Vertical quantum confinement structures

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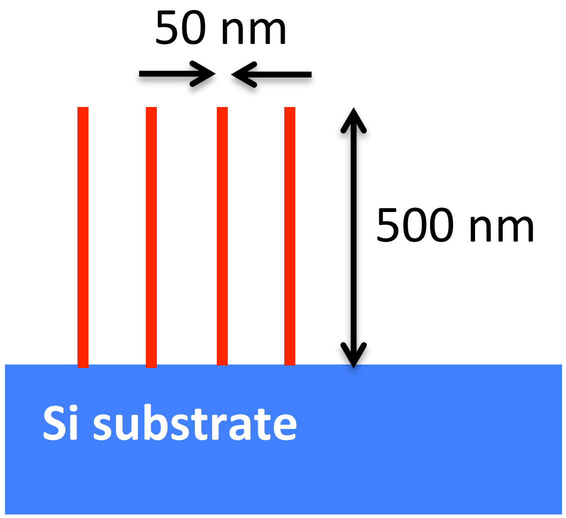
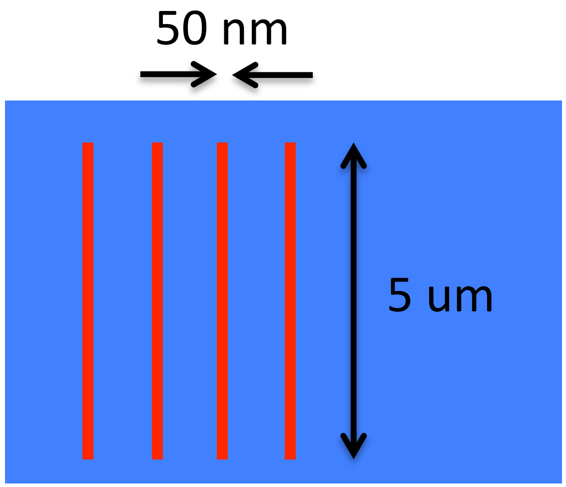
Fabrication Goal

- Fabricate vertical “blades” of InSb or InAs with dimensions:
  - ☐ <50 nm wide
  - ☐ >10:1 height-to-width aspect ratio
  - ☐ several um long

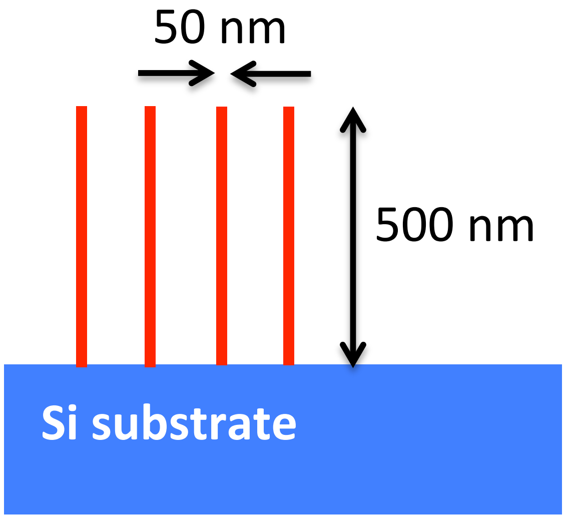


Cross-section

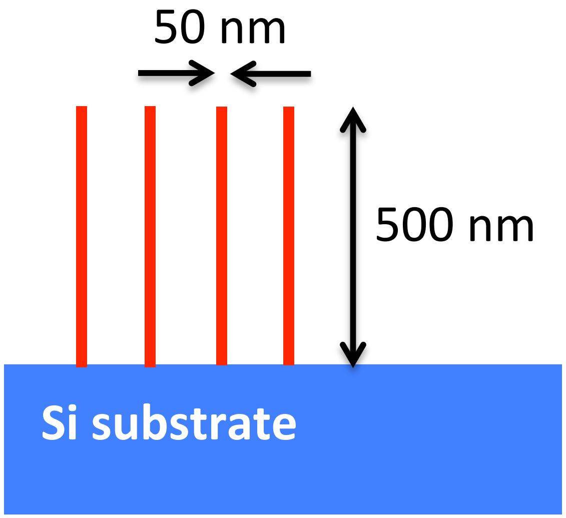


Top view

Challenges



Challenges

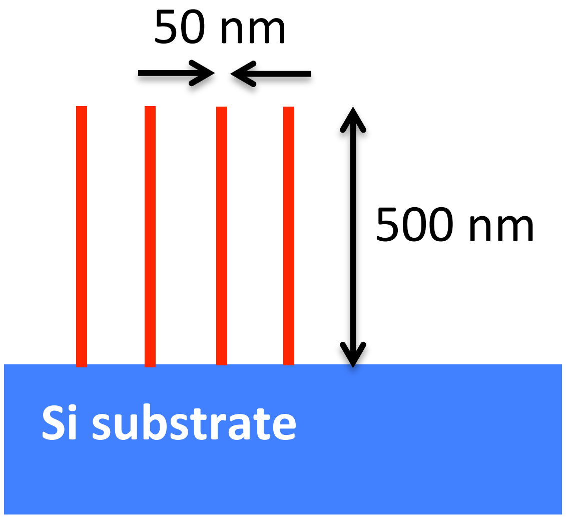


Deposition

InAs
MOCVD
(1)

InSb
Sputtering

Challenges



Deposition

InAs
MOCVD
(1)

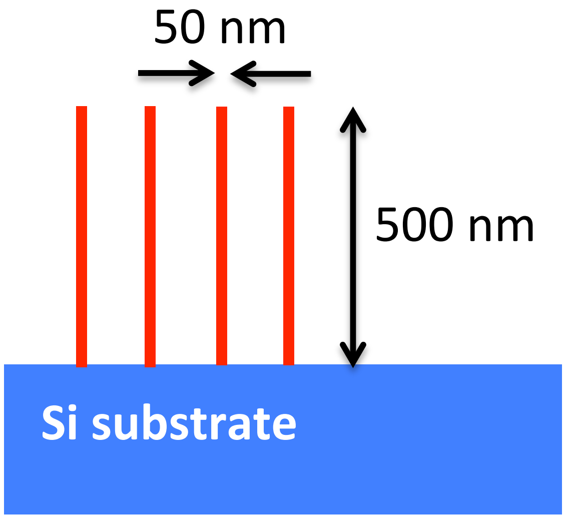
InSb
Sputtering

Lithography

JEOL
e-beam
lithography
(3)

ASML
double
exposure
(2)

Challenges



Deposition

InAs
MOCVD
(1)

InSb
Sputtering

Lithography

JEOL
e-beam
lithography
(3)

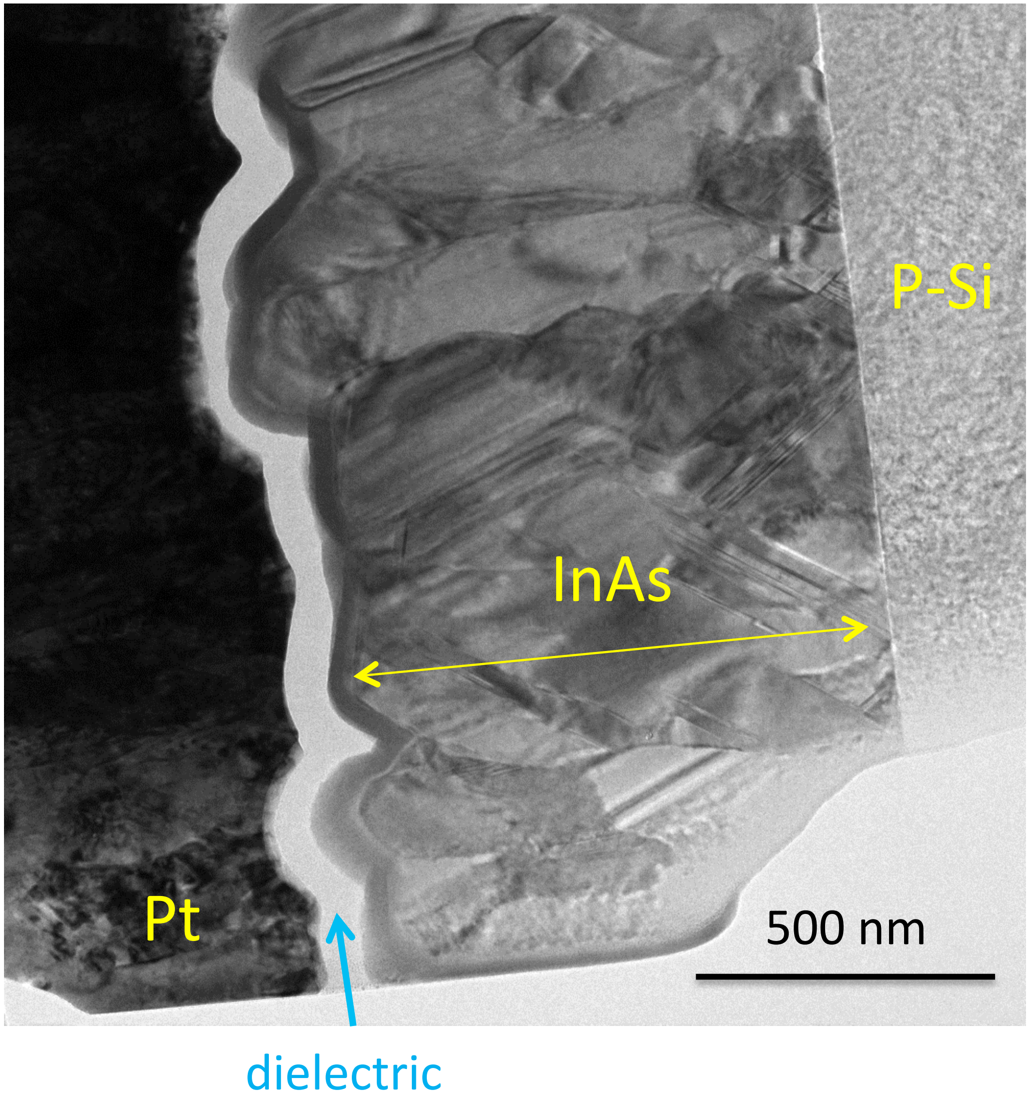
ASML
double
exposure
(2)

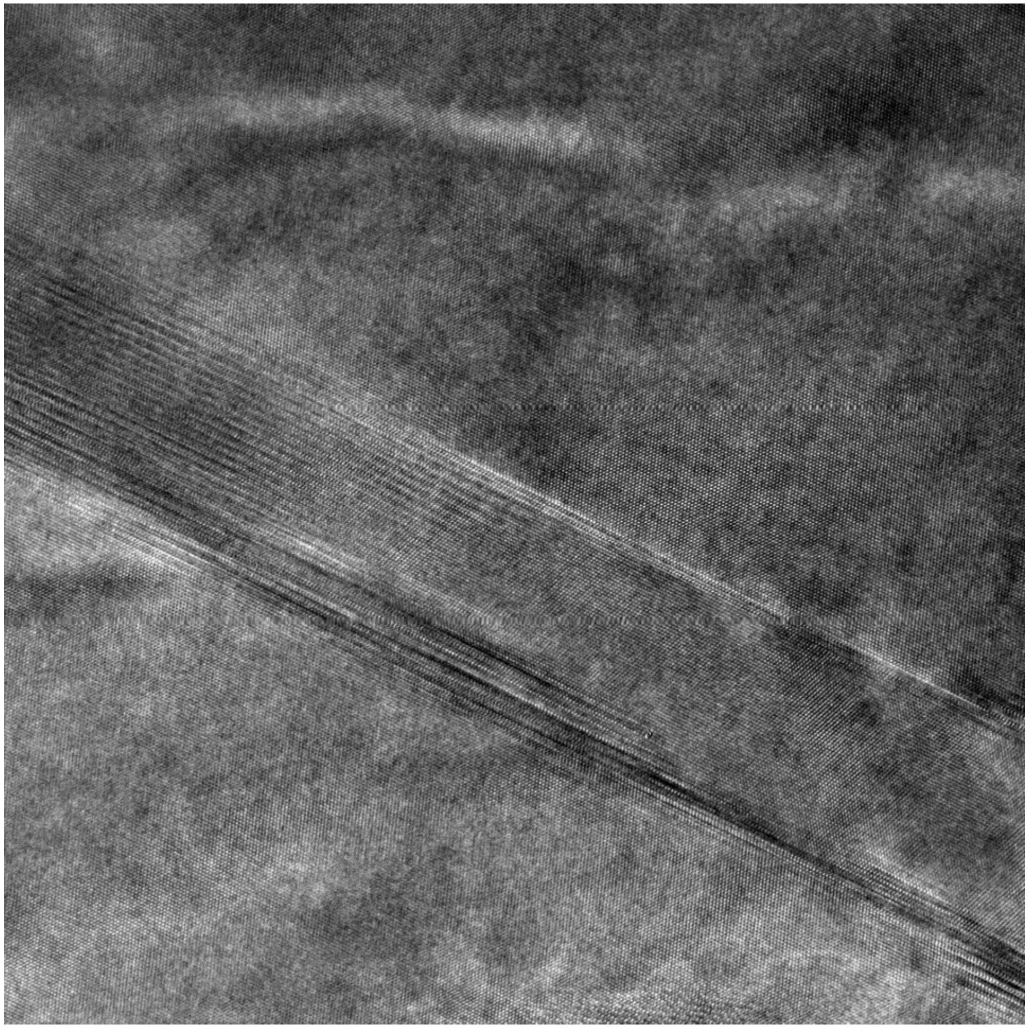
Patterning

RIE in
Ox-35
(4)

