10/31/19

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Sample Preparation for High Resolution Analytical Transmission Electron Microscopy Using Xe PFIB

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Introduction

1. Differences between Ga and Xe
   - Beam profile
   - Deposition
     - Xe nanobubble formation
     - Limited precursor options

2. Projects
   - Perfecting the PFIB TEM sample
   - Pushing the boundaries of large area liftouts

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Typical INLO Ga FIB TEM Preparation

Milling current range = 300pA – 65nA

Pt or C deposition / protective cap

Sample at lift out around 20um x 10um

Grid thinning overtilts around 1 to 1.5°

Electron transparent window around 5um x 5um

Most samples can be made in a few hours

PFIB TEM Sample Preparation

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Beam Profile Differences Between Ga and Xe

Ga

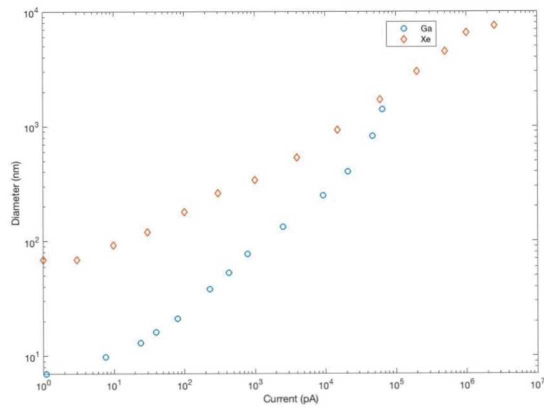
Current	Total Diameter
1.1 pA	7 nm
7.7 pA	9.8 nm
24 pA	12.9 nm
40 pA	16 nm
80 pA	21 nm
0.23 nA	38 nm
0.43 nA	53 nm
0.79 nA	77 nm
2.5 nA	133 nm
9.3 nA	249 nm
21 nA	400 nm
47 nA	820 nm
65 nA	1.4 um

Helios G3

Xe

Current	Total Diameter
1.0 pA	69 nm
3.0 pA	69 nm
10 pA	92 nm
30 pA	120 nm
100 pA	180 nm
0.30 nA	260 nm
1.0 nA	340 nm
4.0 nA	530 nm
15 nA	930 nm
60 nA	1.7 um
0.20 uA	3 um
0.50 uA	4.5 um
1.0 uA	6.5 um
2.5 uA	7.5 um

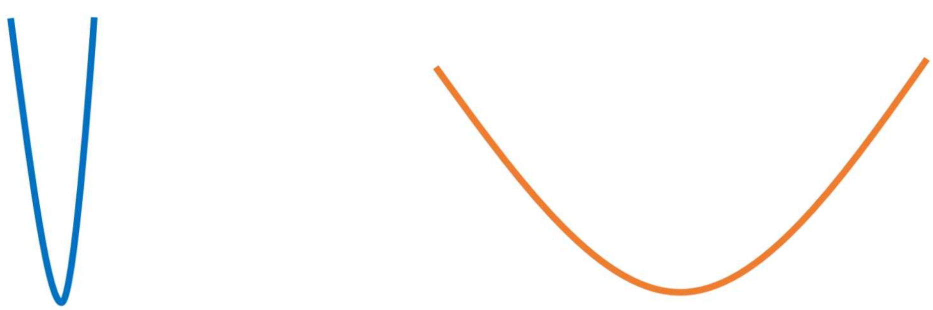
Helios G4



Total diameter = base on D_{50}

D_{50} = width of Gaussian where 50% of the beam is inside

Beam Profile Differences Between Ga and Xe

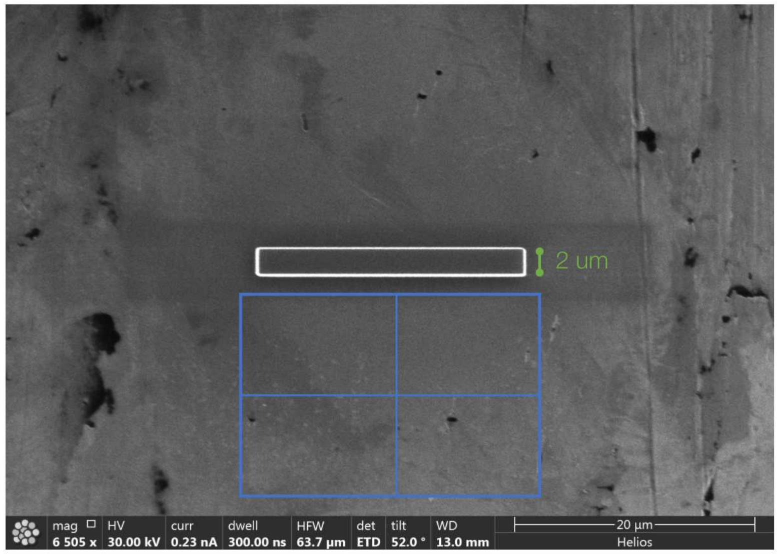


Ga

Xe

Beam Profile Differences Between Ga and Xe

Ga 65nA, ~1 um

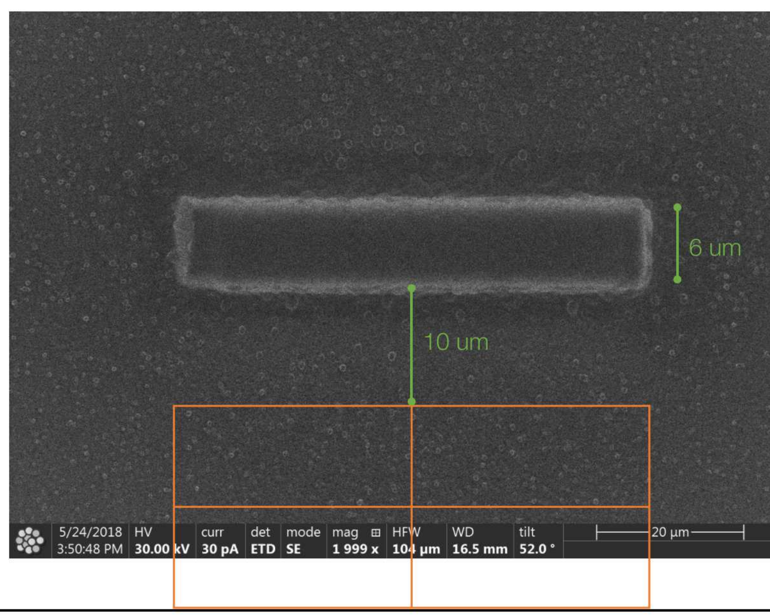


Beam Profile Differences Between Ga and Xe

Ga 65nA, ~1 um

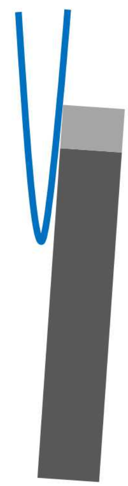
Xe 2.5uA, ~10 um

Xe 60nA, at edge
Xe 200nA, ~3 um
Xe 500nA, ~5 um
Xe 1uA, ~7 um



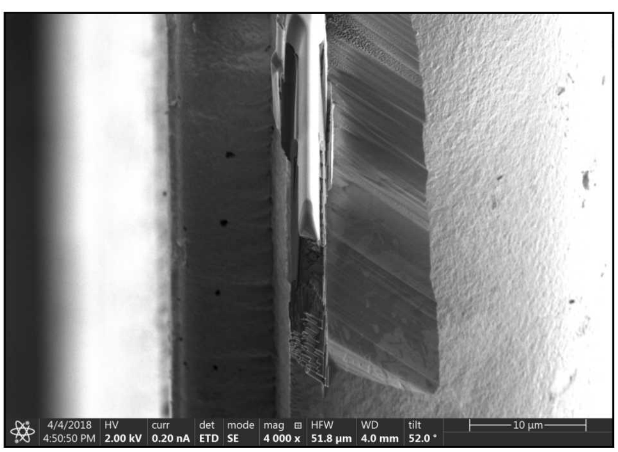
Beam Profile Differences Between Ga and Xe

