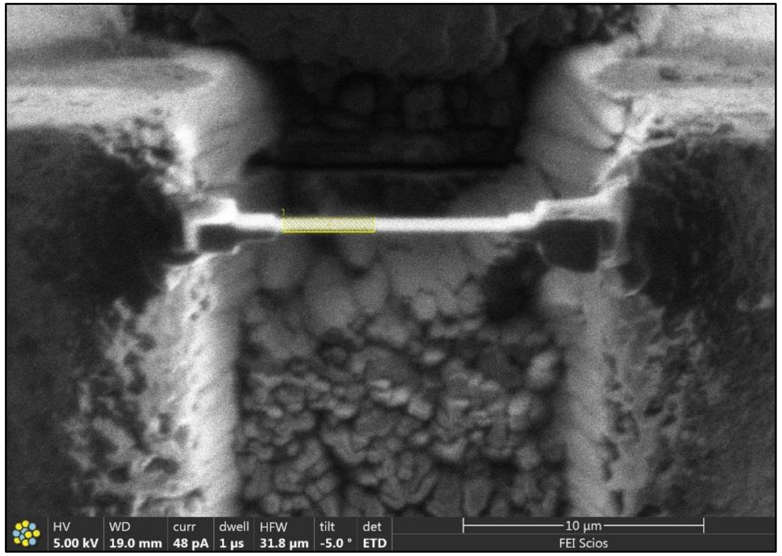
Lamella attached to TEM grid and EasyLift needle is cut off.
After removing the EL,
the lamella is ready for the final thinning step.

Thick lamella during thinning

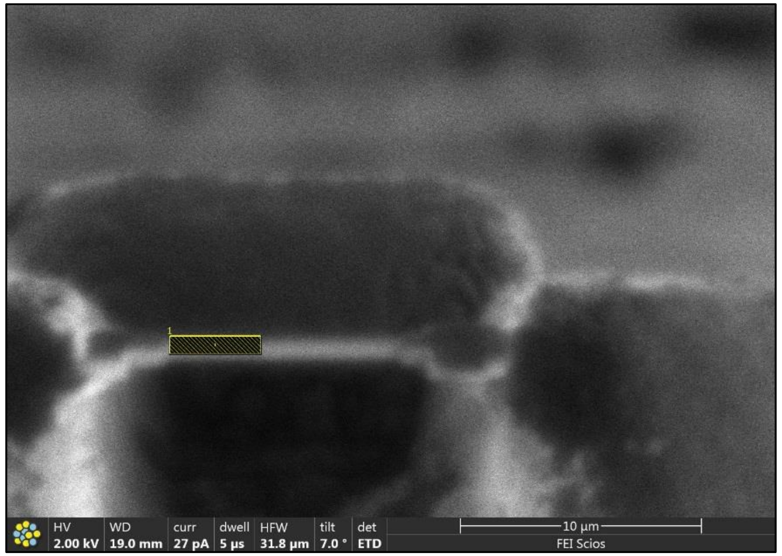
CCS; milling back side
(after front side)
1nA, -2° tilt



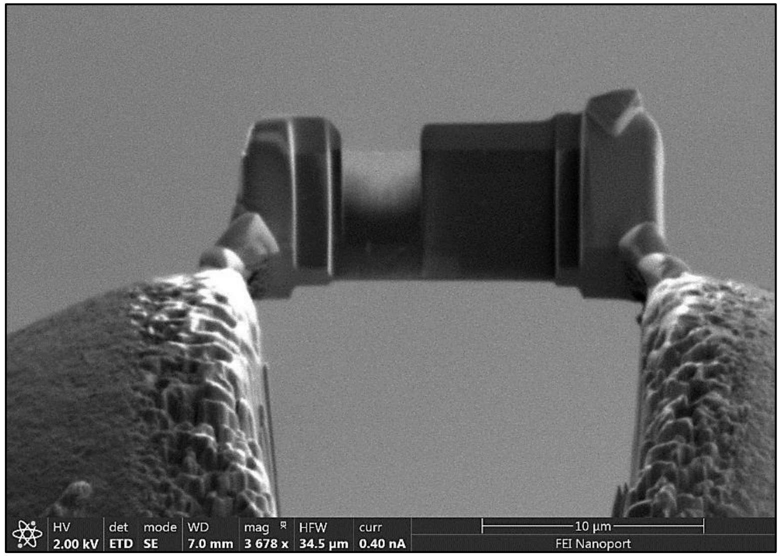
5kV (48pA) cleaning;
Image; back side; -5° tilt
Milling rectangle Z=100nm