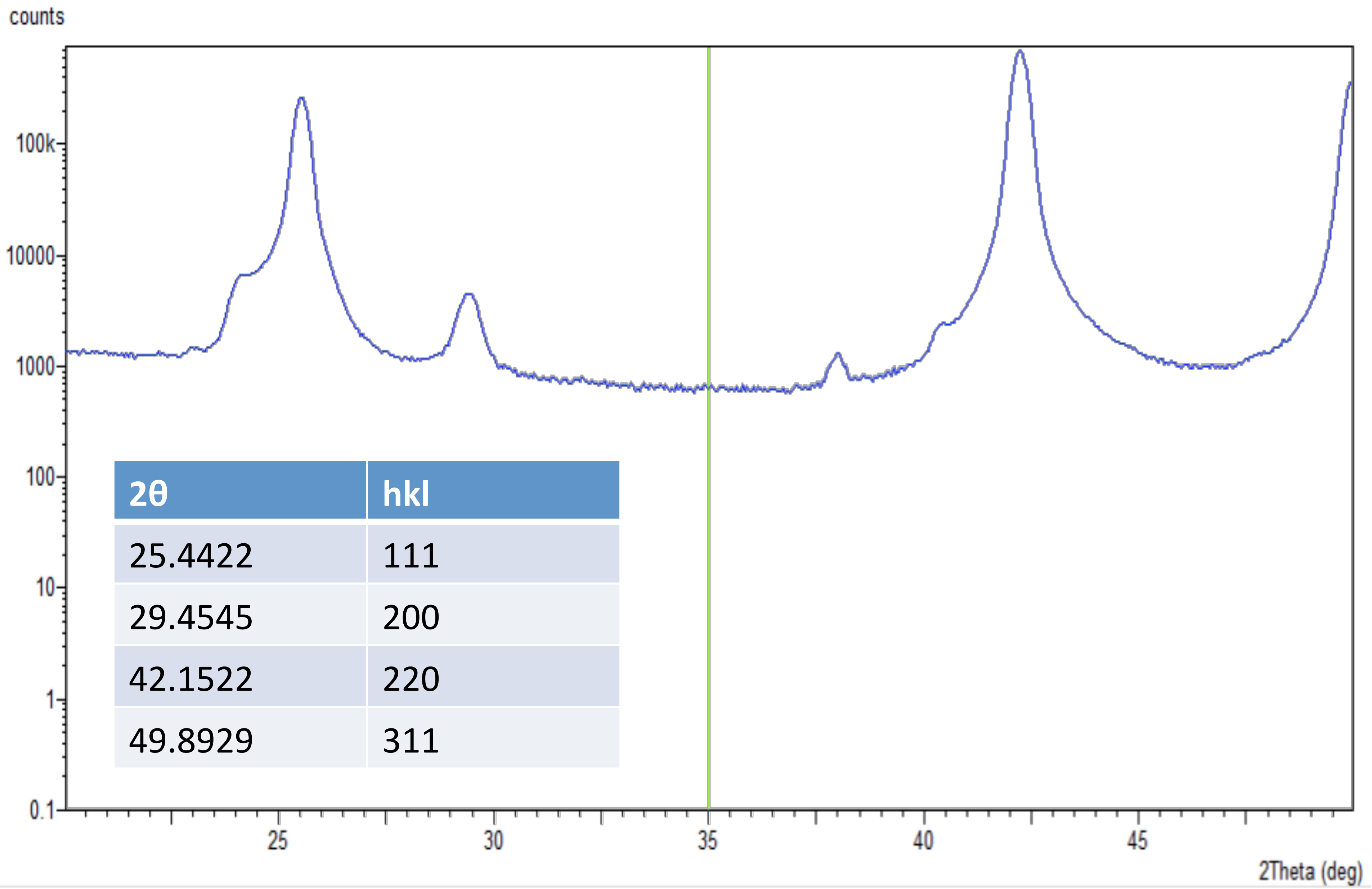
(1) InAs MOCVD growth

Regular deposition at 400C





XRD characterization of MOCVD InAs



2θ	hkl
25.4422	111
29.4545	200
42.1522	220
49.8929	311

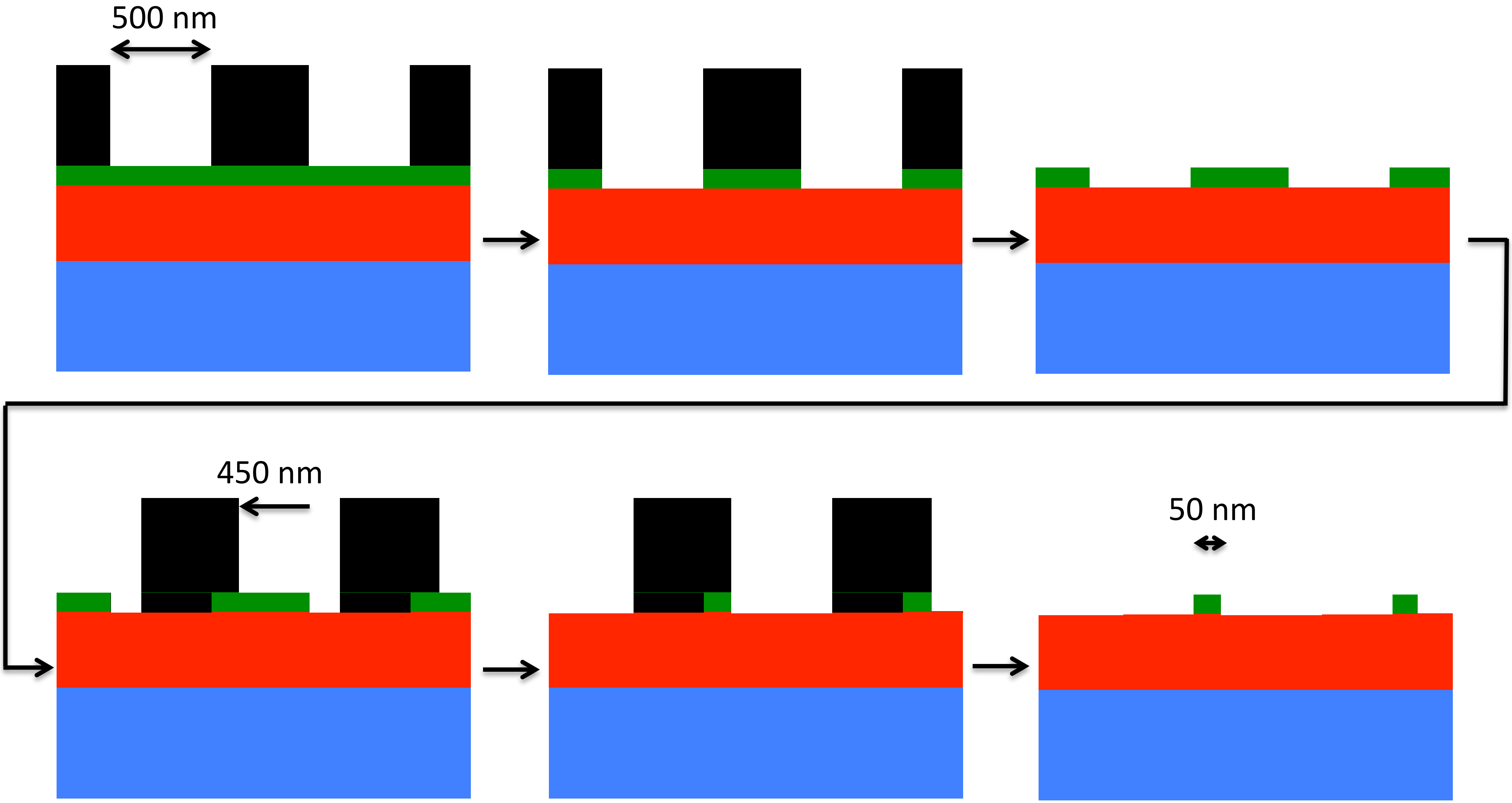
(2) ASML double exposure

- Why try this in the first place?
  - Higher throughput & lower cost than e-beam
  - Well-characterized resist properties for etching

- 2 main options:
  a) Hard mask, regular exposure twice with shift
  b) One resist layer, half-exposed twice with shift

(2) ASML double exposure – Hard Mask

🟦 Si 🟥 InAs / InSb 🟩 hard mask ⬛ SPR 955-.7



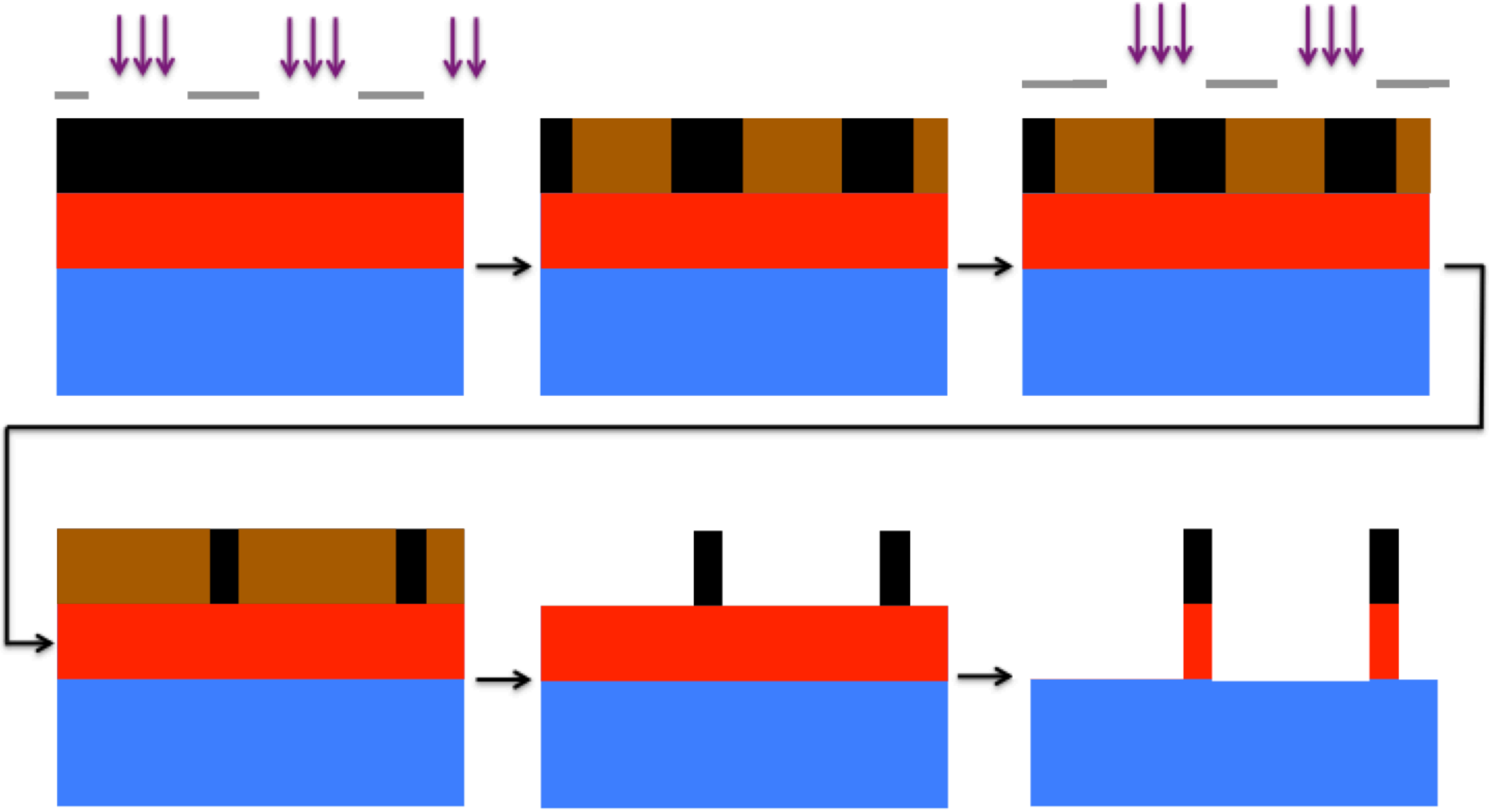
(2) ASML double exposure – Single Resist