D_{50} = 77 nm

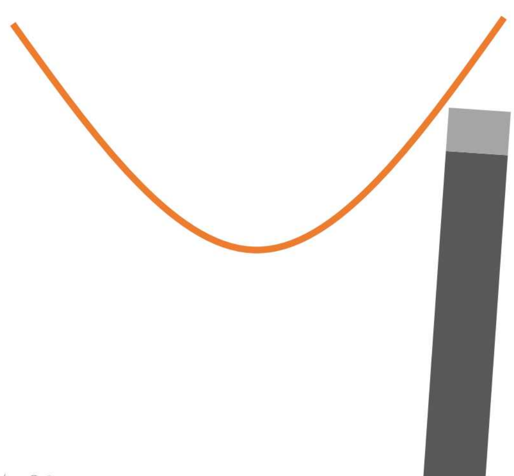


0.79nA, +/- 1.5°

Beam Profile Differences Between Ga and Xe

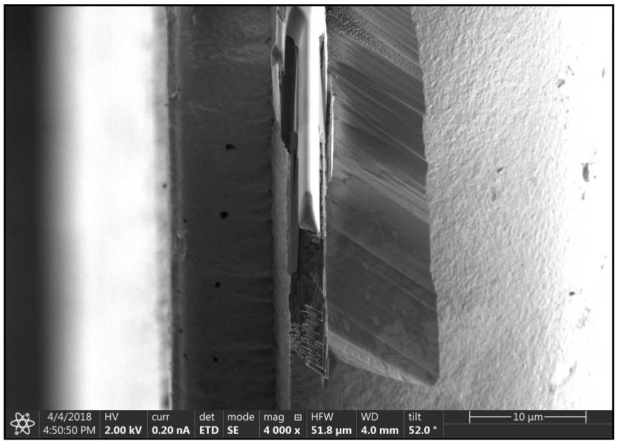


D_{50} = 530 nm

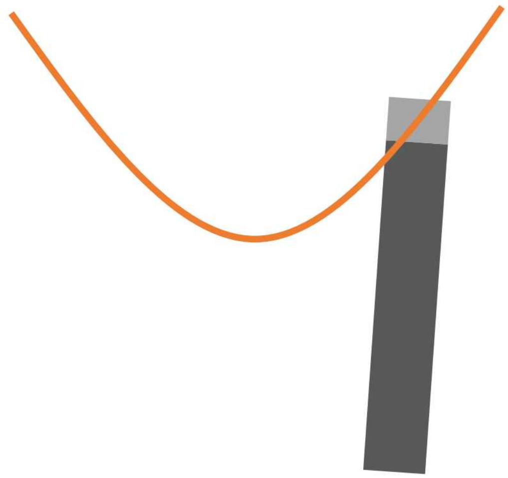


4nA, +/- 2°

Beam Profile Differences Between Ga and Xe

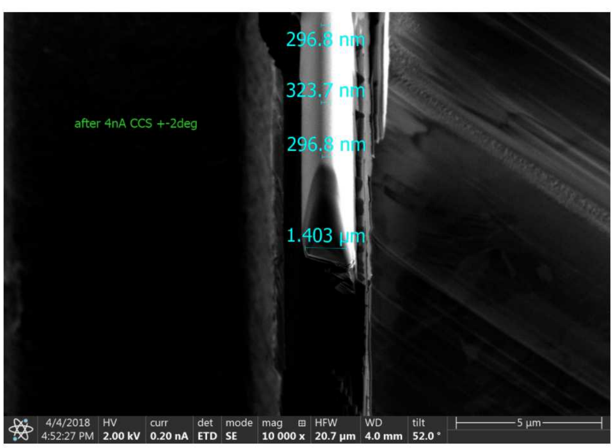


D_{50} = 530 nm



4nA, +/- 2°

Beam Profile Differences Between Ga and Xe



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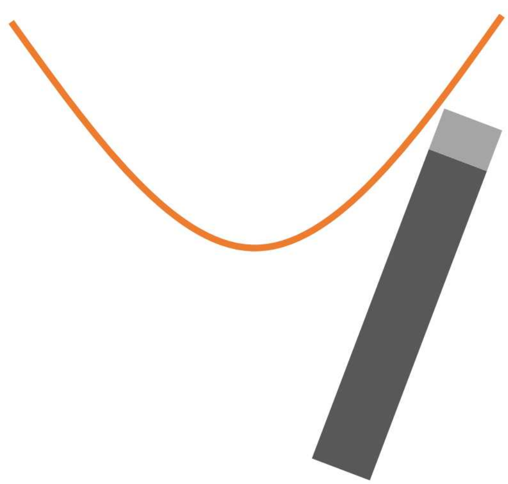
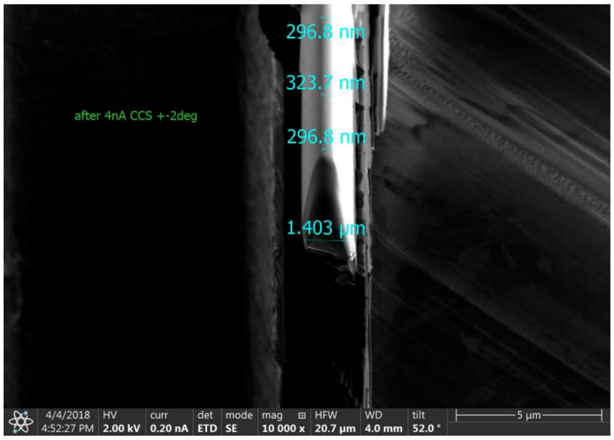
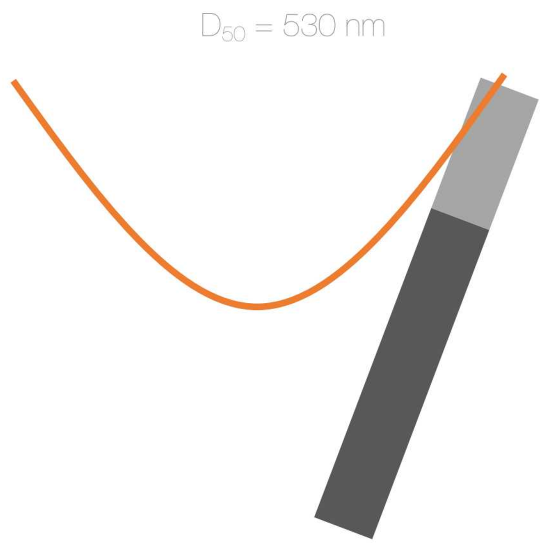


4nA, +/- 5°

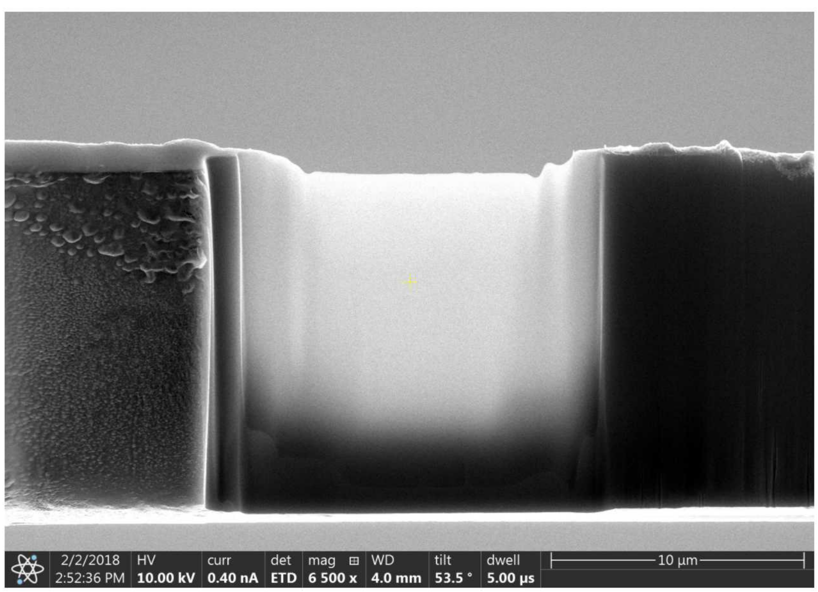
Beam Profile Differences Between Ga and Xe