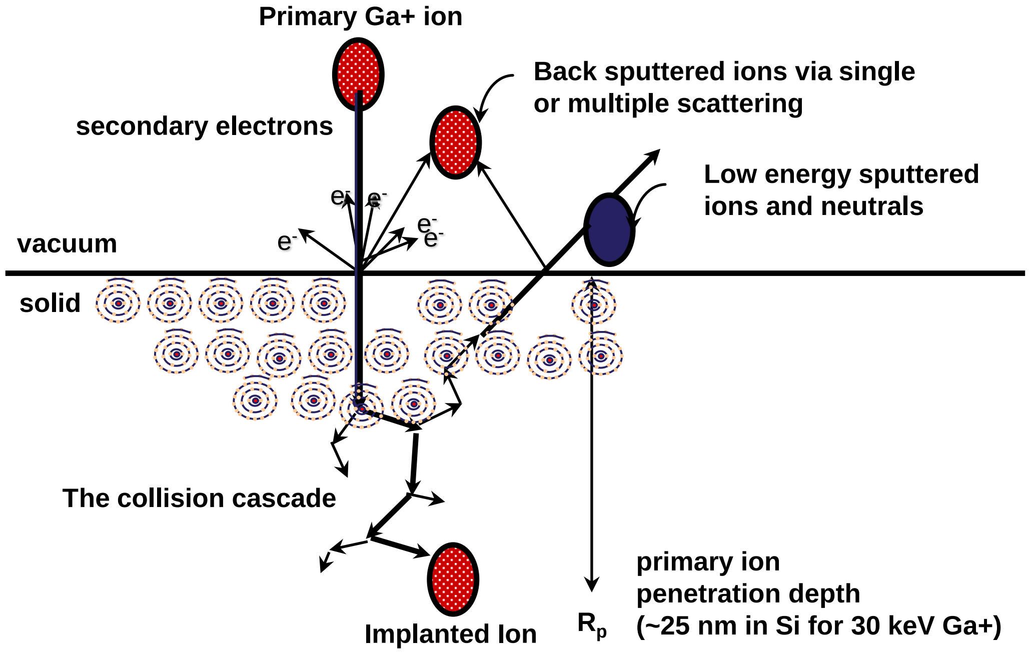
2kV (27pA) cleaning;
Image; front side; +7° tilt
Milling rectangle Z=20nm