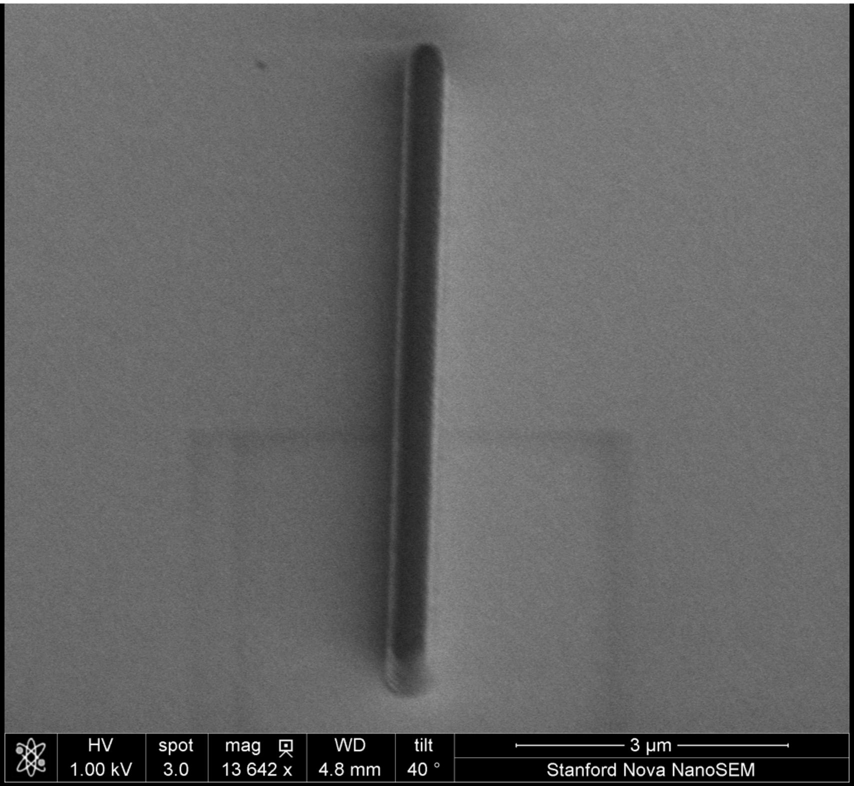
🟦 Si

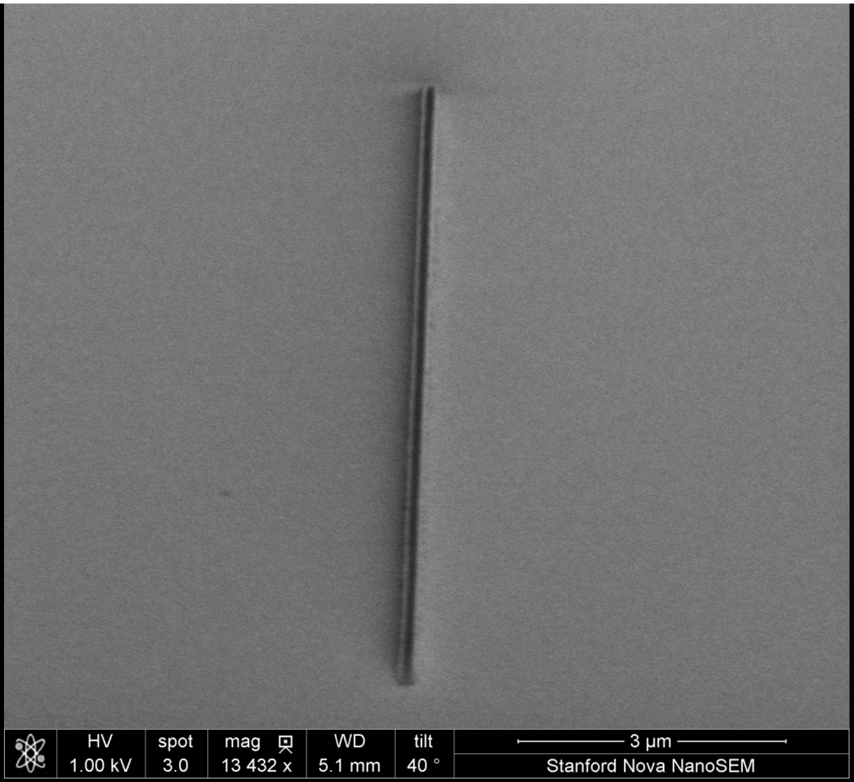
🟥 InAs / InSb

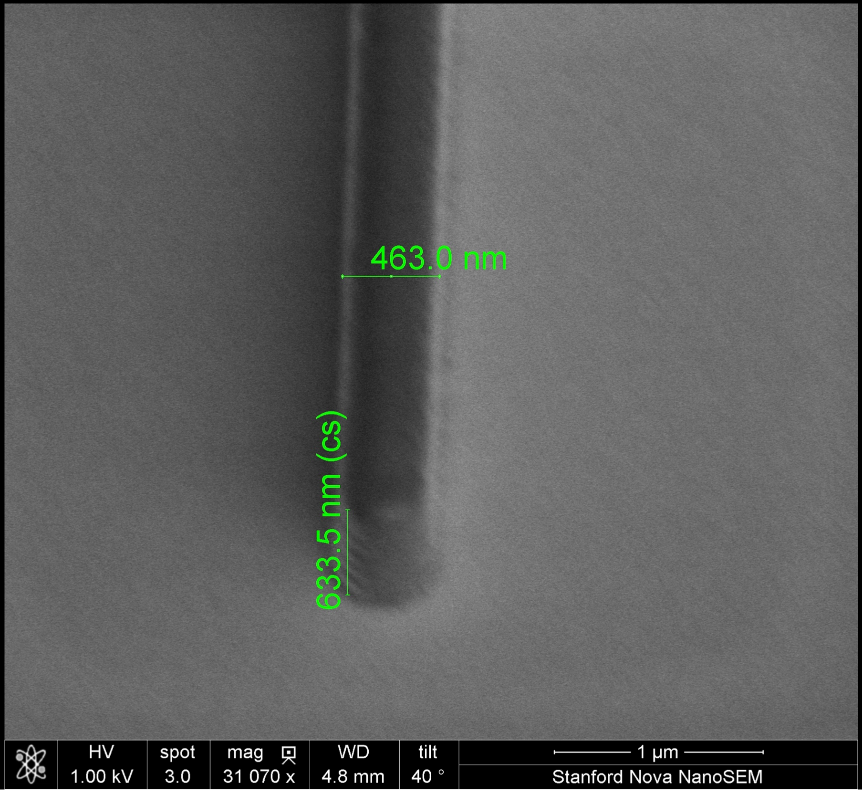
⬛ SPR 955-.7

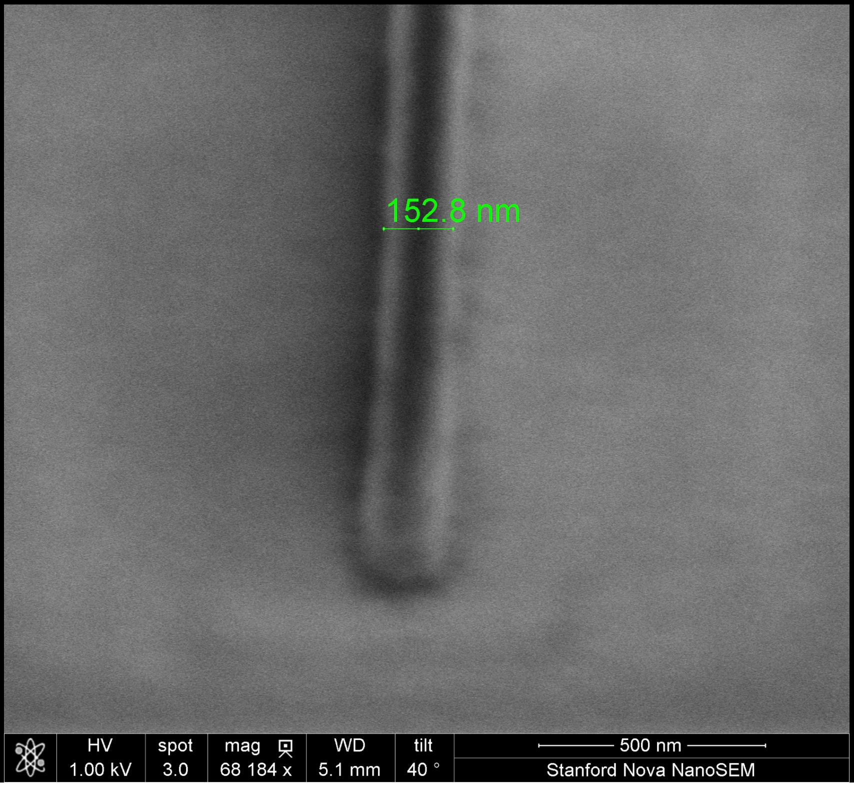
🟫 Exposed 955




HV
1.00 kV
spot
3.0
mag
13 642 x
WD
4.8 mm
tilt
40 °
3 µm
Stanford Nova NanoSEM


HV
1.00 kV
spot
3.0
mag
13 432 x
WD
5.1 mm
tilt
40 °
3 µm
Stanford Nova NanoSEM


463.0 nm
633.5 nm (cs)
HV
1.00 kV
spot
3.0
mag
31 070 x
WD
4.8 mm
tilt
40 °
1 µm
Stanford Nova NanoSEM


152.8 nm
HV
1.00 kV
spot
3.0
mag
68 184 x
WD
5.1 mm
tilt
40 °
500 nm
Stanford Nova NanoSEM

Double Exposure Testing Methodology

Parameters

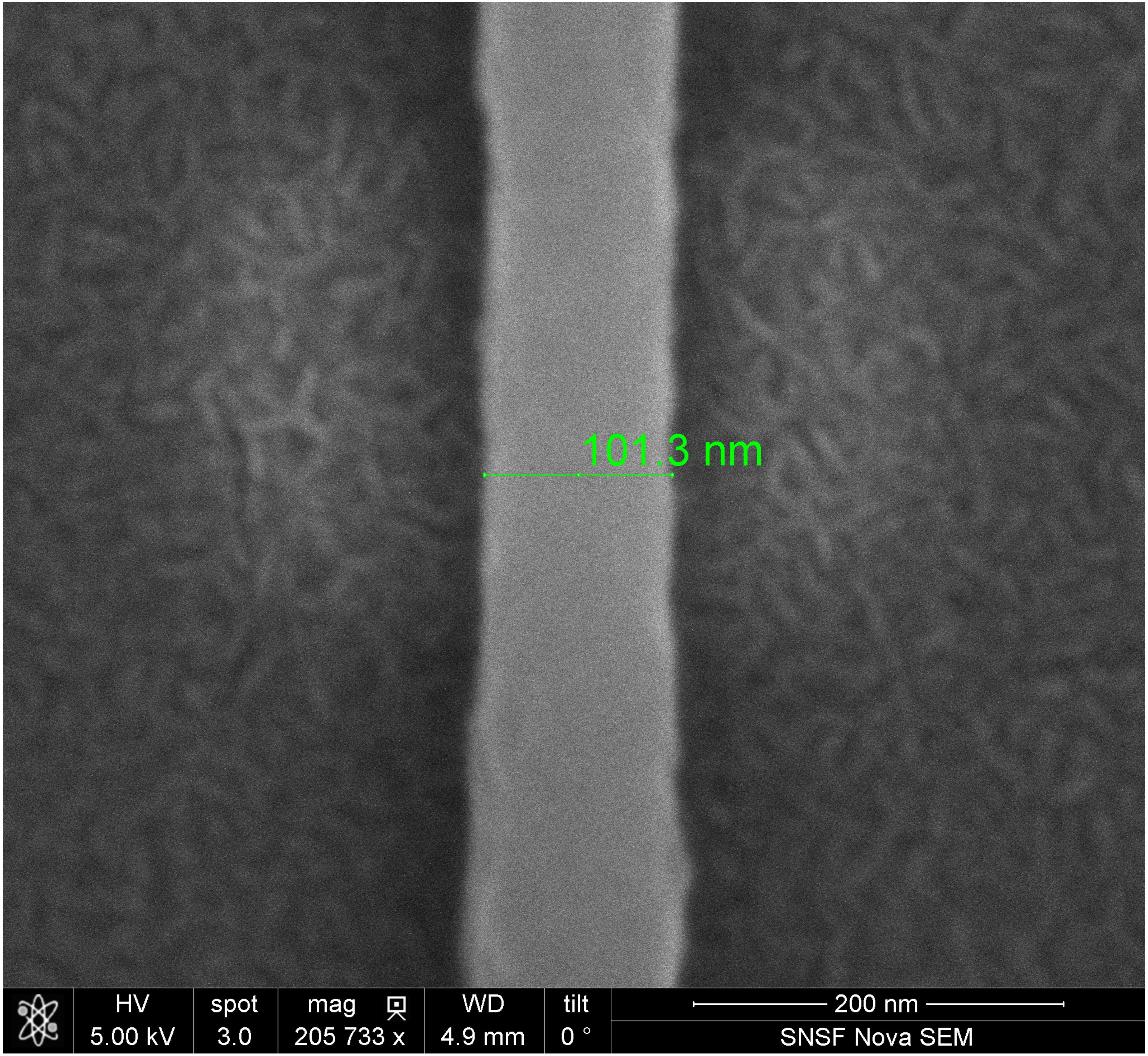
- First Exposure Dose
- Second Exposure Dose

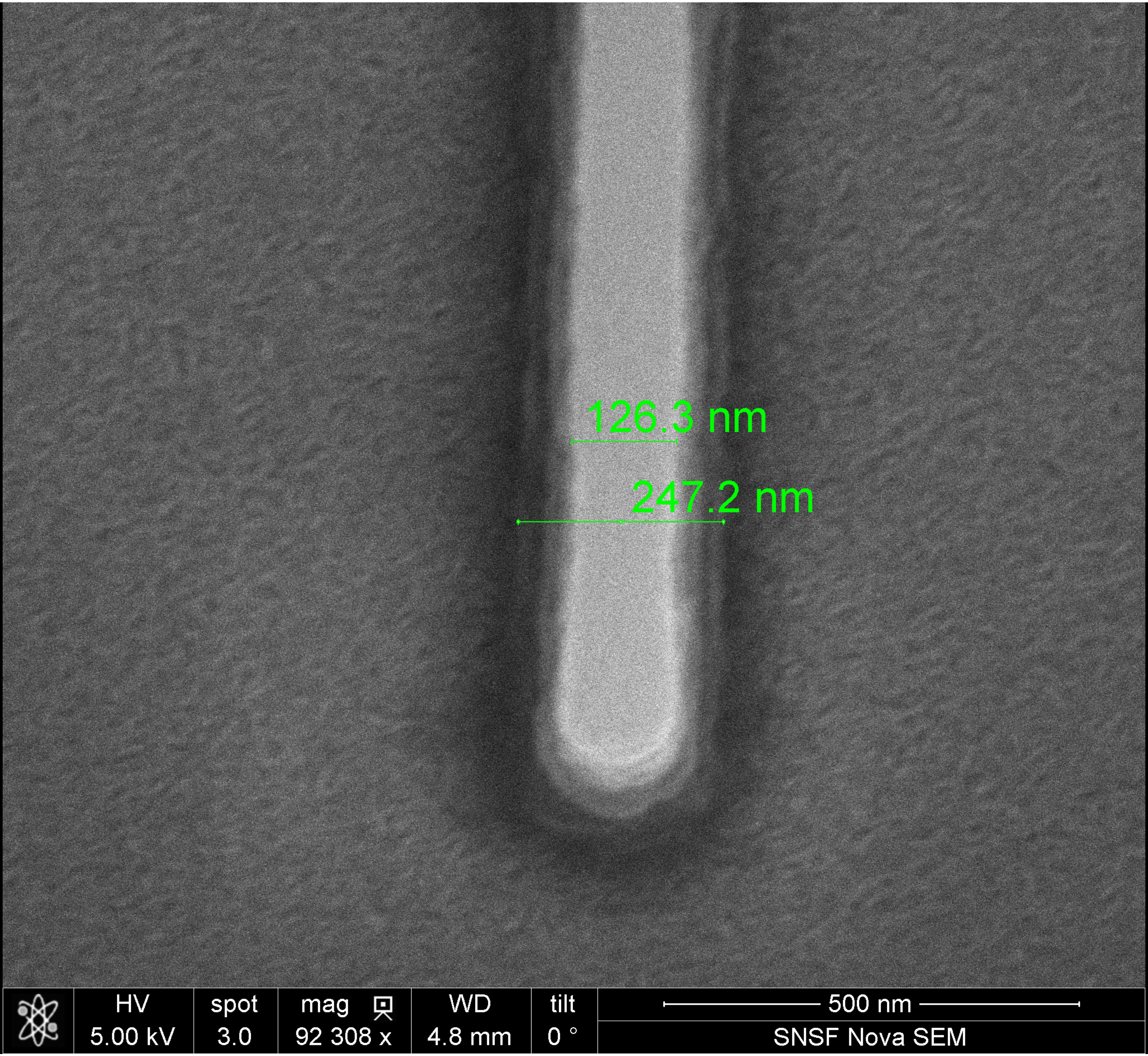
1)

- Offset – Matrix exp. each time
- Linewidth – optimized @ 500 nm
- Shift – 2)
- Surface Reflectivity

Single Resist - Coarse

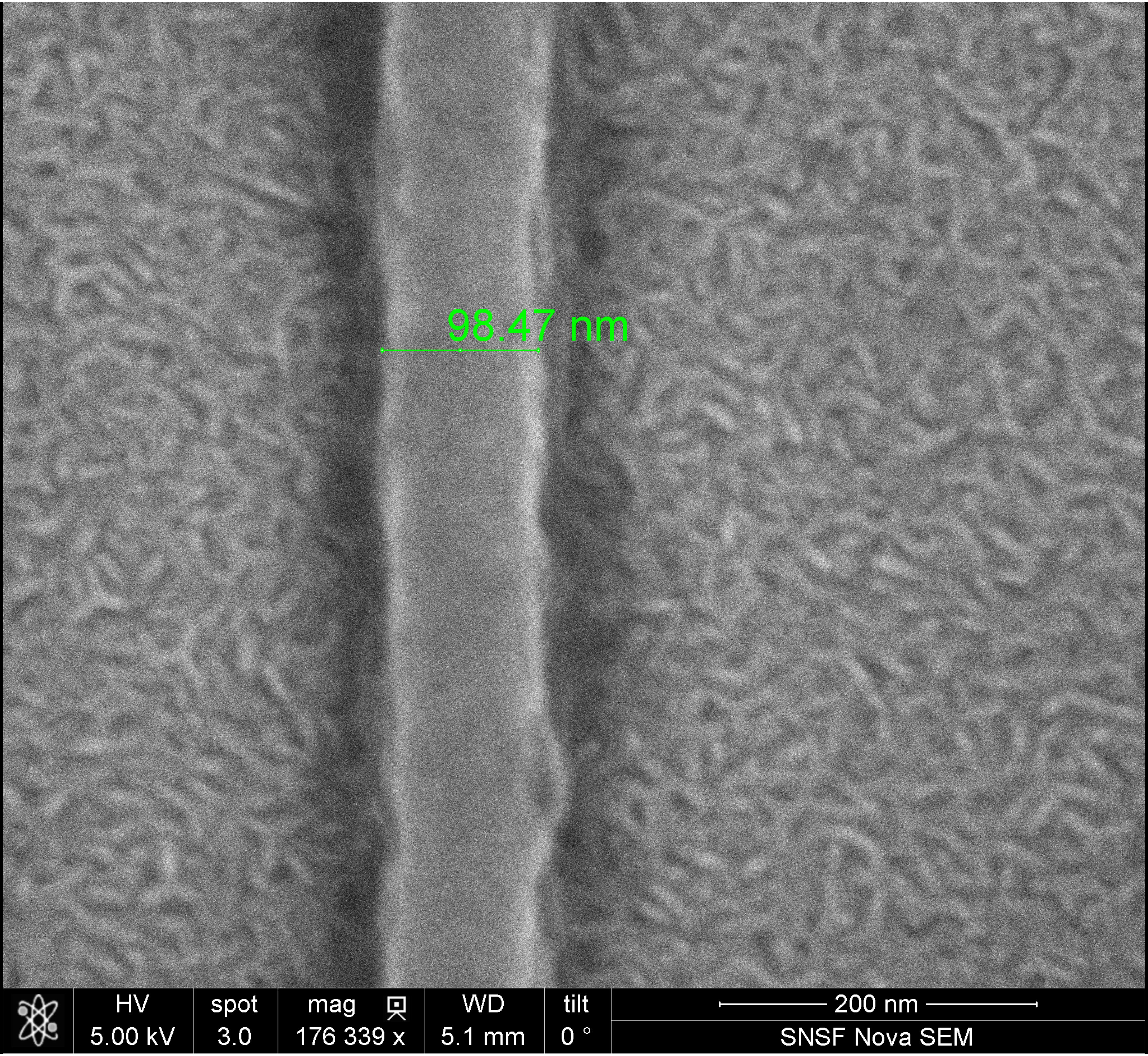
Exp | Shift	200 nm	250 nm	300 nm	350 nm	400 nm	450 nm	500 nm
2x 40 mJ	■						■
2x 60 mJ					202 / 282 (ns)	132 / 237 (vns)	135 / 167 (vns)
2x 80 mJ				158	101	NR	
2x 100 mJ			116 / 159 (ns)	125	NR		
2x 120 mJ			121	NR			
2x 140 mJ		109	NR				
2x 160 mJ	■	NR					■