4nA, +/- 5°

Ga recipe doesn't work for PFIB



PFIB TEM Sample Preparation

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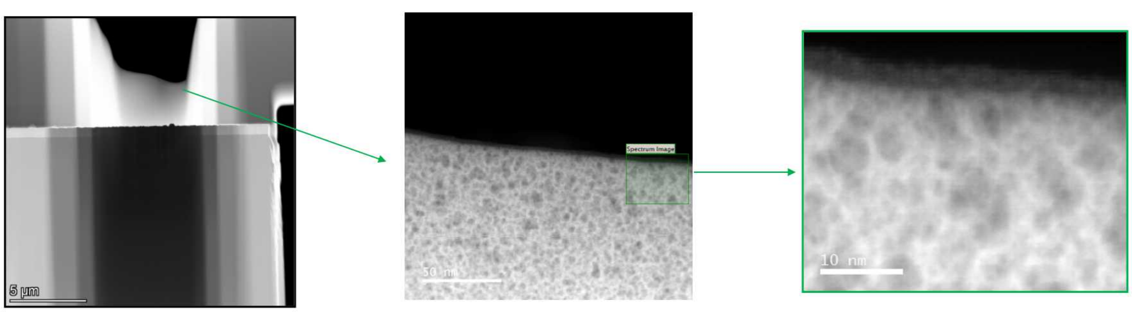
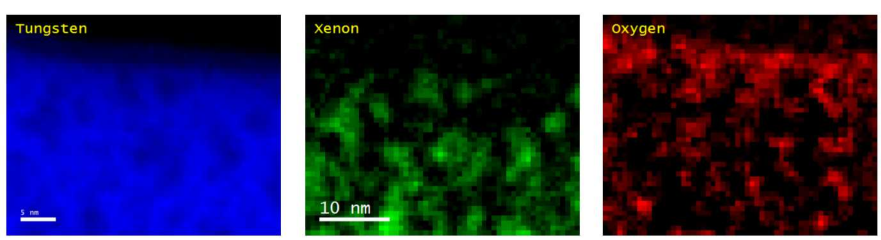
Xe Nanobubbles in W Deposition

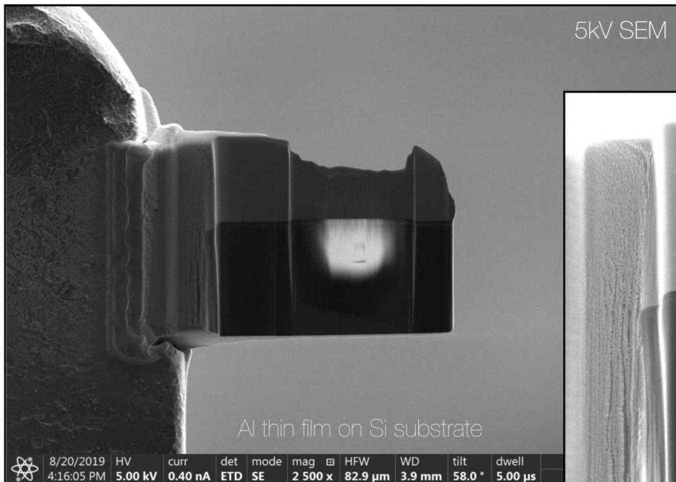


8kV bubbles = 5-7 nm
12kV bubbles = 8-10 nm

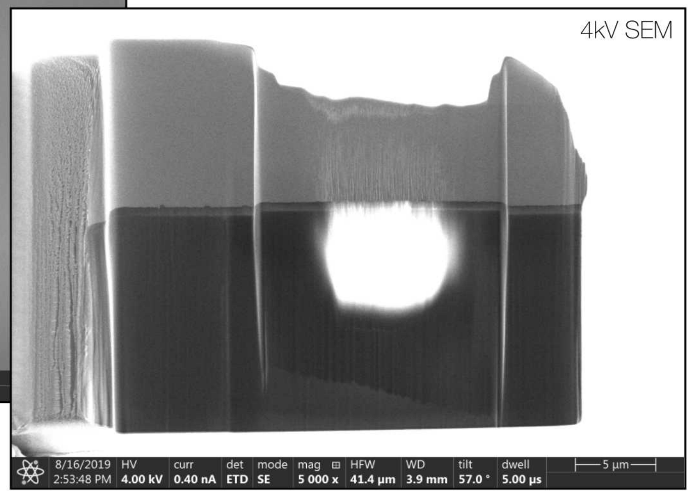
8kV W dep has bubbles but not enough to cause excessive curtaining in thin samples



Problems with 12kV W Deposition

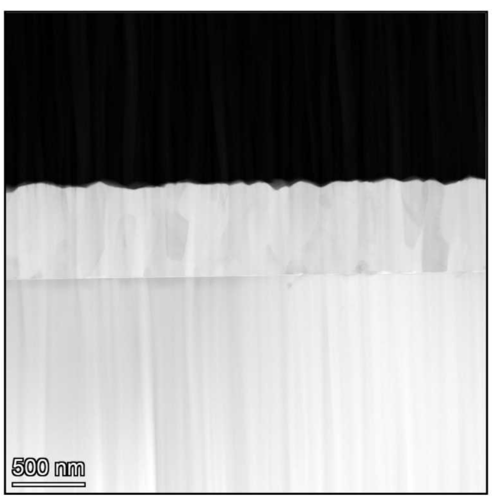


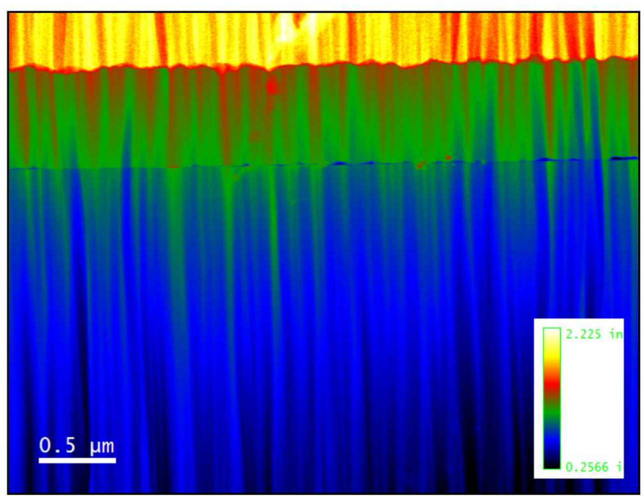
8kV bubbles = 5-7 nm
12kV bubbles = 8-10 nm



12kV dep bubbles are large enough to cause significant curtaining, resulting in a lower quality sample