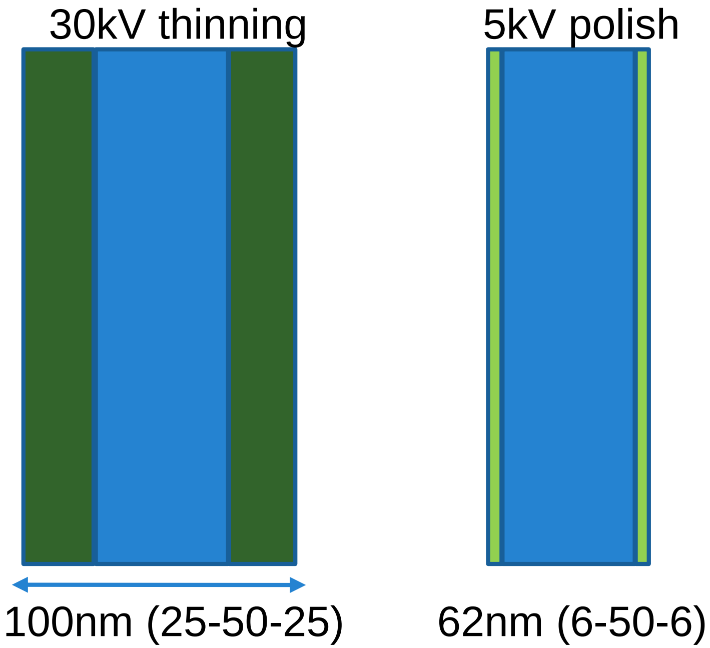


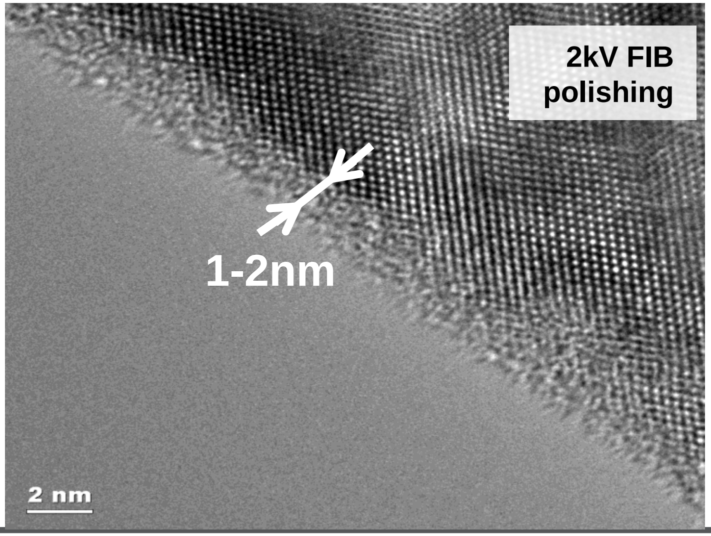
Sample ready for TEM



Ion-Solid Interactions

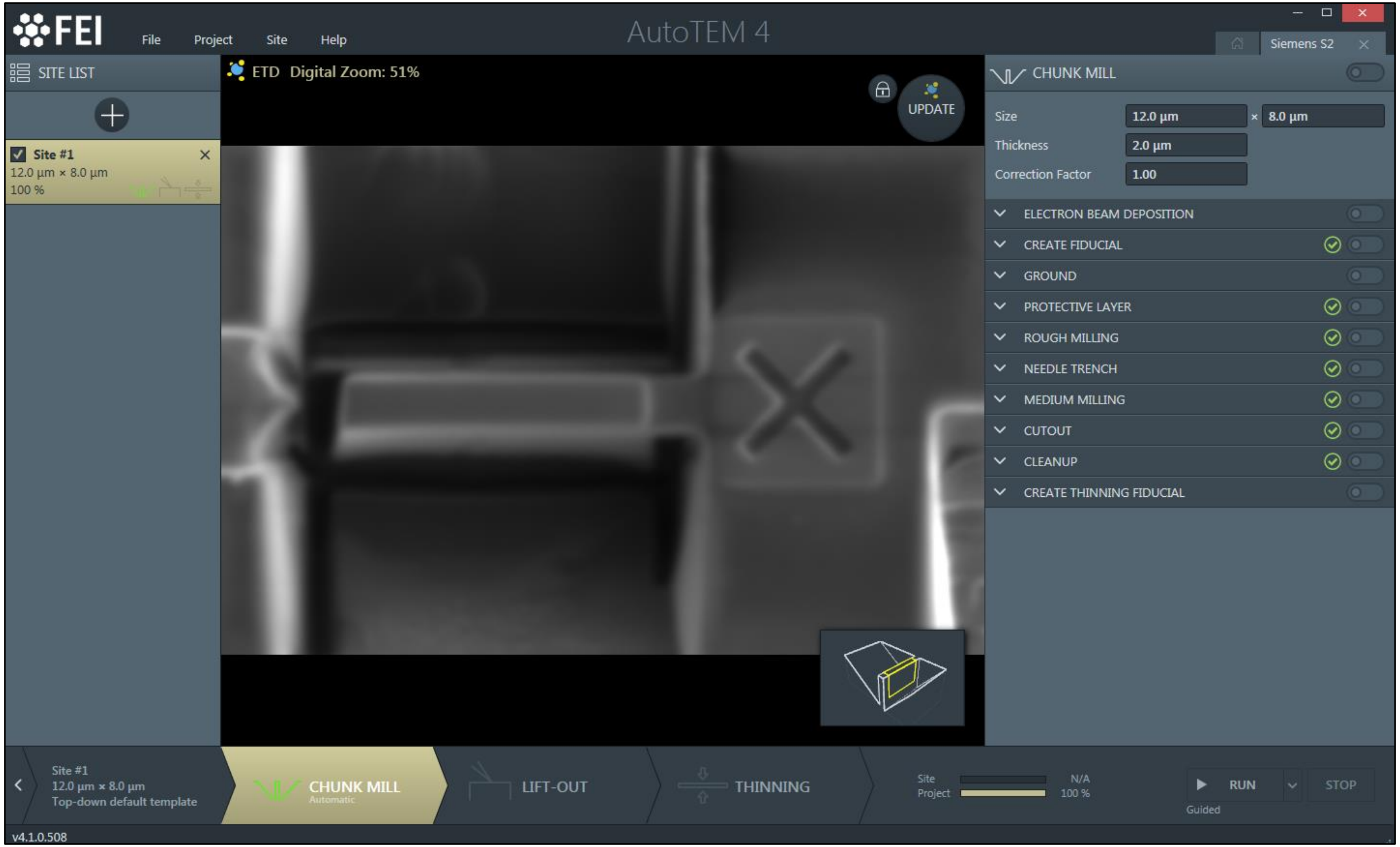






For good quality TEM lamellae the amorphous layers need to be minimized.

Automated thick lift-out sample (=chunk) preparation by: autoTEM4



ETD Digital Zoom: 51%

☑ Site #1
12.0 µm × 8.0 µm
100 %

CHUNK MILL

Size	12.0 µm × 8.0 µm
Thickness	2.0 µm
Correction Factor	1.00

- ELECTRON BEAM DEPOSITION
- CREATE FIDUCIAL ✓
- GROUND
- PROTECTIVE LAYER ✓
- ROUGH MILLING ✓
- NEEDLE TRENCH ✓
- MEDIUM MILLING ✓
- CUTOUT ✓
- CLEANUP ✓
- CREATE THINNING FIDUCIAL