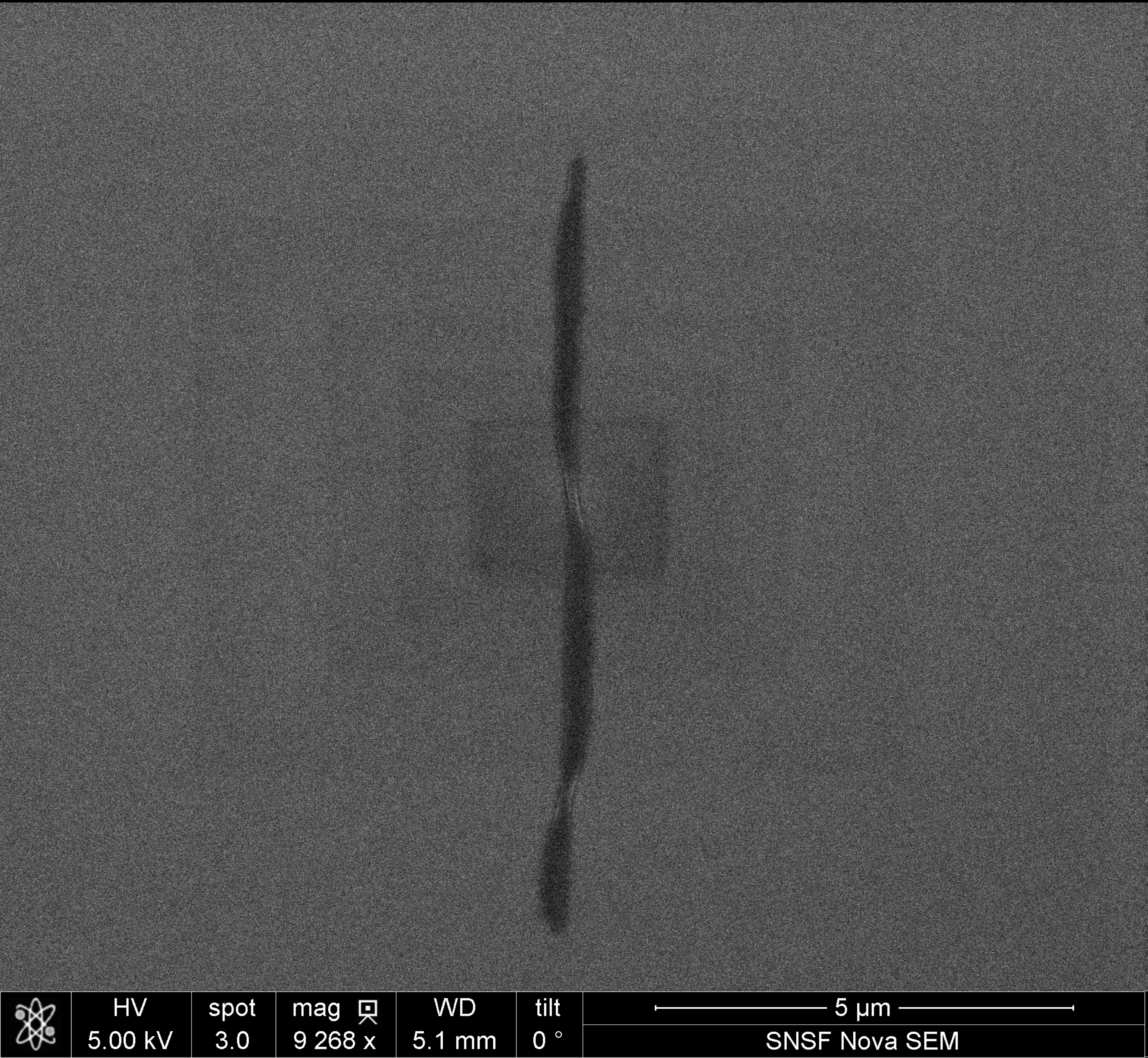

101.3 nm
HV
5.00 kV
spot
3.0
mag
205 733 x
WD
4.9 mm
tilt
0 °
200 nm
SNSF Nova SEM

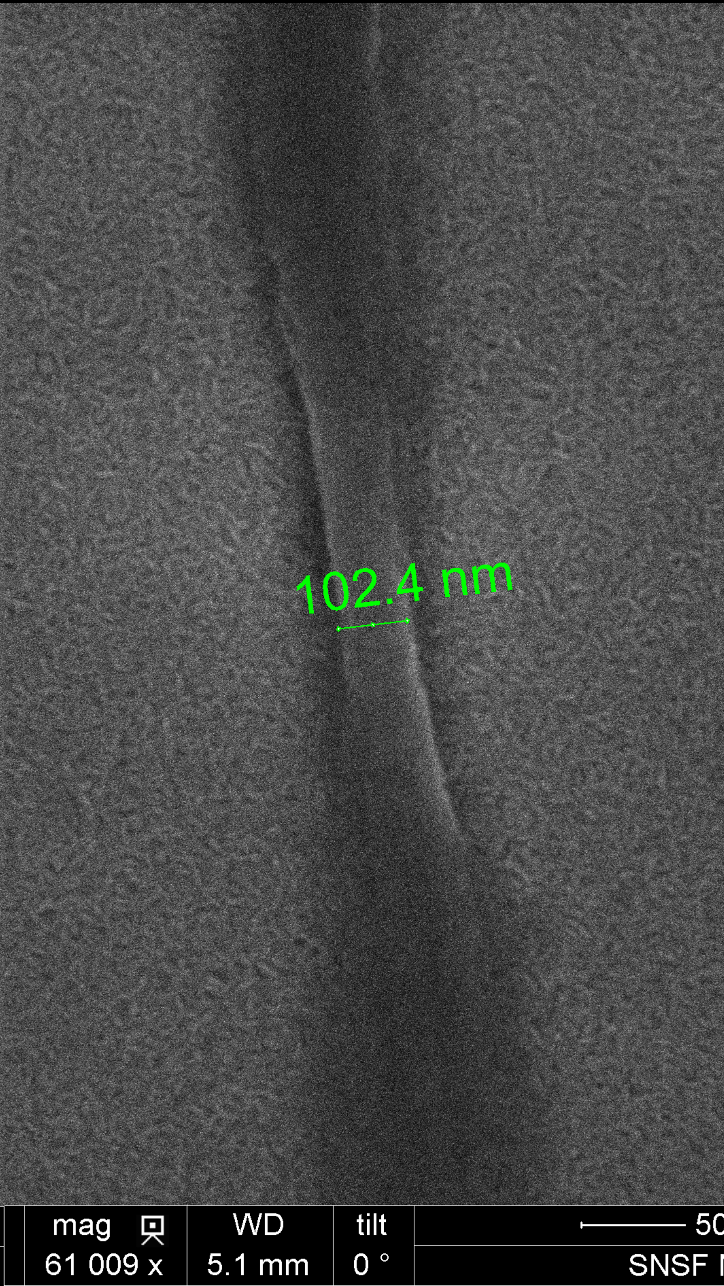

126.3 nm
247.2 nm
HV
5.00 kV
spot
3.0
mag
92 308 x
WD
4.8 mm
tilt
0 °
500 nm
SNSF Nova SEM

Single Resist - Fine

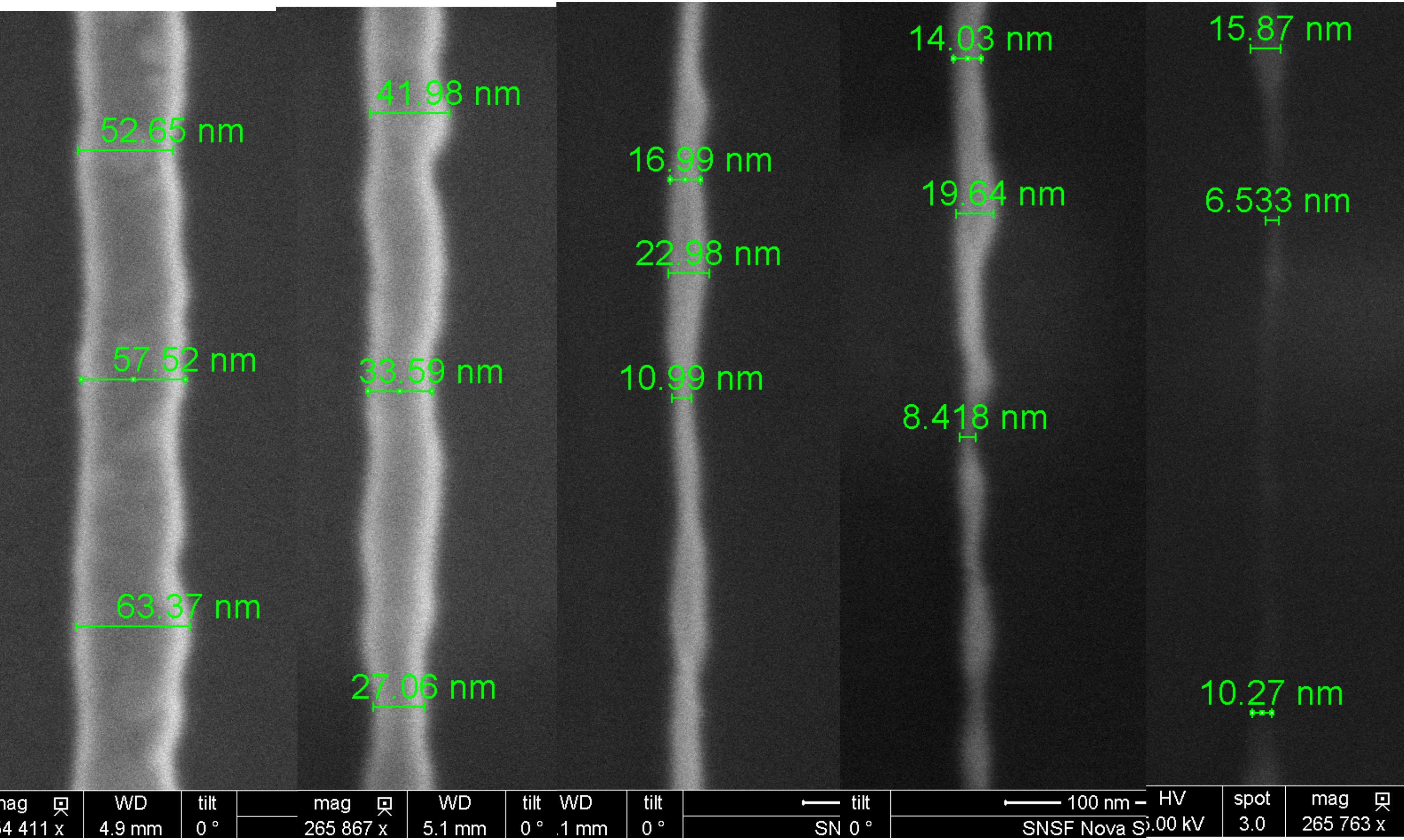
Exp | Shift	340 nm	360 nm	380 nm	400 nm	420 nm	440 nm	460 nm
2x 40 mJ	■						■
2x 50 mJ							
2x 60 mJ							145 / 289 (ns)
2x 70 mJ				141 / 202 (ns)	135 / 184 (ns)	118 / 150 (ns)	NR
2x 80 mJ					115	98	NR
2x 90 mJ				110	115 (BR)	NR	
2x 100 mJ	■		BR	BR	NR		■


98.47 nm
HV
5.00 kV
spot
3.0
mag
176 339 x
WD
5.1 mm
tilt
0 °
200 nm
SNSF Nova SEM