Significant Curtaining from 12kV W Deposition





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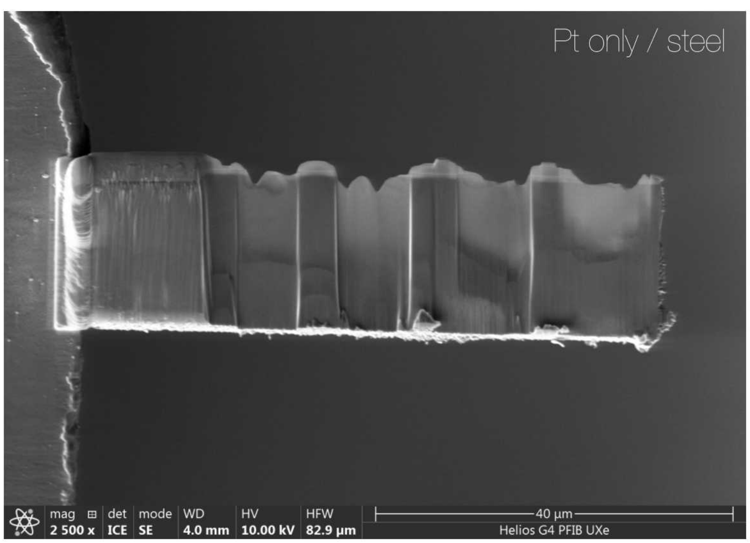
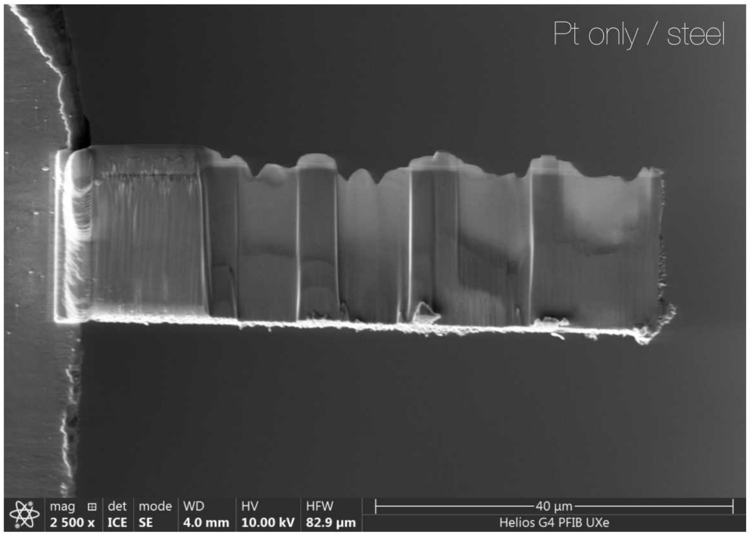
Pt Deposition Meets PFIB



Pt Deposition Meets PFIB

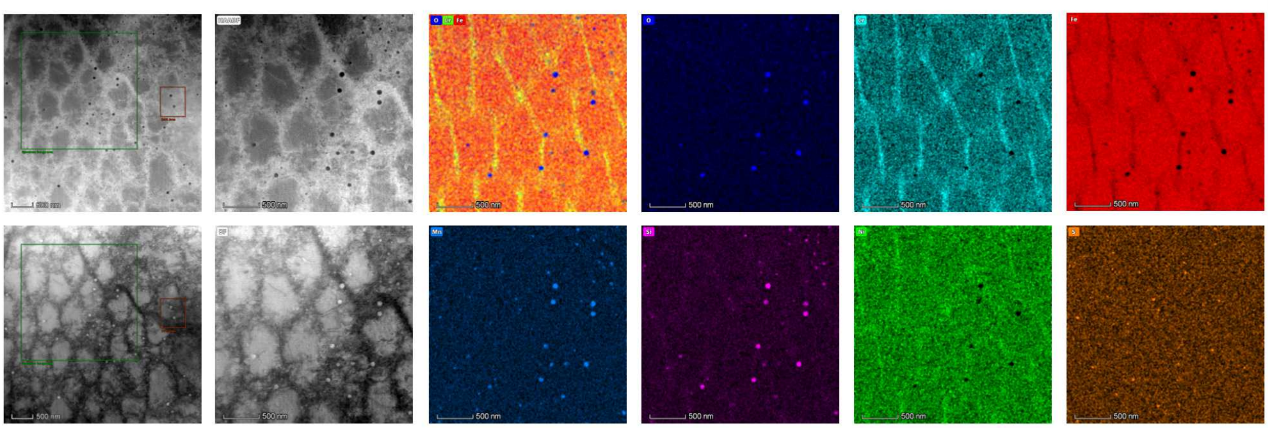


Depositions that do work with PFIB:

- W
- Pt + C (MultiChem)
- capless

Ron Kelley says…

Ugly Sample, Beautiful STEM Imaging / EDS



Sample doesn't have to be perfect, just good enough to answer the question.

PFIB TEM Sample Preparation

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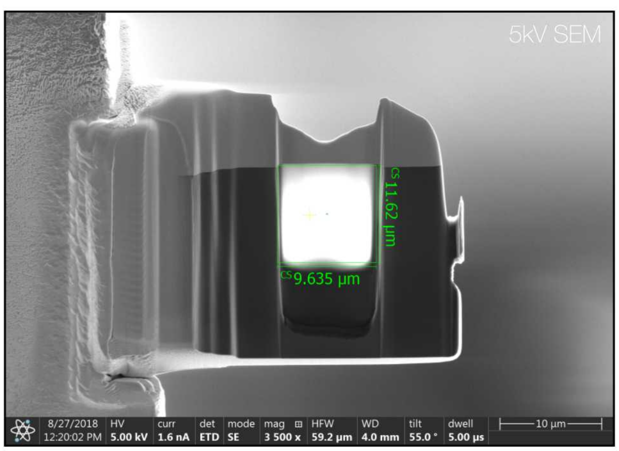
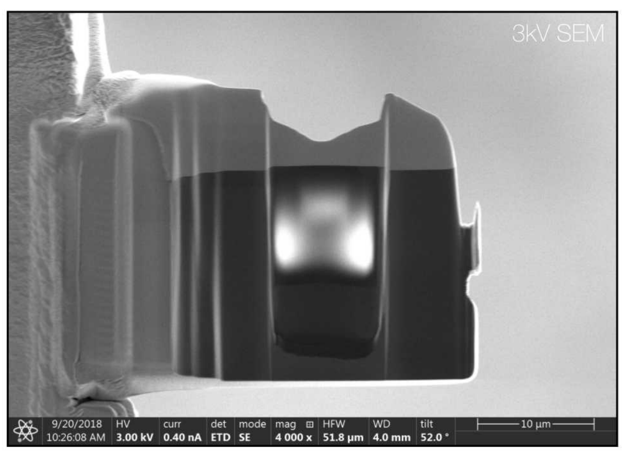
Si

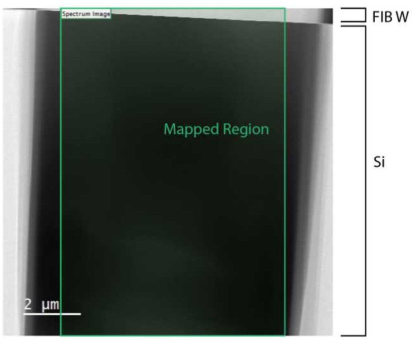
Trench 200nA
Wedge 15nA +/- 1.5
+/- 2 4nA 10
+/- 1.8 1nA 10
+/- 1.5 300pA 10
5kV +/- 5 100pA