Site #1
12.0 µm × 8.0 µm
Top-down default template

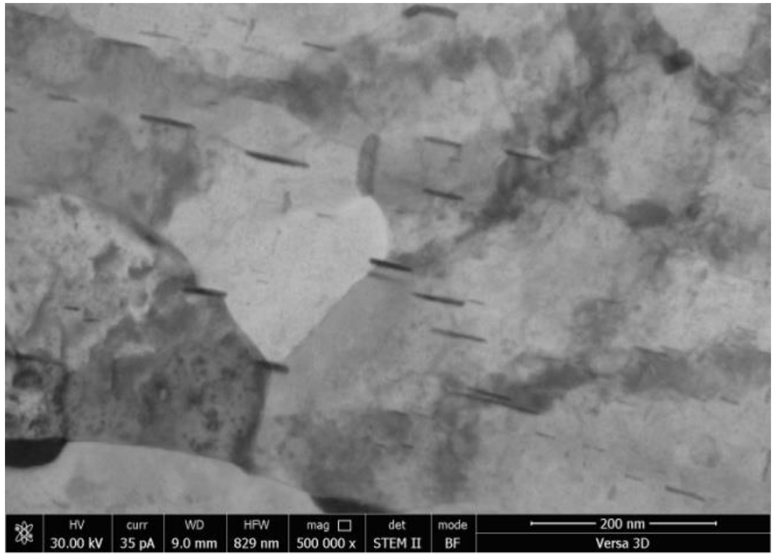
CHUNK MILL
Automatic

Site N/A
Project 100 %

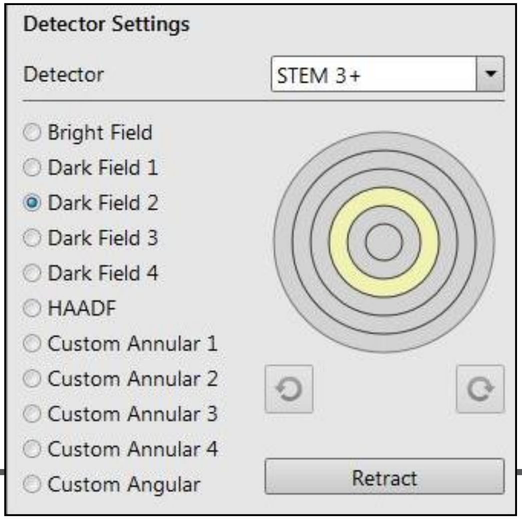
v4.1.0.508

Imaging of sample using the STEM 3+ detector

- High resolution imaging and high resolution EDS analysis
- 14-segment STEM detector for transmission imaging in bright field, dark field and high-angle (annular) dark field.



Bright field (BF) STEM image of Aluminum showing precipitates of 2nm width


Detector Settings

Detector: STEM 3+

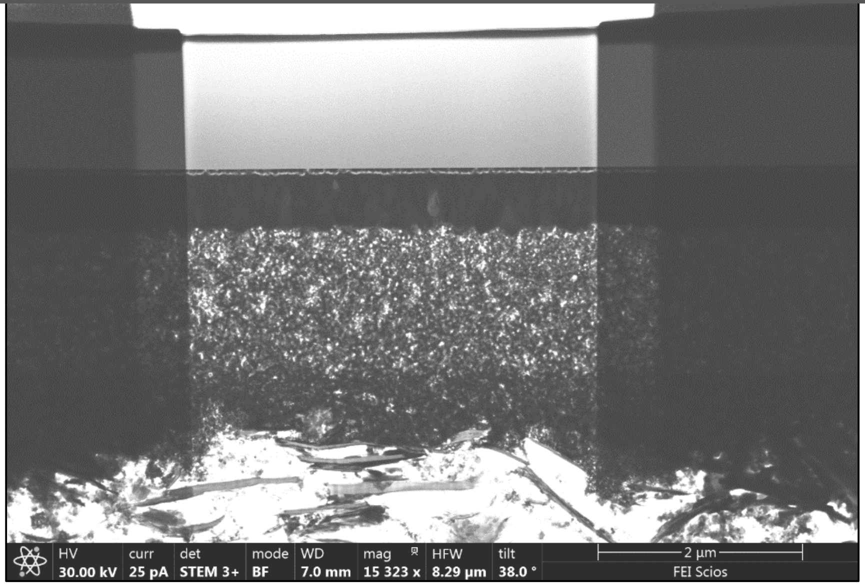
○ Bright Field
○ Dark Field 1
● Dark Field 2
○ Dark Field 3
○ Dark Field 4
○ HAADF
○ Custom Annular 1
○ Custom Annular 2
○ Custom Annular 3
○ Custom Annular 4
○ Custom Angular