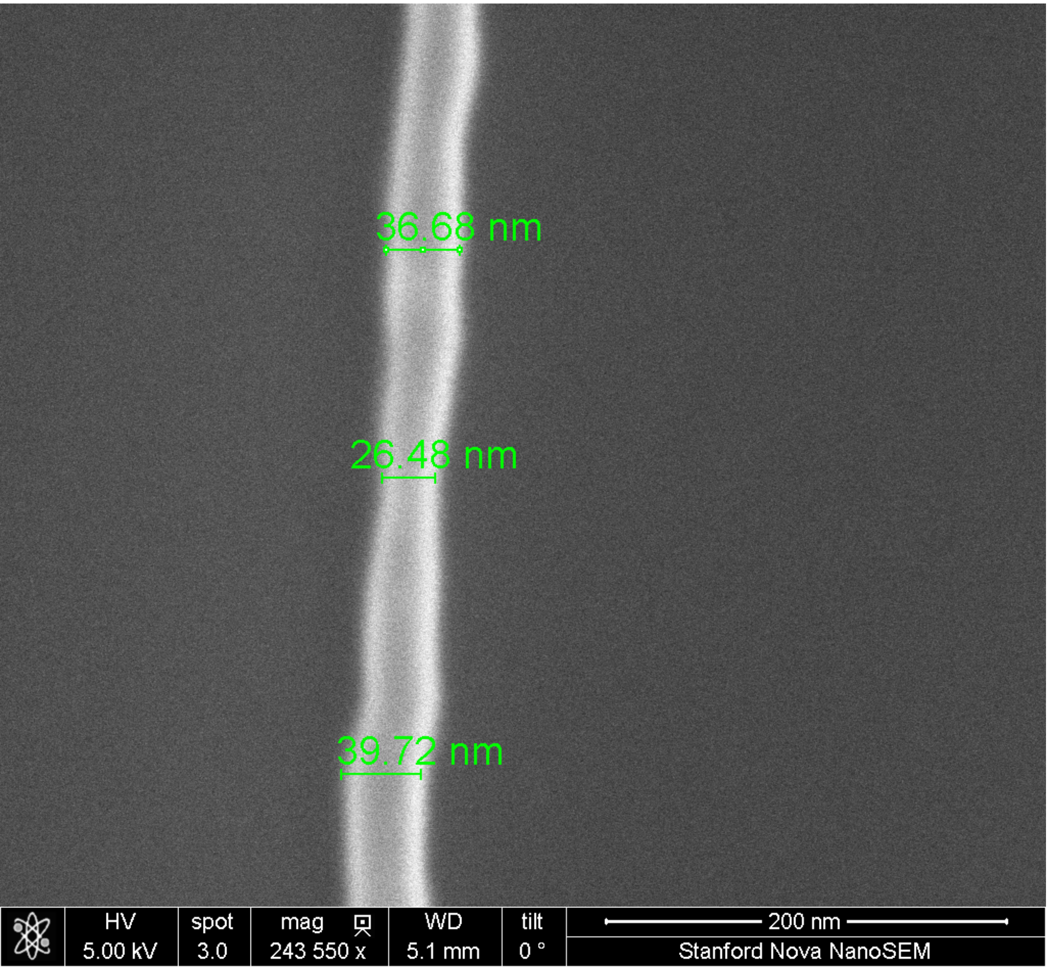

HV
5.00 kV
spot
3.0
mag
9 268 x
WD
5.1 mm
tilt
0 °
5 µm
SNSF Nova SEM

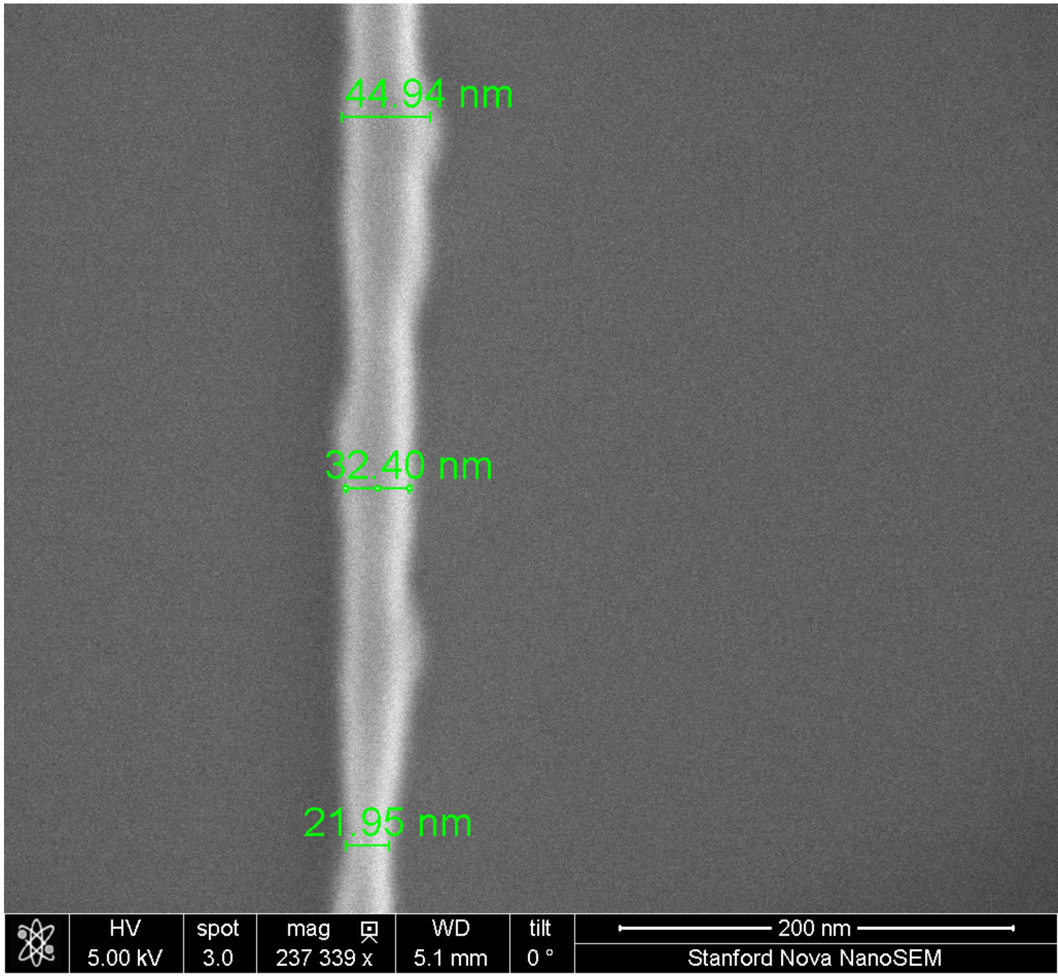

102.4 nm
mag
61 009 x
WD
5.1 mm
tilt
0 °
50
SNSF

Hard Mask


52.65 nm
57.52 nm
63.37 nm
41.98 nm
33.59 nm
27.06 nm
16.99 nm
22.98 nm
10.99 nm
14.03 nm
19.64 nm
8.418 nm
15.87 nm
6.533 nm
10.27 nm
nag
4 411 x
WD
4.9 mm
tilt
0 °
mag
265 867 x
WD
5.1 mm
tilt
0 °
WD
.1 mm
tilt
0 °
SN
tilt
0 °
100 nm
SNSF Nova S
HV
.00 kV
spot
3.0
mag
265 763 x

ccp-dep & p5000etch vs. LTO & PT-OX



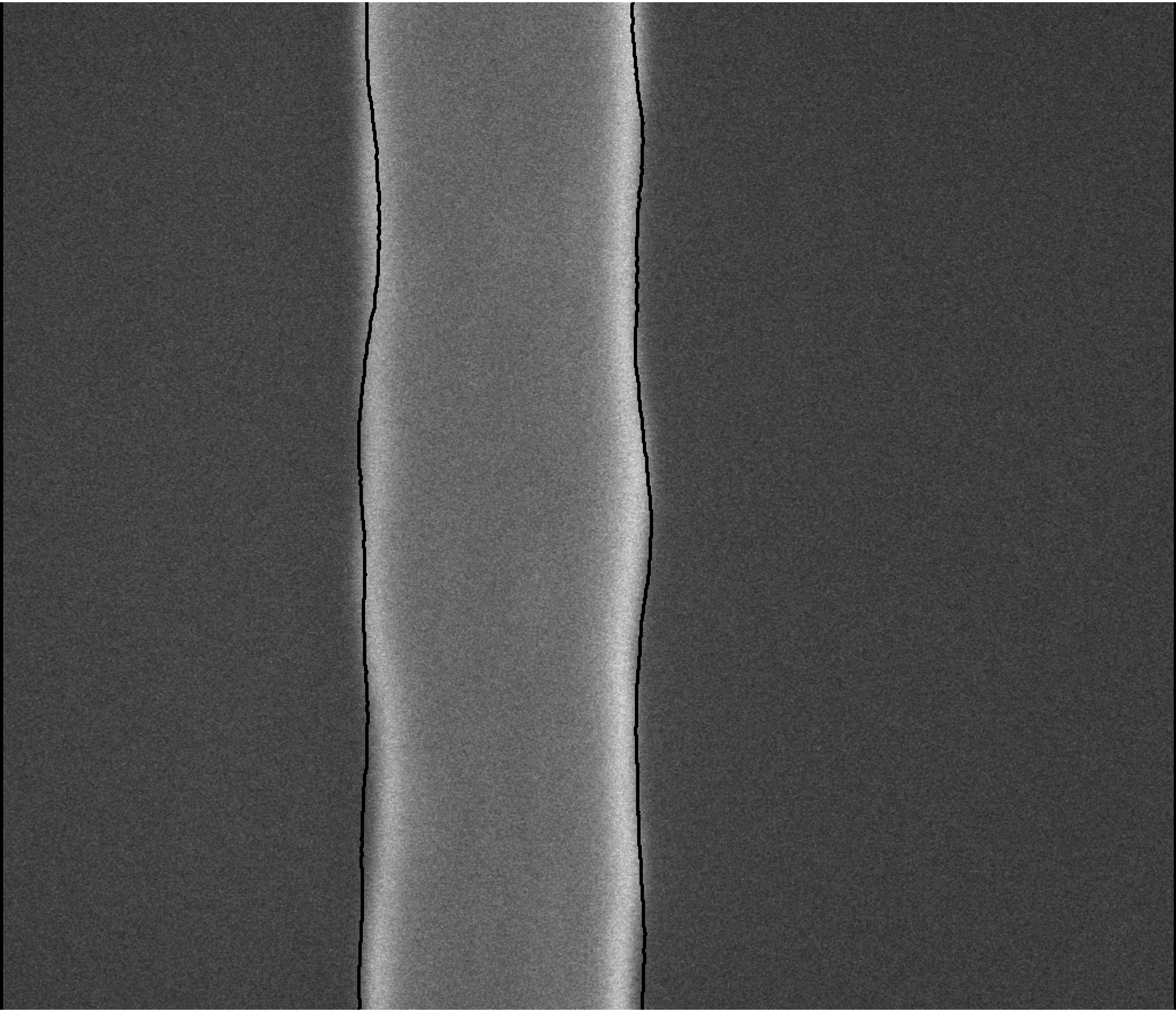


Hard Mask Variability testing

within each line
lines per shot
shots per parameter set
parameter sets per wafer
wafers
total

Hard Mask Variability testing

within each line	882 measurements
lines per shot	4 measurements
shots per parameter set	4 measurements
parameter sets per wafer	4 measurements
wafers	4 measurements
total	256 SEMs / 225,792 measurements

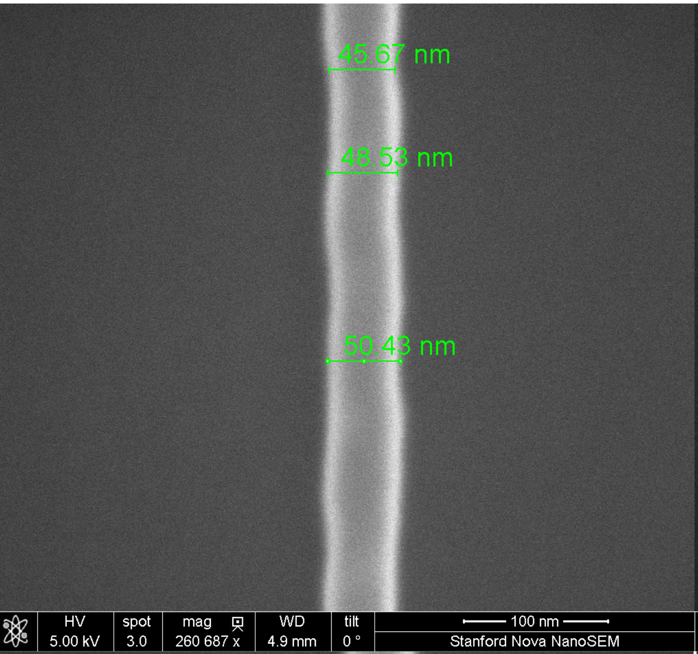


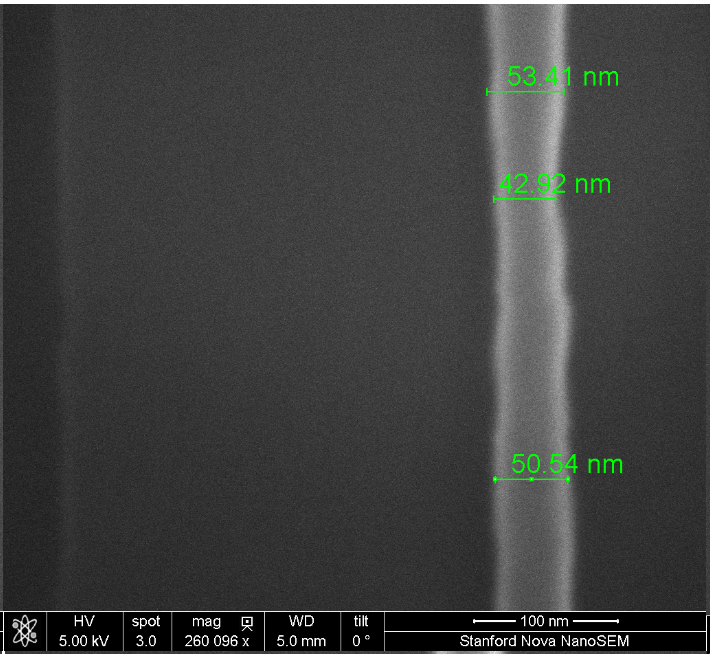
Hard Mask Variability testing

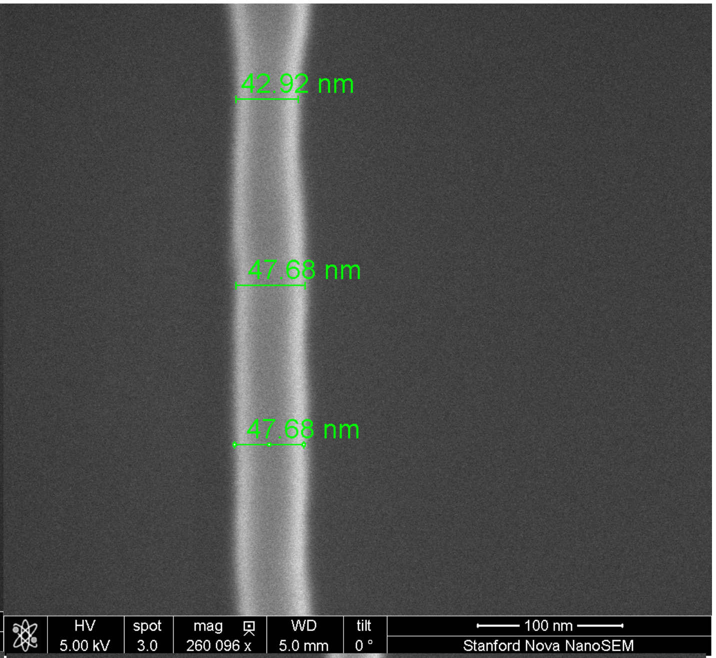
within each line	882 measurements
lines per shot	4 measurements
shots per parameter set	4 measurements
parameter sets per wafer	4 measurements
wafers	4 measurements
total	256 SEMs / 225,792 measurements

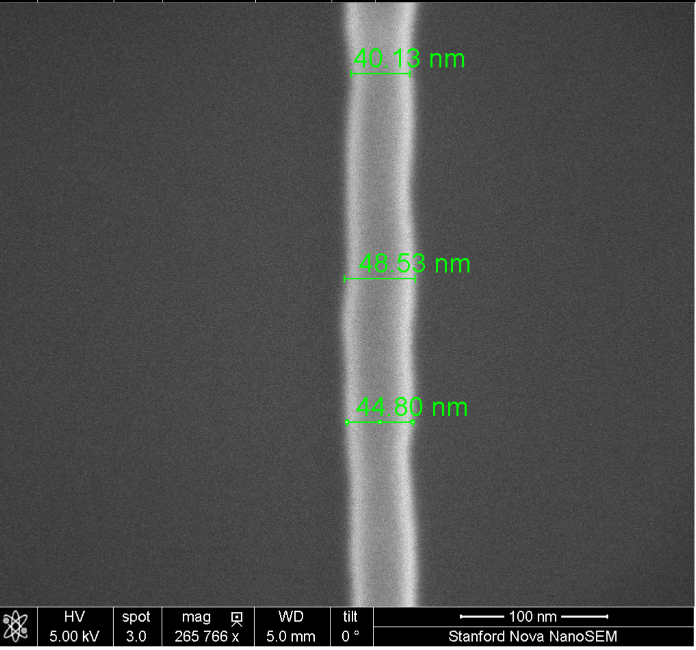
Within each line – RMS roughness	**3.0 nm**
Within each shot – standard deviation of line width (pre-averaged for each line)	**3.8 nm**
Within same settings – standard deviation of line width (pre-averaged for each shot)	**2.7 nm**
Between wafers – standard deviation of line width (pre-averaged for whole wafer)	**16 nm**