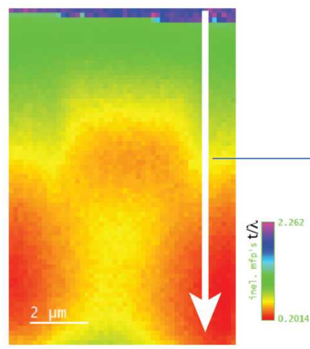


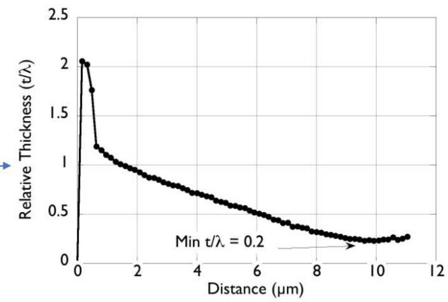


Thin area near middle of sample is a result of higher overtilts

Si Sample Thickness EELS







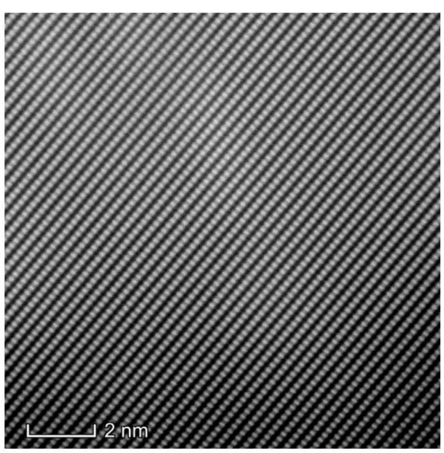
Thickness determined by log ratio method

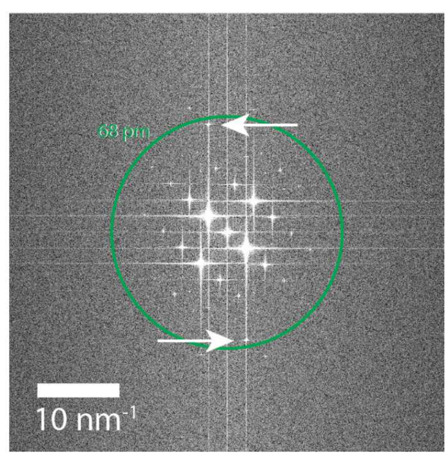
T. Malis, et al., J. of Microscope Technique 8 (1988)

Thinnest area = 35 nm

Thickness ranged from 140 nm to 35 nm

High Quality Si Sample for TEM

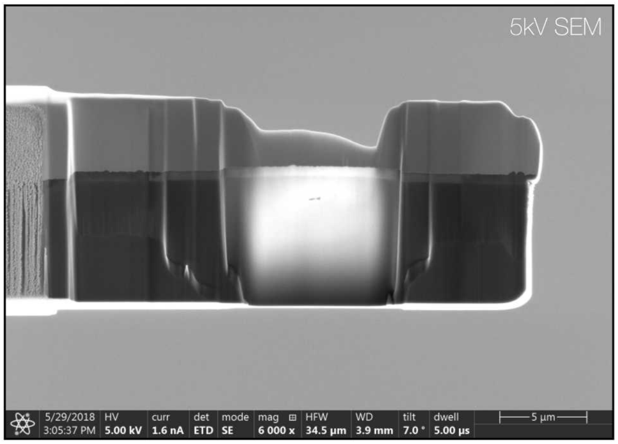




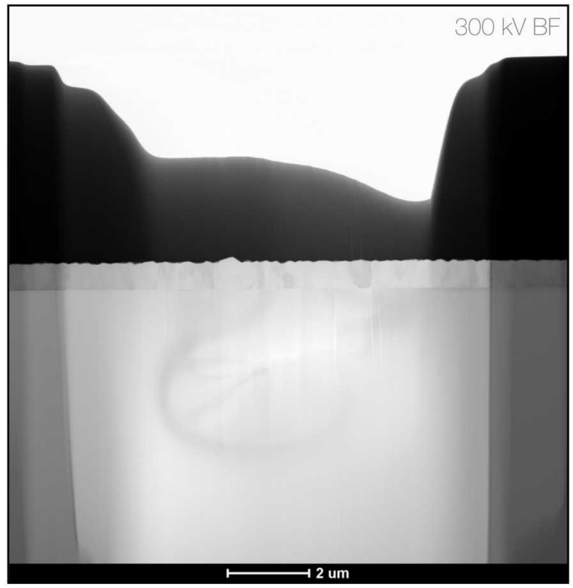
[110] Si is high quality. A few tweaks of the stigmator and the dumbells would be better resolved. Clearly, however, features sub 1 nm can be resolved.

Al on Si (2) Preparation

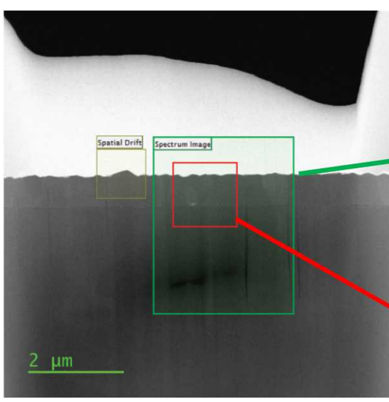
Trench 60nA
Wedge 15nA +/- 1.5
+/-1.5 1nA 10
+/-1 300pA 10
+/-1 100pA 10
5kV +/-5 100pA

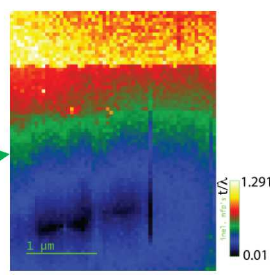


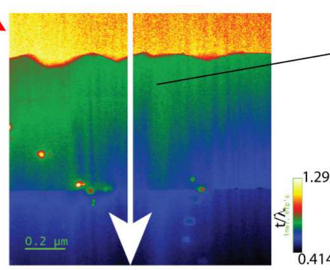
Electron transparent window is 10 um x 7 um

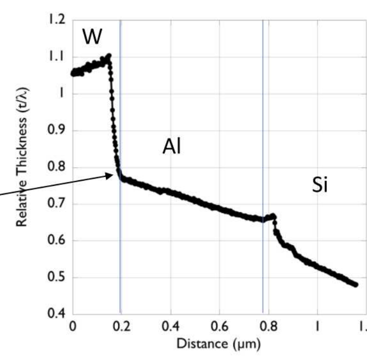


Al on Si (2) EELS Thickness Measurements







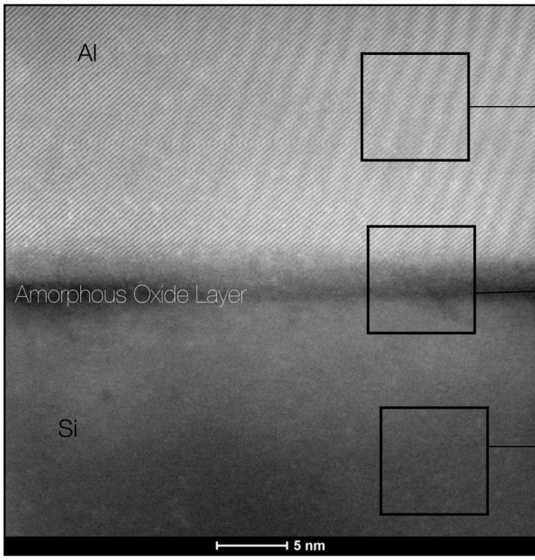


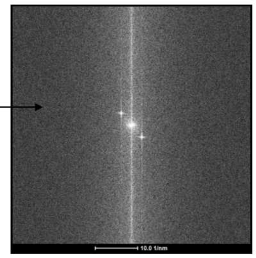
W = 110 nm
Al = 70 nm
Al/Si interface = 60nm
Si = 50 nm

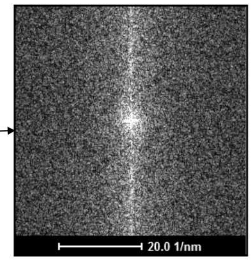
Thickness determined by log ratio method

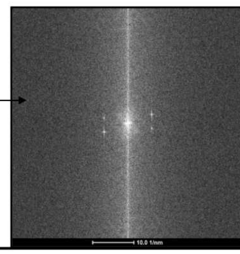
T. Malis, et al., J. of Microscope Technique 8 (1988)