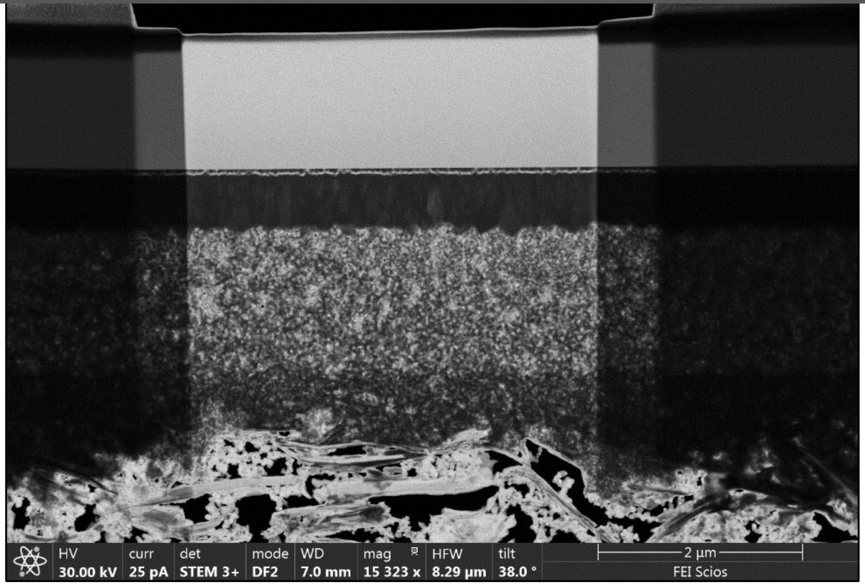
No TEM grid manipulation;
the samples are made and imaged without manipulating sample or breaking the vacuum

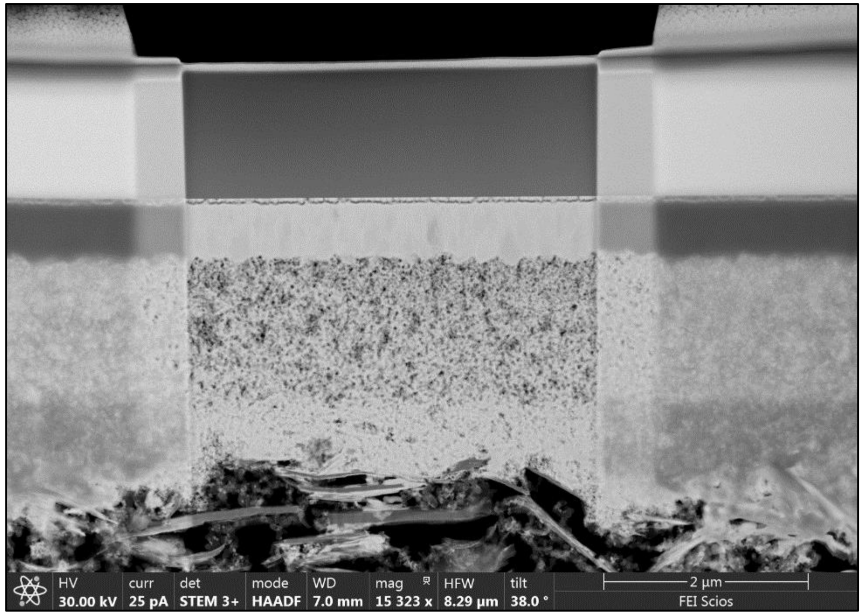
Selection of active segments in the UI.

(S)TEM prepared sample imaged with the STEM 3+ detector

STEM 3+ images;
BF, DF2, HAADF.
Simultaneously acquired
+ CCD image showing sample holder (tilted to horizontal)
+ inserted STEM 3+ detector







Milling in beam sensitive/soft material;

- Lower BC (500pA and below) -> less melting -> more efficient milling
- Lower ion dose; use InP or PMMA application file
- Use neg. OL -150% and faster DT
- Don’t use e-beam while patterning
- Sputter coat sample
- Total process will take longer; use autoTEM4 to prepare a chunk

NOTE: Temperature increase during milling;

conductive material a few degrees (<10K), non-conductive material (low thermal conductivity)
high temp increase (SiO2 ± 230K)