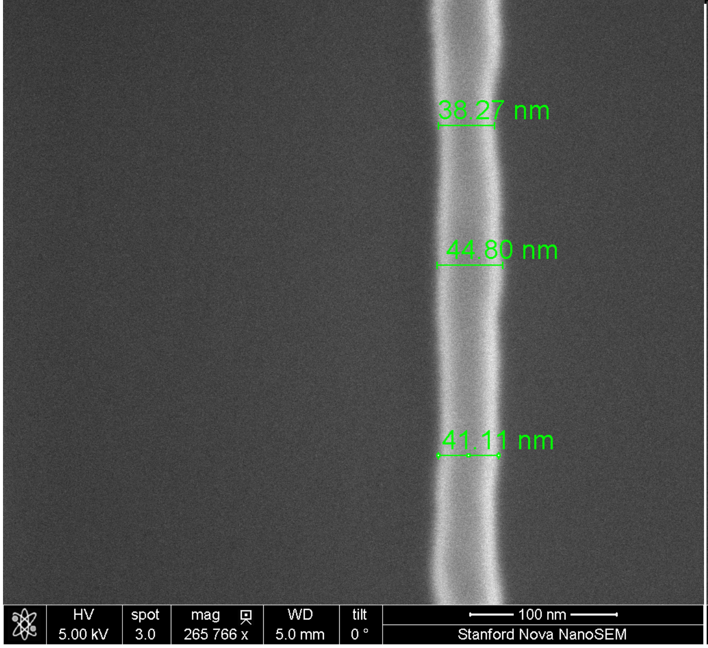
Intra- and inter-shot variability

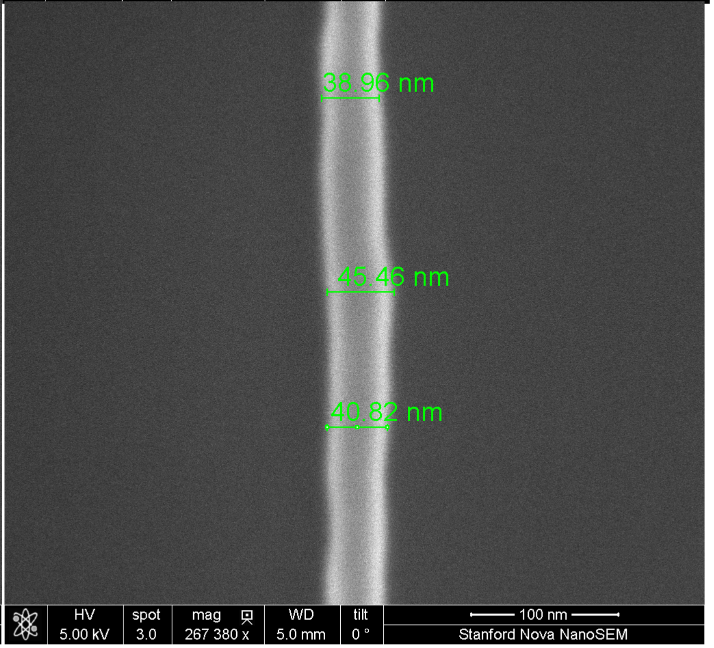












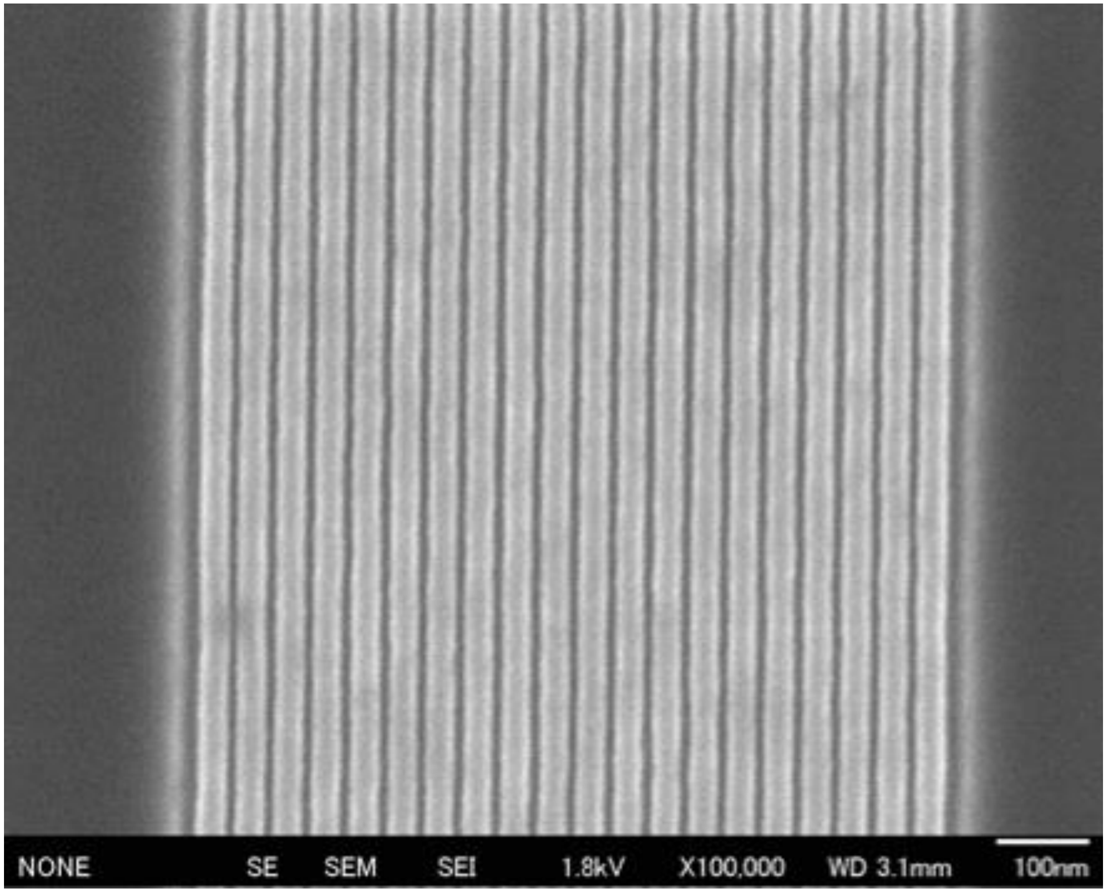
Electron Beam Lithography

- Electron beam lithography uses a finely focused beam of electrons to define patterns onto a polymer-coated wafer.

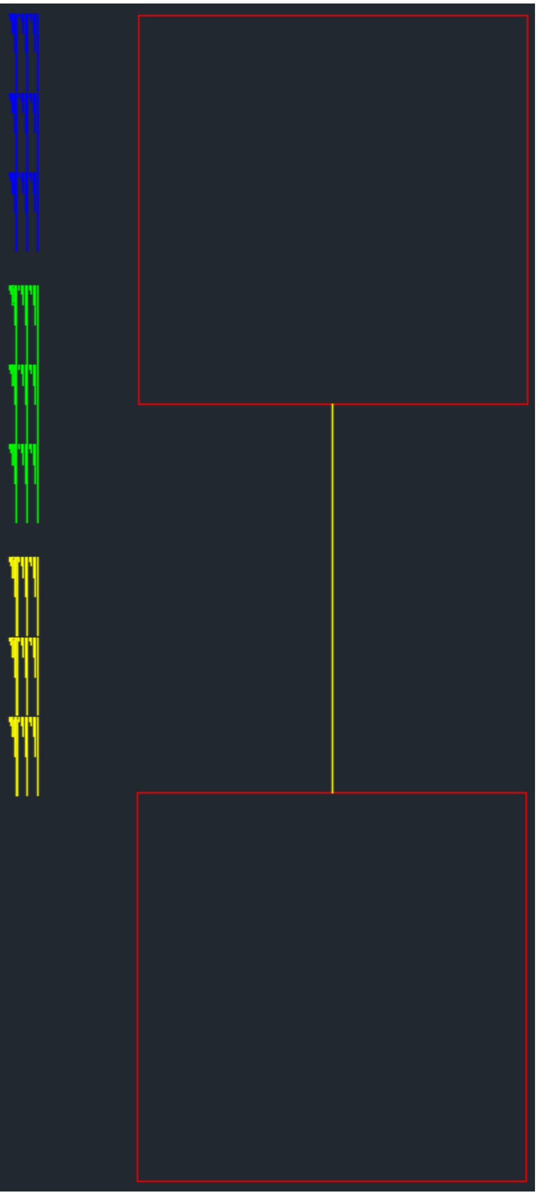
- No physical mask .

- Can work with small pieces.

- Can define beam diameter down to 2 nm.

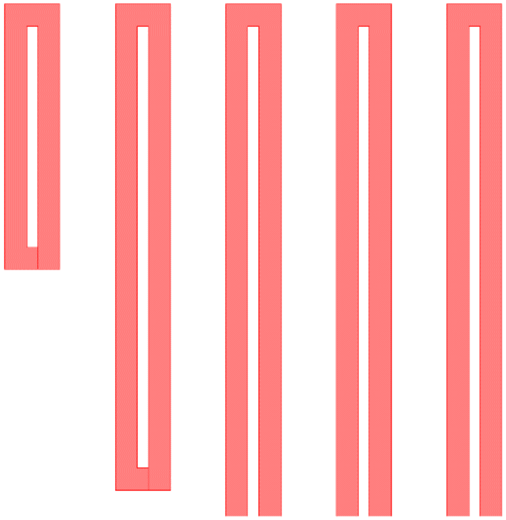


40 nm gratings with 8 nm lines by Rich Tiberio



Mask making

- Make sure it is a GDSII file, BEAMER software is not forgiving!
- If you have features of differing lengths, make sure they are on different layers
- If you typically make a pattern and use negative resist and want to make the same thing with a positive resist, you can use the same mask.



Resist selection

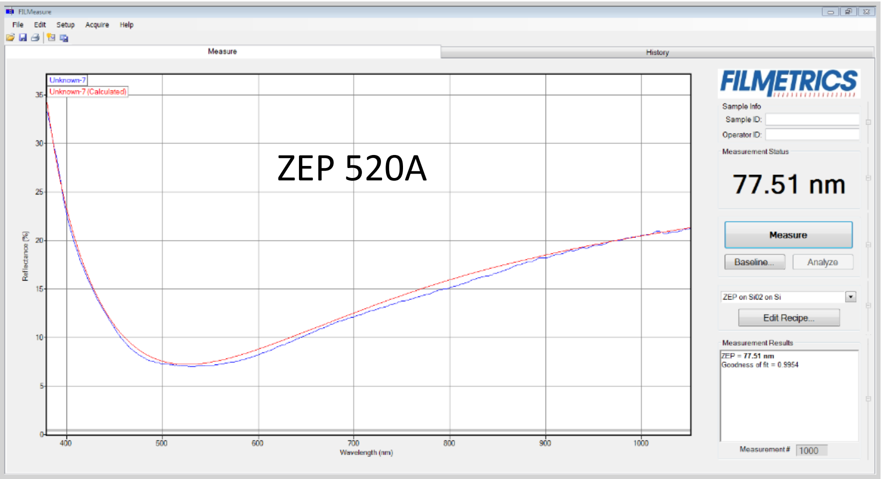
Resist	Tone	Dilution	Pre-Bake Temperature	Bake Time	Developer
Zep 520 A	+	1:2 Anisole	70 C	30 s	MIBK:IPA (1:1)
PMMA	+	-	180 C	90 s	MIBK:IPA (1:3)
*HSQ	-	-	80 C	60 s	MF 319
MaN-2405	-	1:2 Anisole	90 C	45 s	MF 319

All resist were spun with recipe 8 (5s 505 rpm, 40 s 4500 rpm) in SNC on spin coater

* HSQ is very expensive ($1200/250mL) and not available in SNC

After spinning resist

- Filmetrics to get thickness measurement, spectral reflection based method



ZEP 520A

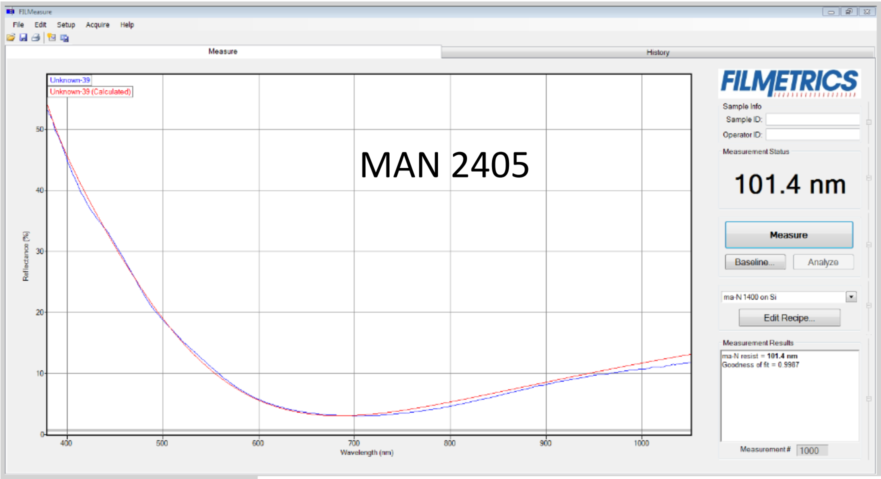
Sample Info
Sample ID:
Operator ID:

Measurement Status
77.51 nm

ZEP on Si02 on Si

Measurement Results
ZEP = **77.51 nm**
Goodness of fit = 0.9954

Measurement # 1000



MAN 2405

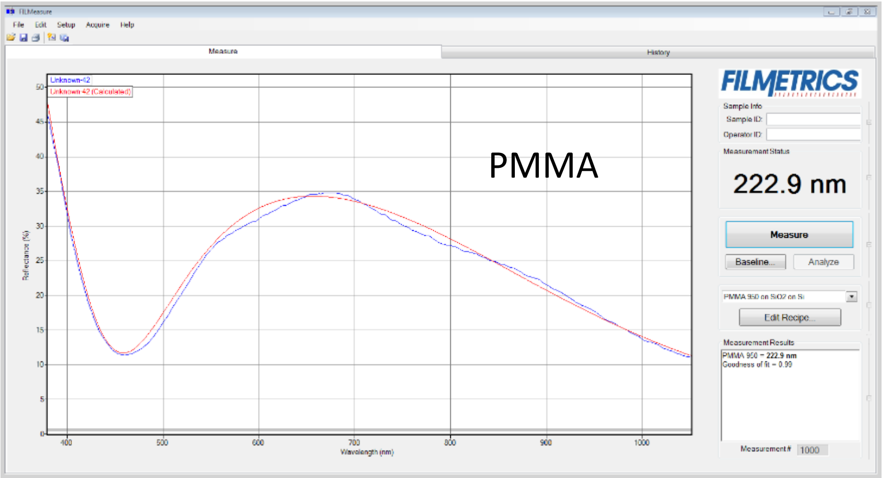
Sample Info
Sample ID:
Operator ID:

Measurement Status
101.4 nm

ma-N 1400 on Si

Measurement Results
ma-N resist = **101.4 nm**
Goodness of fit = 0.9987

Measurement # 1000



PMMA

Sample Info
Sample ID:
Operator ID:

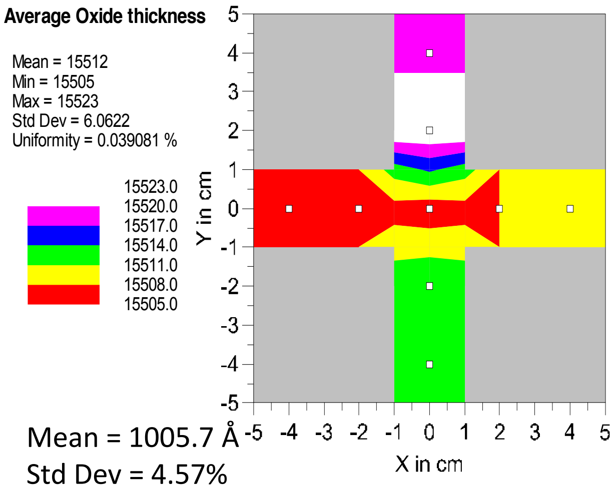
Measurement Status
222.9 nm

PMMA 950 on SiO2 on Si

Measurement Results
PMMA 950 = **222.9 nm**
Goodness of fit = 0.99

Measurement # 1000

HSQ



Mean = 1005.7 Å
Std Dev = 4.57%

Determining dosage

- D = (I*t)/A, where D = dose (µC/cm^2) I = current (A) t = time (sec) A = exposure area (cm^2)
- In the Jeol JBX-6300FS, Rich uses the following

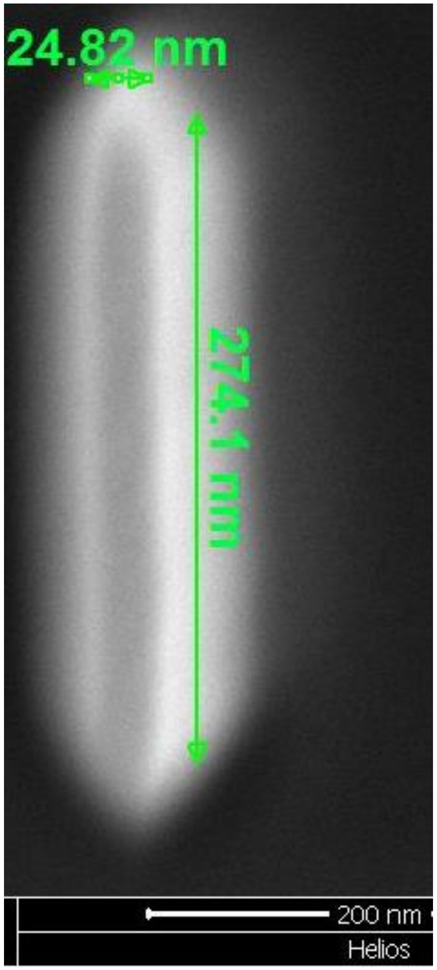
4000*I(nA)/(step size (nm))^2 = D (µC/cm^2)