Al on Si (2) HR-STEM









Lattice fringes appear in in the Al and Si, but not in the interfacial layer, showing the quality of the sample

Lessons Learned from Round 1

1. It is possible to make a high quality 30-40 nm thick sample using PFIB.
2. The sample thickness is non-uniform.
   - High overtilts – wedge shape

Lessons Learned from Round 1

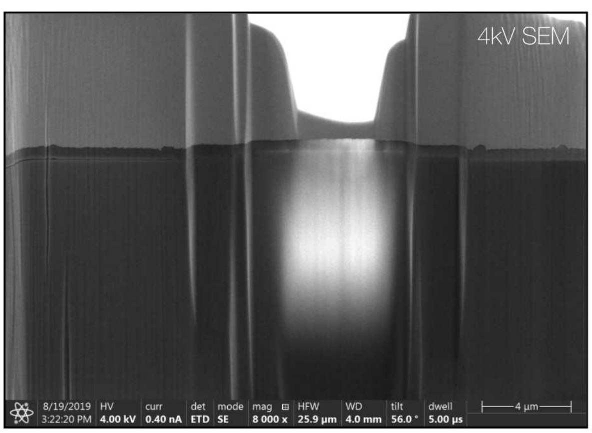
1. It is possible to make a high quality 30-40 nm thick sample using PFIB.
2. The sample thickness is non-uniform.
    - High overtilts – wedge shape

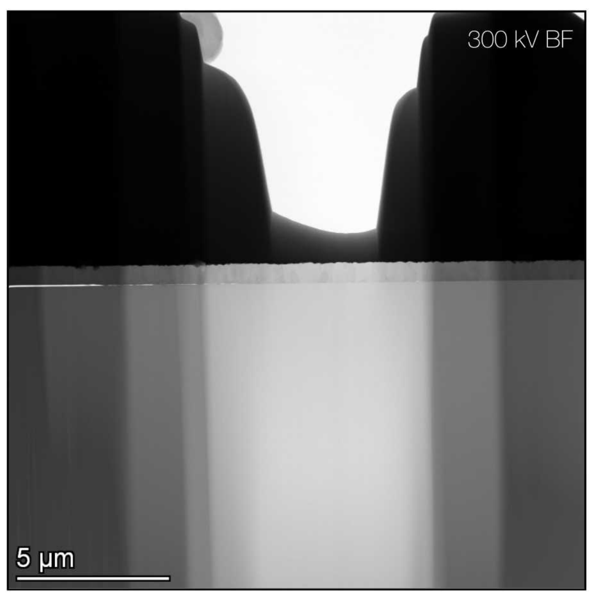
Next round…

- W / Al / Si all uniformly thin
- 50-80 nm final thickness

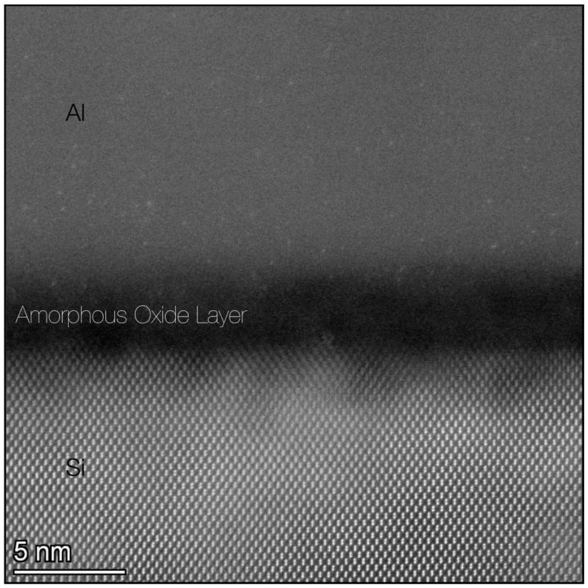
Al on Si (4) Sample Preparation

Trench 200nA
Wedge 60nA +/- 1.5
Grid coarse 15nA +1.5 front
+/-2 4nA watching line
+1.5 300pA 12W front
-1.5 300pA 12 back
-1 100pA 8W back
+1 100pA 8 front
5kV +1 100pA 6 back
5kV +1.5 100pA 6 front

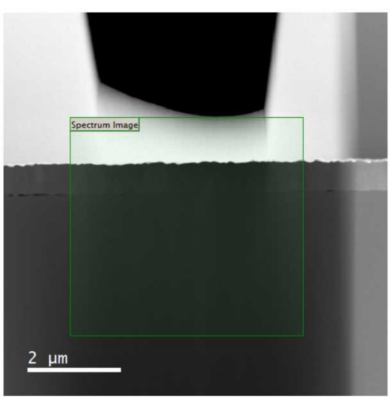


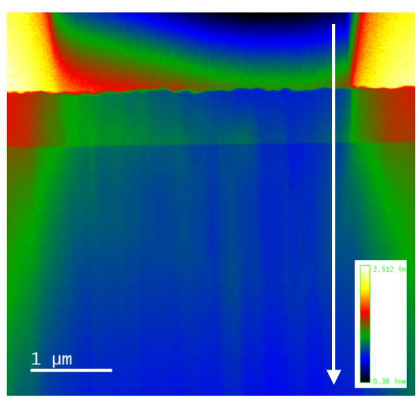


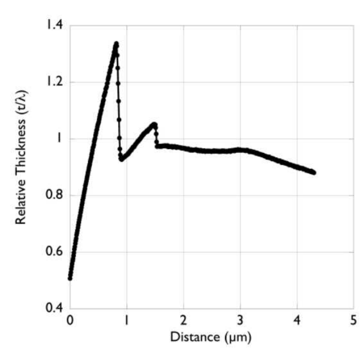
High Quality Sample



Al on Si (4) EELS Thickness Measurements



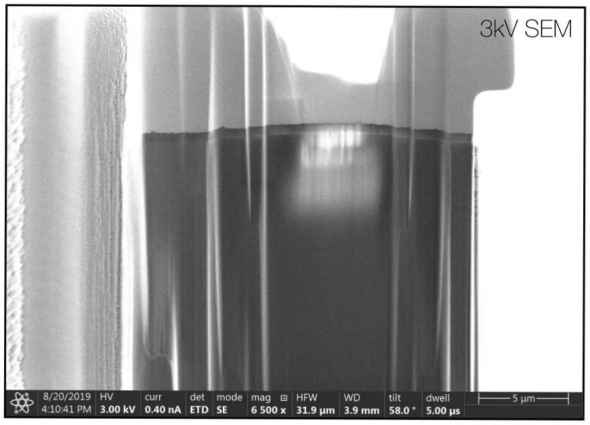


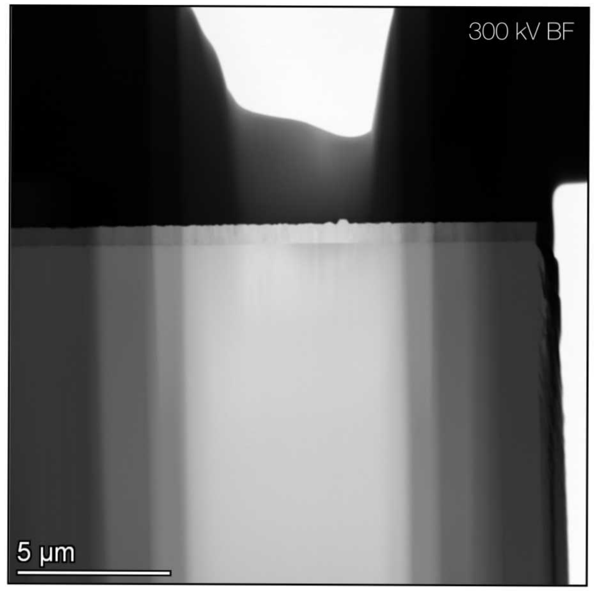


All layers ~120nm

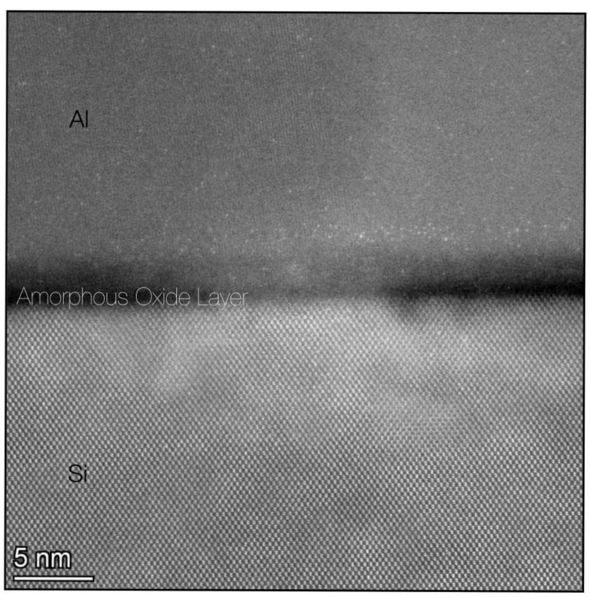
Al on Si (5) Sample Preparation

Trench 200nA
Wedge 15nA +/- 1.5
+/-2 4nA 20
+/-2 1nA 30
+/-1.5 300pA
+/-1 100pA
+1 100pA front only
5kV +/-2 100pA
5kV +4 100pA front

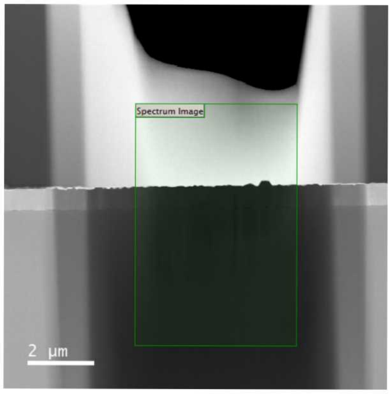




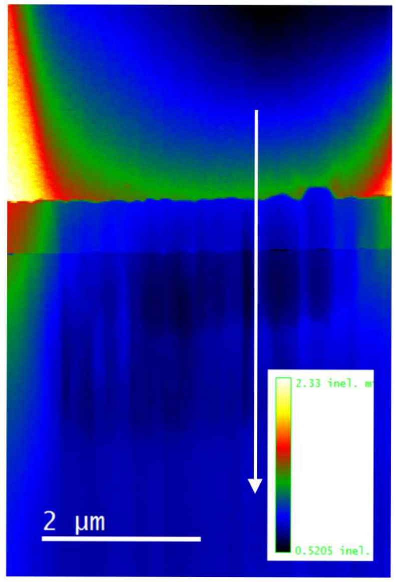
High Quality Sample

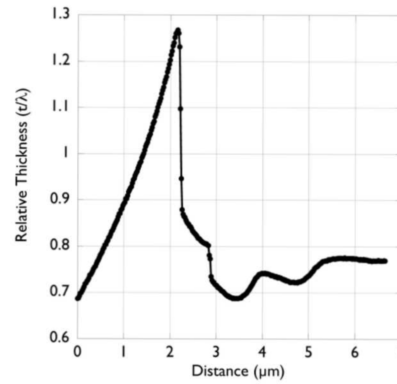


Al on Si (5) EELS Thickness Measurements



All layers ~100nm





Lessons Learned from Round 2

1. It is possible to make uniformly thin samples.
2. These samples are thick* (~100-120nm) because I stopped before running out of dep.

Lessons Learned from Round 2

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2. These samples are thick* (~100-120nm) because I stopped before running out of dep.

Next round of samples…

- More dep to get ROI to dead center of sample?
- Lower currents? (smaller beam profile)

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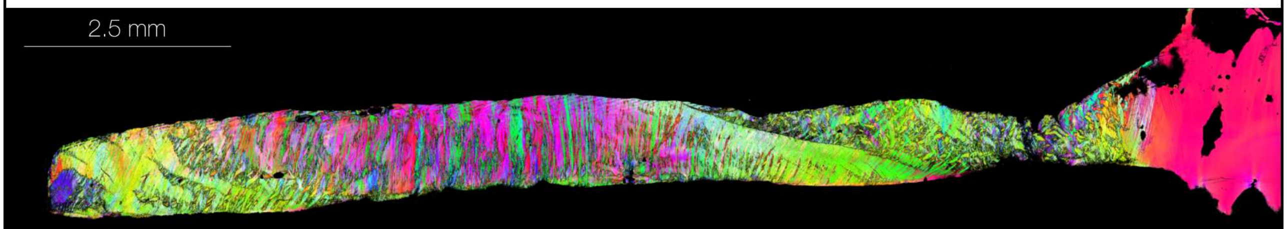
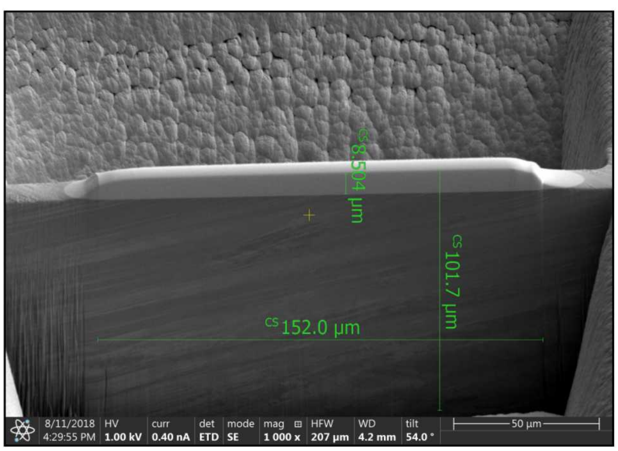
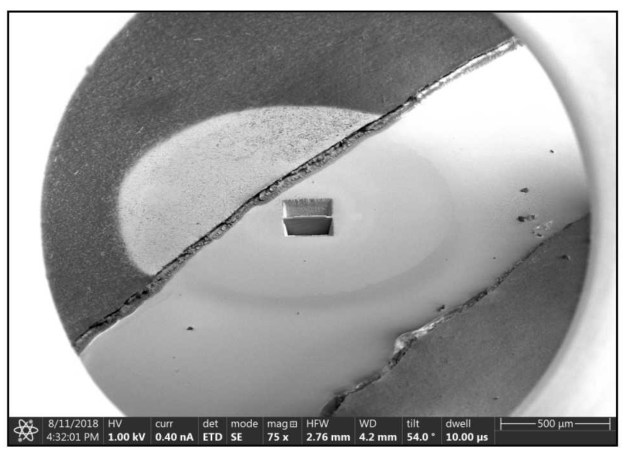
Single Crystal Steel Tensile Specimen



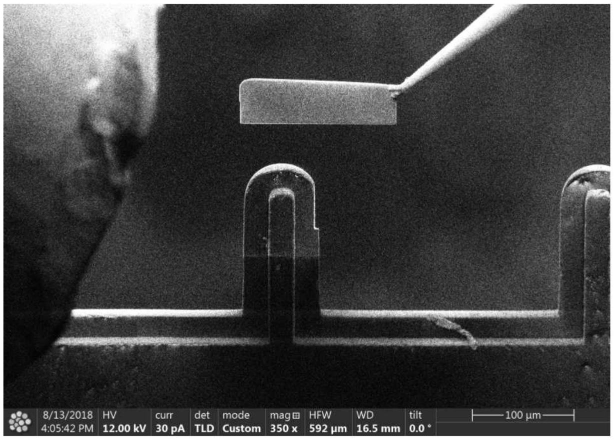
Can we use PFIB to make a site specific sample with an electron transparent region as large as a conventionally prepared sample?