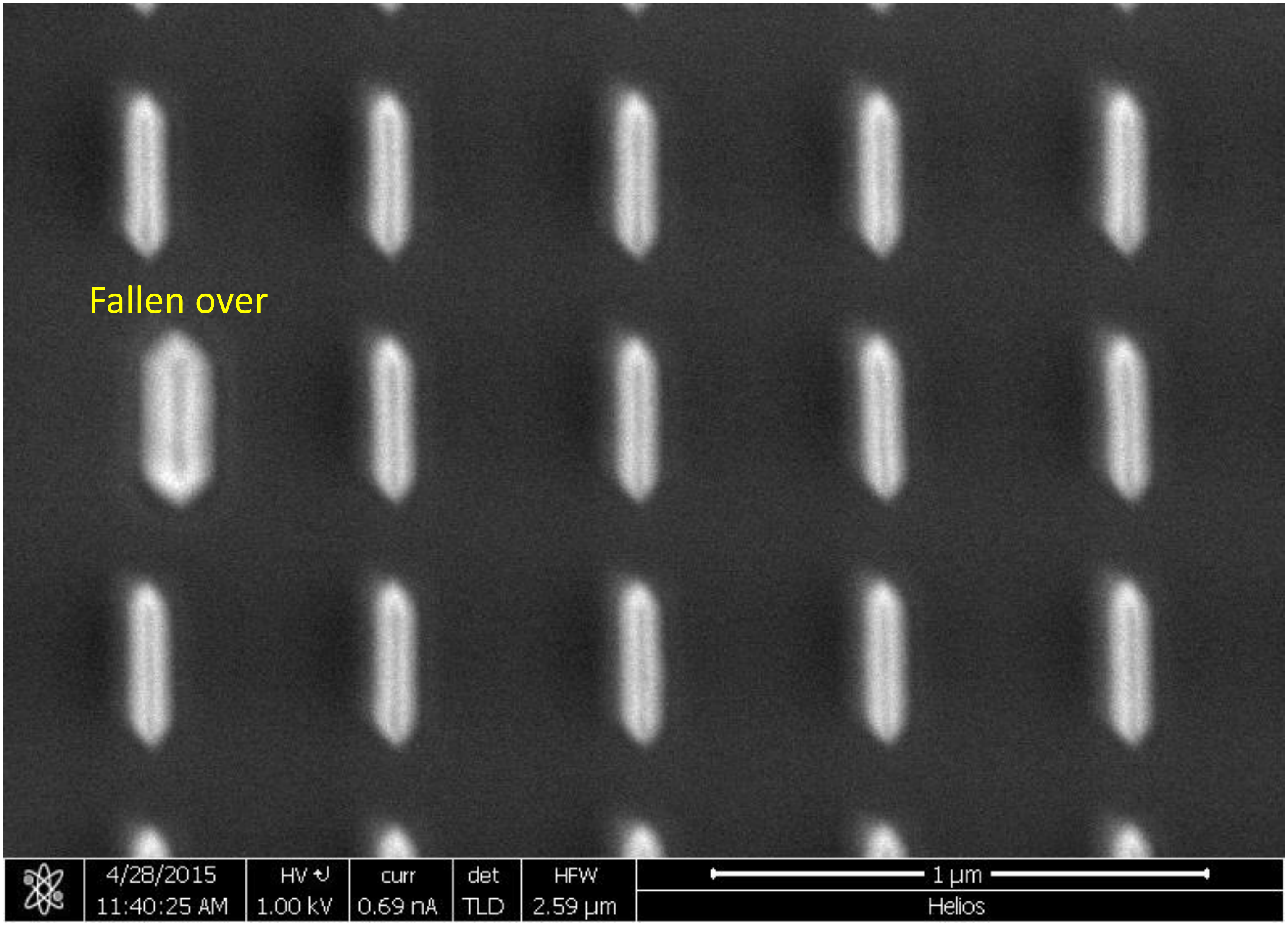
- The smaller the feature size, the higher the base dose. Choose aperture and current appropriately.
- Depending on resist, the base dose needs to be changed.

In our case - Zep 520A, PMMA, and MaN-2405 required base does of 400 µC/cm^2 but HSQ required 1000 µC/cm^2.

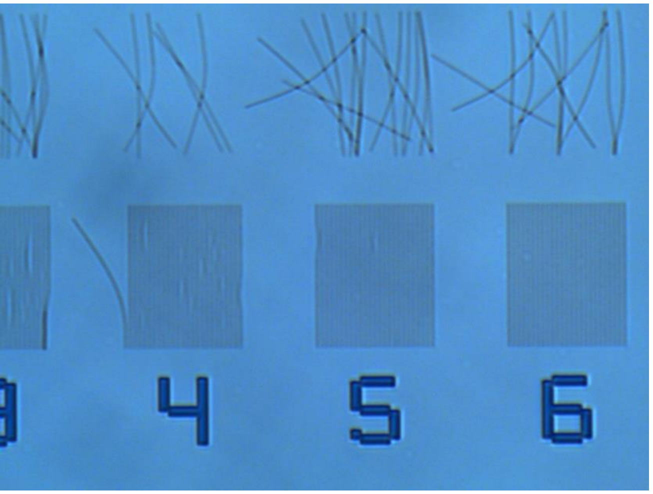
Limit MaN-2405 resist 10 x 300 nm

Nearly 4:1 aspect ratio

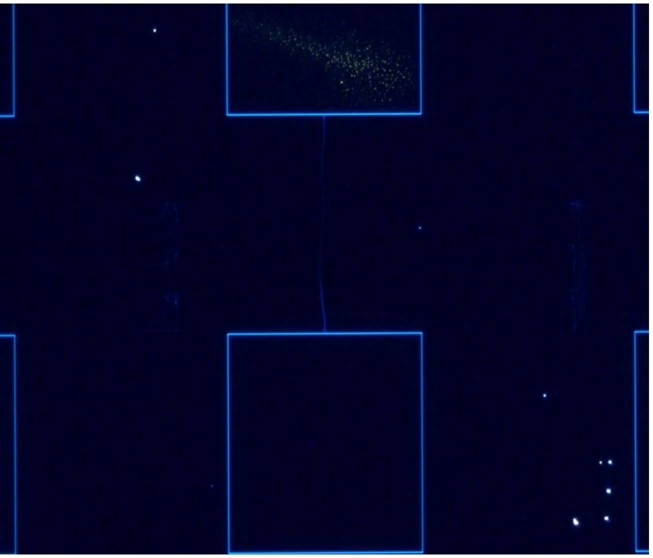




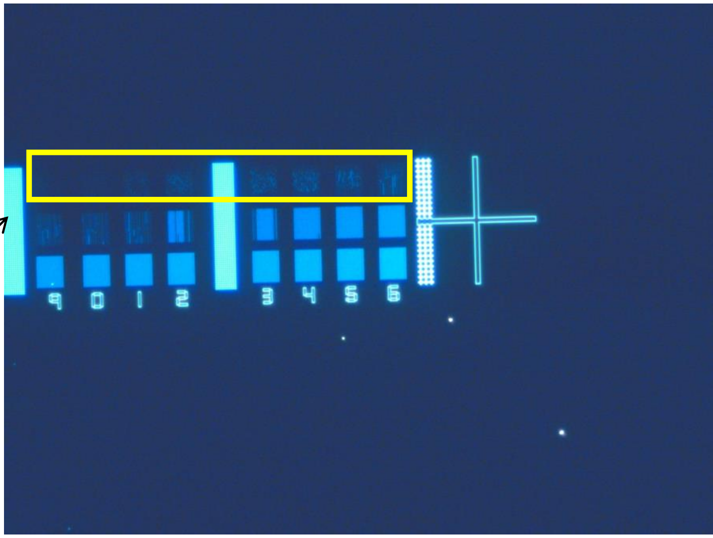
Better dosage and adhesion after using SUPASS



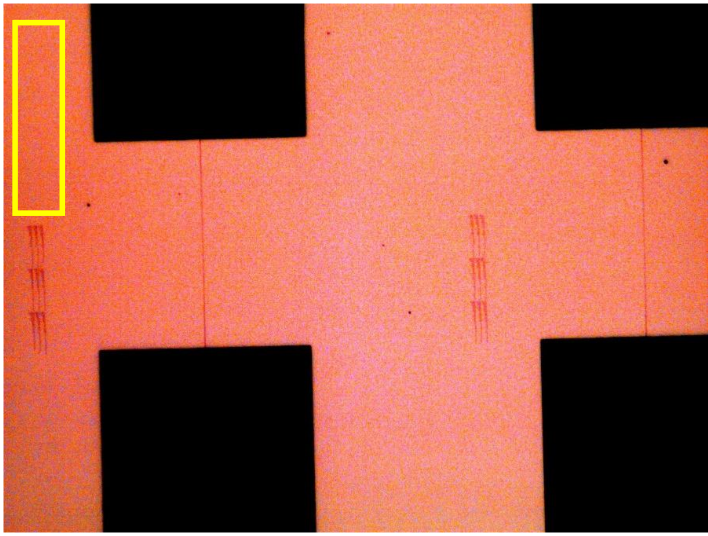
No SURPASS!



MaN-2405

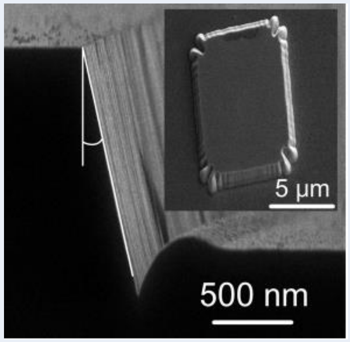
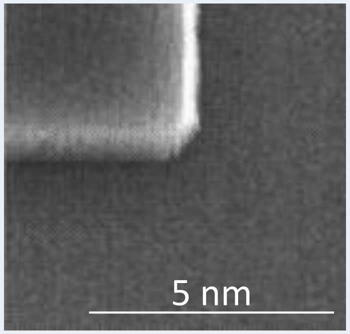
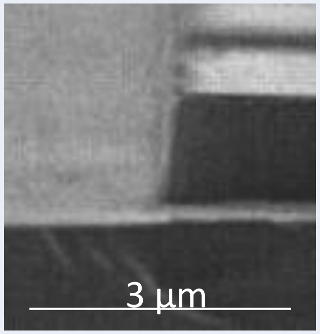


Needs higher dose, but overall better when comparing to previous figures on left



InSb Reactive Ion Etching

- Two reported process for InSb etching

Process	BCl_3, Cl_2 based	CH_4, H_2 based
Volatile Product	$InCl_x$ (>240°C)	SbH_3
Side wall profile	Sloped	Sloped, Barreled
Roughness (RMS)	0.25 – 33 (nm)	10 – 40 (nm)
SEM Images	 	 
Problem	$InCl_x$ can cause electrode to short	

Journal of Vacuum Science & Technology A 27, 681 (2009)
Microelectronic Engineering 98, 222 (2012)
Applied Surface Science 143, 183 (1999)

JMP Setup

- Etch Conditions
  - Photoresist (SPR955-.7) mask
  - Substrate kept at 20 ^{o}C
  - ICP power set to 600W
  - RF power set to 100W
  - Chamber pressure set to 20 mTorr
  - Variables
    1. CH_4 flowrate (15 – 45 sccm)
    2. H_2 flowrate (4 – 10 sccm)
    3. Ar flow (40 – 80 sccm)

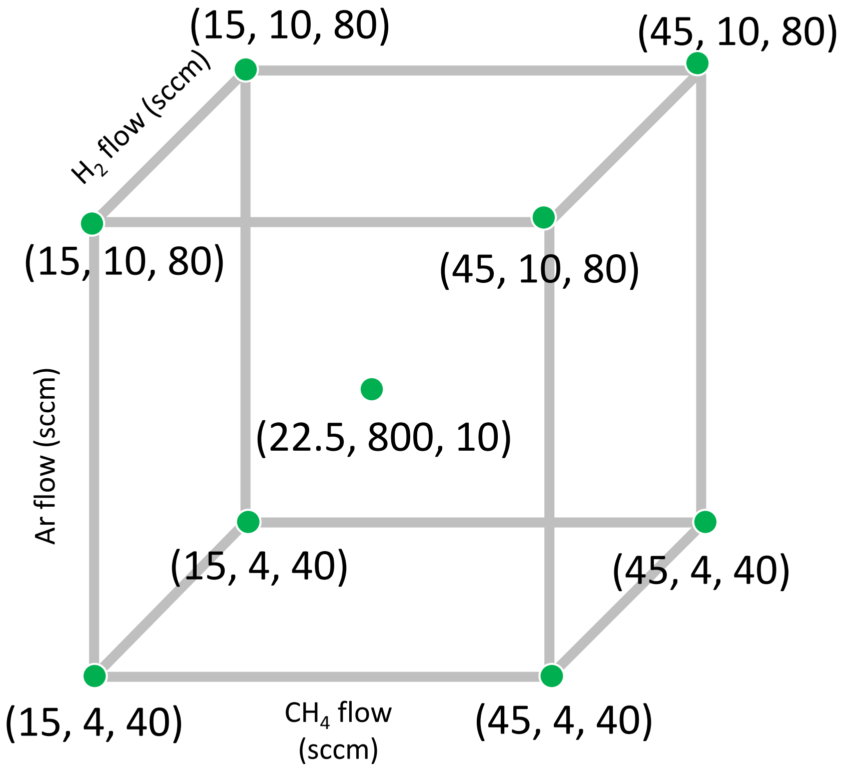


Fig.1 Jump variable setting

JMP Results

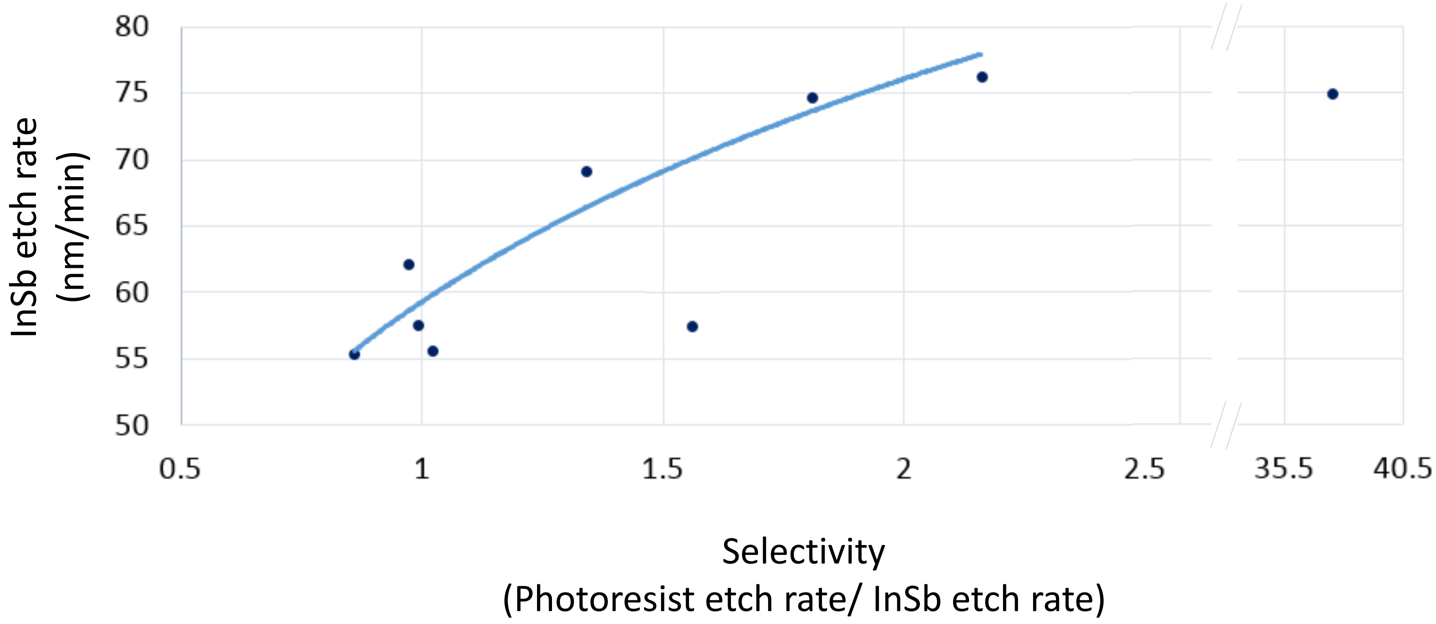
- Samples were etched for 3 min

	Factor			Results			
Sample #	CH4 flow (sccm)	H2 flow (sccm)	Ar flow (sccm)	Roughness (nm)	Angle (deg)	InSb Etch rate (nm/min)	Selectivity
1	+	+	+	18.4	60	57.6	1.56
2	+	+	-	31.6	90	75	37.48
3	+	-	+	23.4	90	76.3	2.16
4	+	-	-	32.4	72	74.7	1.81
5	-	+	+	27.6	74.3	57.6	0.99
6	-	+	-	26.8	70.2	69.1	1.34
7	-	-	+	20.2	72.2	62.2	0.97
8	-	-	-	32	75.5	55.4	0.86
9	0	0	0	27.1	90	55.7	1.02