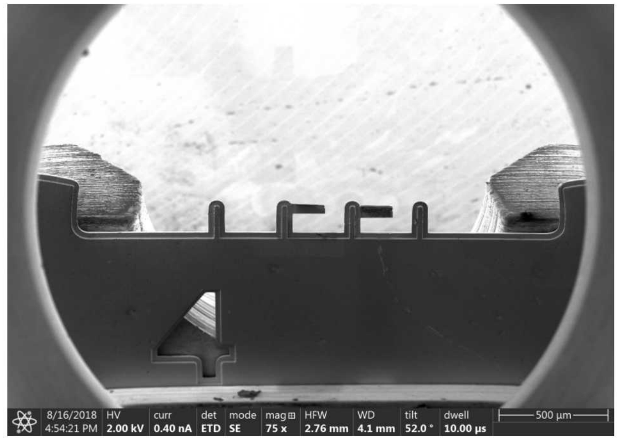
Single Crystal Steel Sample Preparation





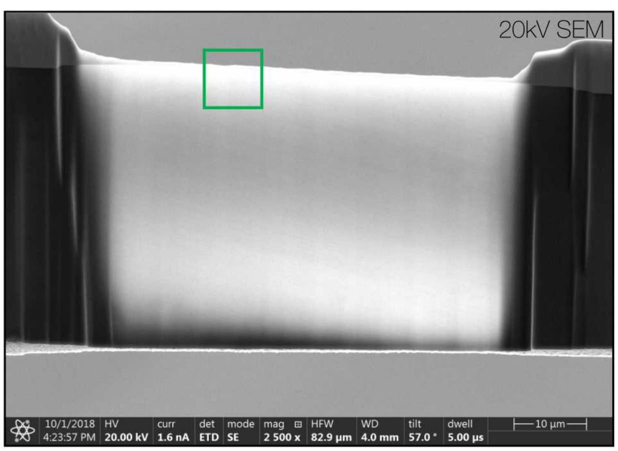
Single Crystal Steel Sample Preparation





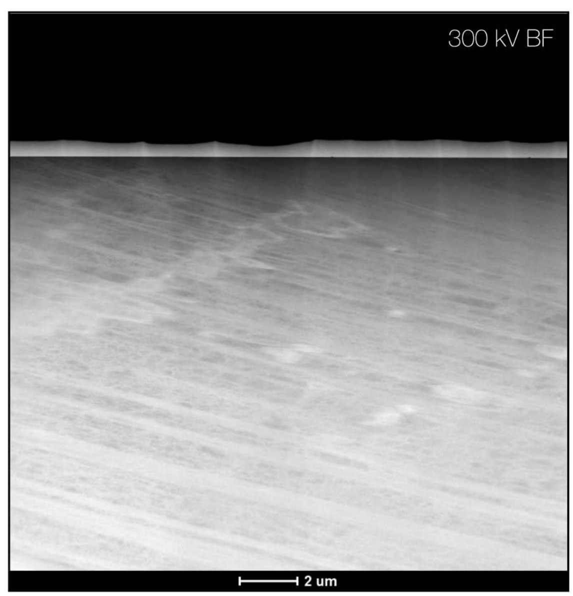
Lift out is 143 um x 43 um

Single Crystal Steel STEM

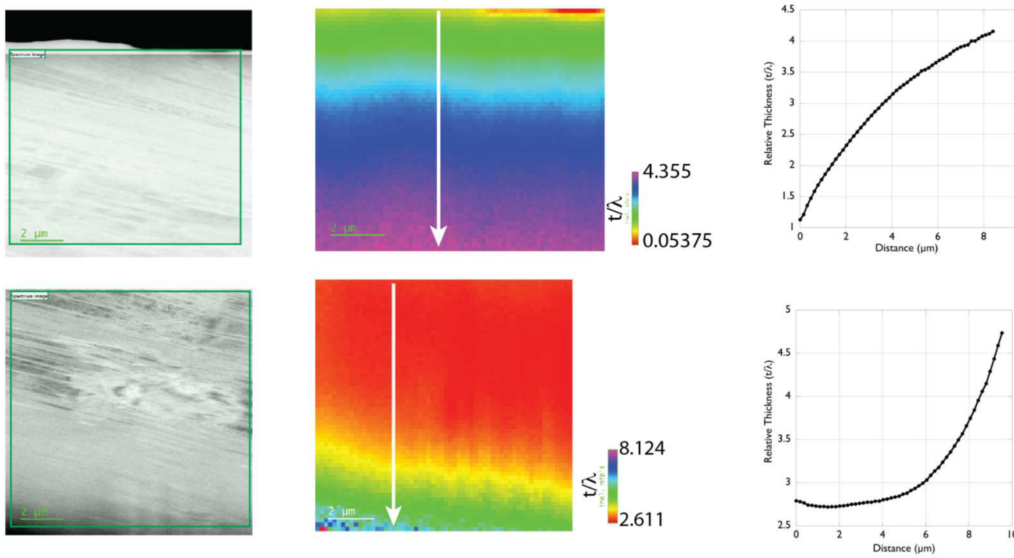


Electron transparent window is 53 um x 41 um

At lowest magnification, only 1/15th of the entire window is viewable.

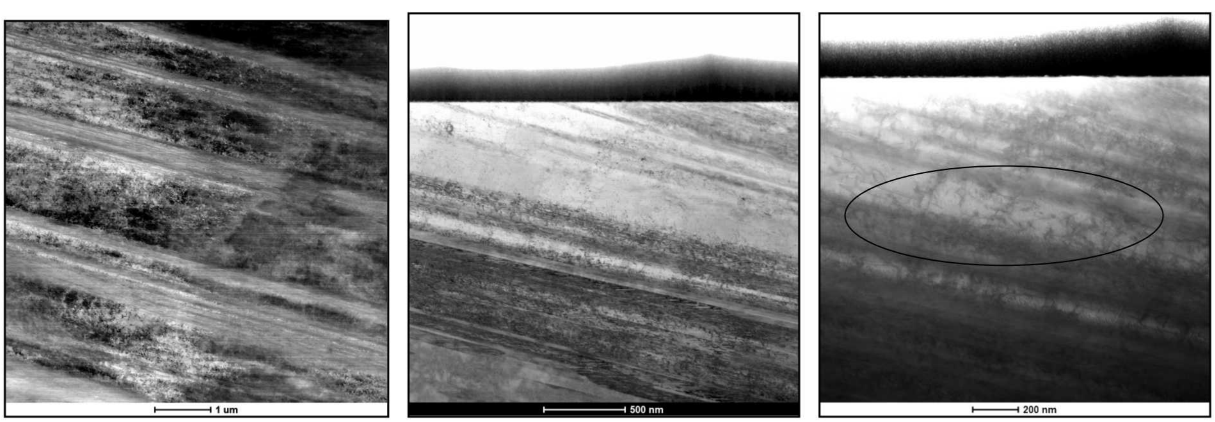


Single Crystal Steel EELS Thickness Measurements



Thinnest area ≈ 120 nm (near top) at bottom thickness ≈ 500 nm

Single Crystal Steel STEM Deformation Microstructures



Despite the thickness, we are still able to perform diffraction contrast STEM and image dislocation structures resulting from deformation

Conclusions

- Despite larger beam profiles, high quality TEM samples can be made with PFIB
- New geometries, precursors, and milling strategies should continue to be explored to perfect the PFIB TEM process
- PFIB is pushing limits of "big samples" to include larger electron transparent regions, rivaling conventional preparation methods
- These techniques work on a wide variety of other more challenging materials – porous, heterogeneous, insulators – let's chat!