CH_4 flowrate (15 – 45 sccm), H_2 flowrate (4 – 10 sccm), Ar flow (40 – 80 sccm)

Etching

- Based on the etch rate of InSb, selectivity can be estimated



$$\boxed{InSb\ etch\ rate\ =\ 24.2\ ln(Selectivity)\ +\ 59.3}$$

JMP Results

- No strong correlation was found between factors, roughness, angle, etch rate and selectivity

	Factor			Results			
Sample #	CH4 flow (sccm)	H2 flow (sccm)	Ar flow (sccm)	Roughness (nm)	Angle (deg)	InSb Etch rate (nm/min)	Selectivity
1	+	+	+	18.4	60	57.6	1.56
2	+	+	-	31.6	90	75	37.48
3	+	-	+	23.4	90	76.3	2.16
4	+	-	-	32.4	72	74.7	1.81
5	-	+	+	27.6	74.3	57.6	0.99
6	-	+	-	26.8	70.2	69.1	1.34
7	-	-	+	20.2	72.2	62.2	0.97
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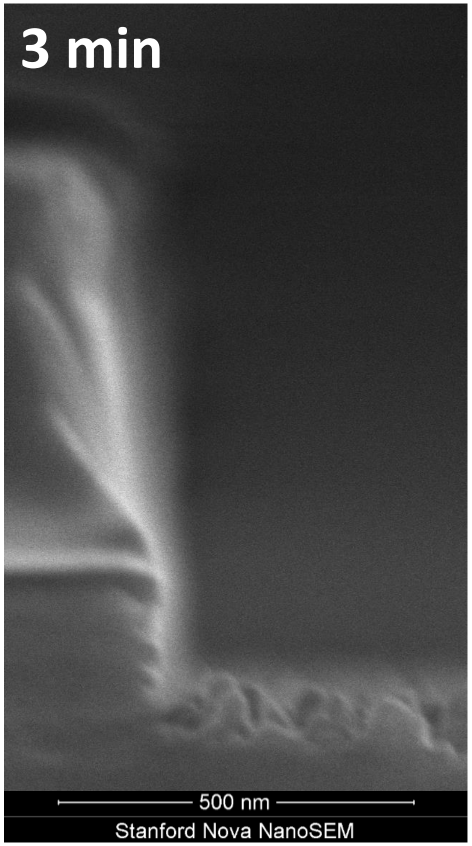
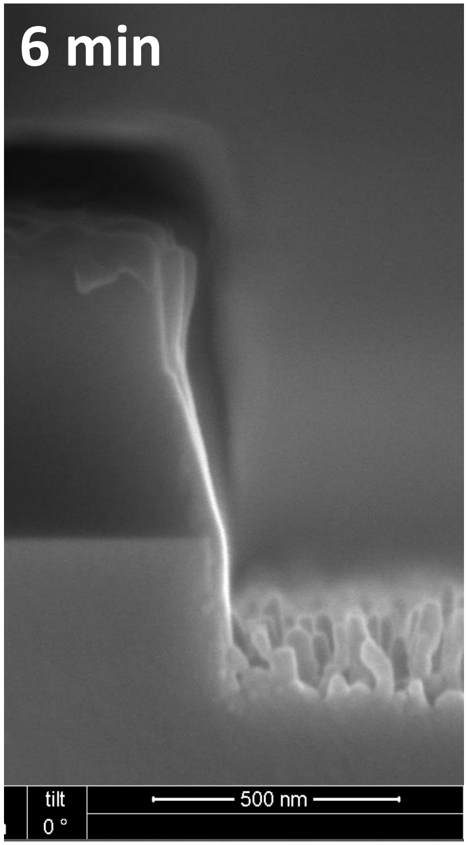
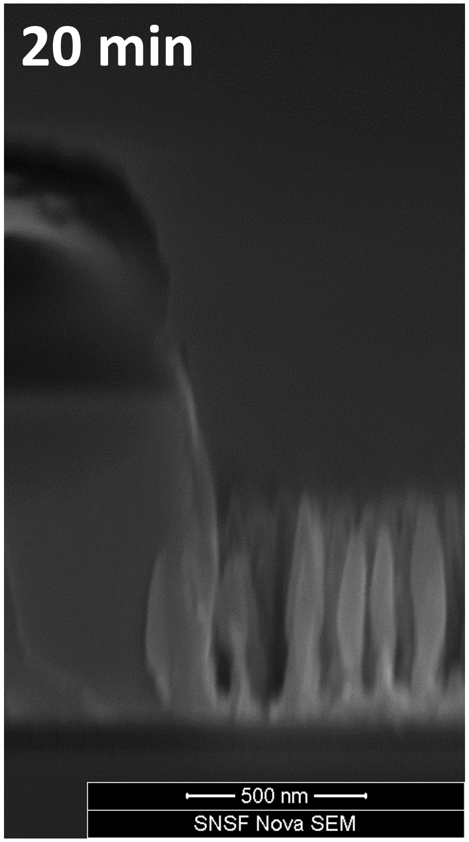
CH_4 flowrate (15 – 45 sccm), H_2 flowrate (4 – 10 sccm), Ar flow (40 – 80 sccm)

Surface morphology v.s. Etch time

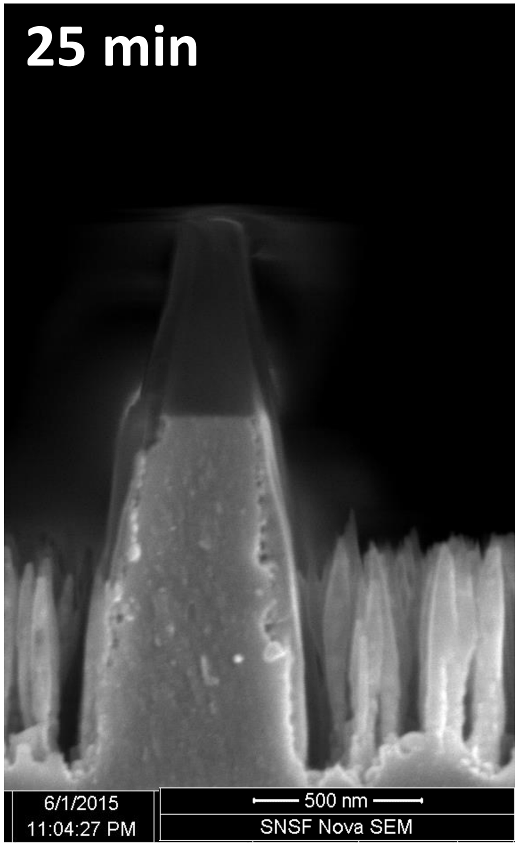




- Grass shows up

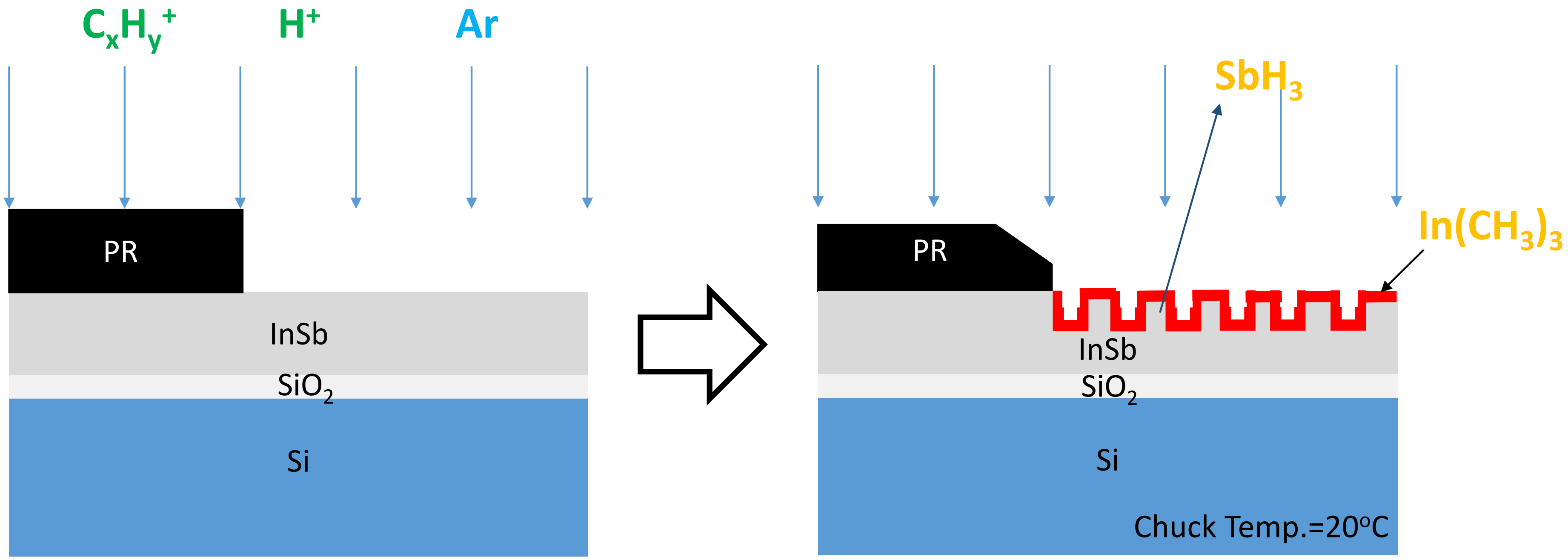


- Instead of being etched, nanowires grow



What is Happening?

- Why are we seeing nanowires?



$CH5^+$, H^+: Chemical Etching
Ar: Physical Etching

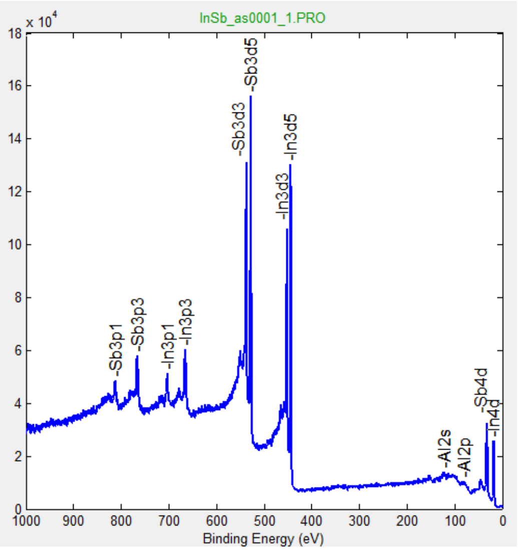
SbH_3: Boiling Temp. = -17°C
$In(CH_3)_3$: Sublimation Temp. = 50°C

Weidlein.J (2013) In Organoindium Compounds, Springer

What is Happening?

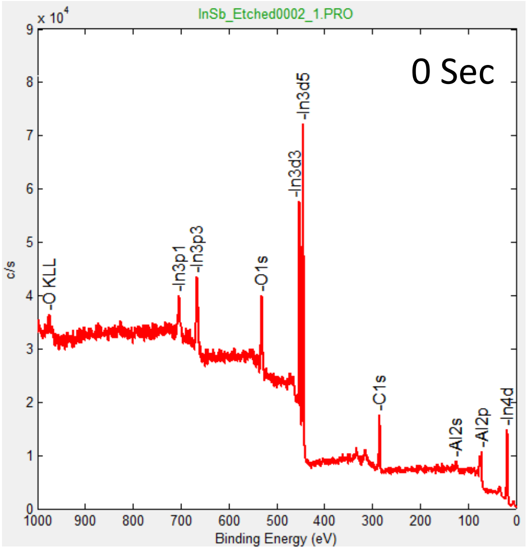
- XPS data for InSb surface after etching for 6 min

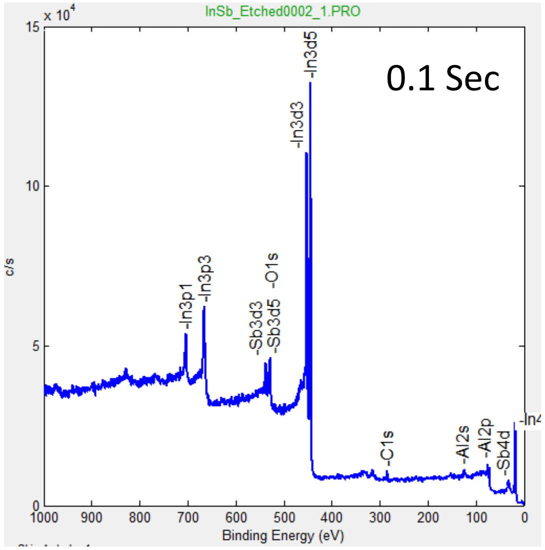
As Deposited



As Deposited InSb	
C1s	0 %
In3d5	50.40 %
Sb3d5	49.60 %

RIE for 6 min





Sputtering time	0 sec	0.1 sec
C1s	64.92 %	**20.54 %**
In3d5	35.08 %	70.13 %
Sb3d5	**0 %**	**9.33 %**

What is Happening?

- This explains….
  1. Why there are no visible relationship between JMP samples
     - Many factors are effecting the etch
  2. Why CH4/H2/Ar (+/-/+) flow give relatively smooth surface
     - Balance between chemical etching and physical etching
  3. Why higher ICP power doesn't yield higher InSb etch rate

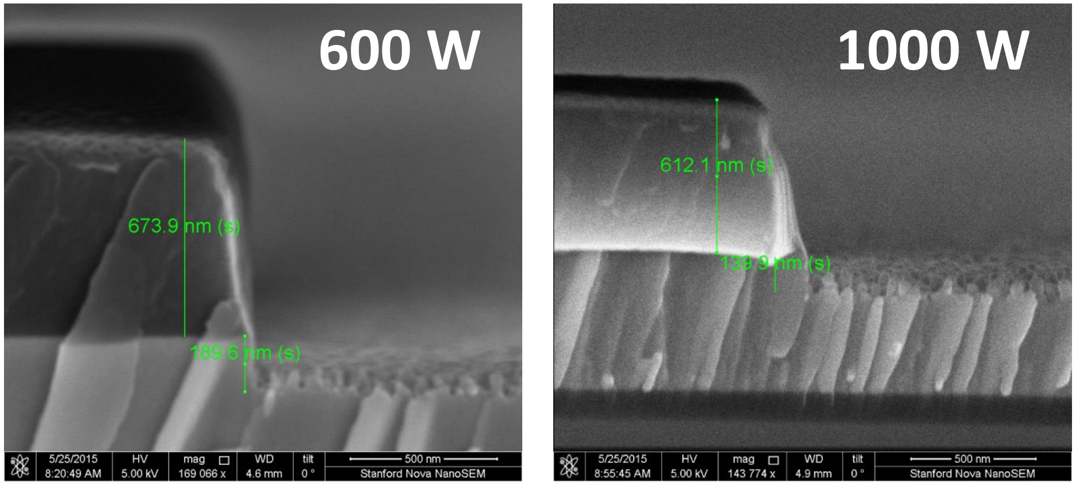


Fig.2 Comparison between ICP power of 600W and 1000W

Future Work

- Steps to smooth InSb etching
  1. Heat the chuck temperature higher than 50°C (If possible)
  2. Increase the RF power if the side walls are sloped or barreled
  3. Balance the chemical etching and physical etching
  4. Find the optimum point for etching

- E-beam resist samples waiting to be measured for selectivity

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- J
- Mahnaz, Usha, Rich
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- 412 class