



THE GEORGE WASHINGTON UNIVERSITY PHYSICS DEPARTMENT

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NPSAM variety in 6 fibers

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Abstract

We deposited gold nanoparticles on 6 fibers simultaneously. The goal was to see if the variation in AuNP deposition rate would disappear when fibers were batch functionalized. If so, that means the rate of formation varies because of some external problem, for example the amount of time fibers spend in air prior to AuNP deposition, or the amount of time they spend in water prior to AuNP formation. It turns out that within the six fibers, a range of 20% coverage variability was observed, with the least coated fiber being approximately 10% covered, and the most being about 30%. The fibers were all cleaved perfectly and all exhibited clean AuNP formation. All the images were clear except for fiber 1 perhaps, which had contamination likely due to sitting out on the grid for a while while I worked and soldered nearby. These results are at a glance commensurate with the variability in rates I typically observed functionalizing one at a time. This work is complimentary to some results last fall where we explored various ways to control formation upper limit, including PH changes and AuNP concentration. This is intended to be incorporated into a discussion section in an outlined paper skeleton which I'll distribute soon for feedback and brainstorming.

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1 Introduction

Because had good fibers with new silanes, decided was good time to do this experiment. Idea is to simultaneously run 6 fibers in NPSAM solution; 1 in optical setup. Then transfer all to ethanol immediately after the fiber in setup gets to pre-set level. Then, sacrifice all and compare them on via SEM to measure the differences for batch-functionalization. Will use this in a later paper; already outlined.

2 Experimental

Prep. Date	7/10/2014
Prepped by	Adam/Clay
Fiber type	GIF-625
# Fibers	6
HF Etching time	N/A
Silane type	TMSDE
Annealing time/temp	30min 125°C

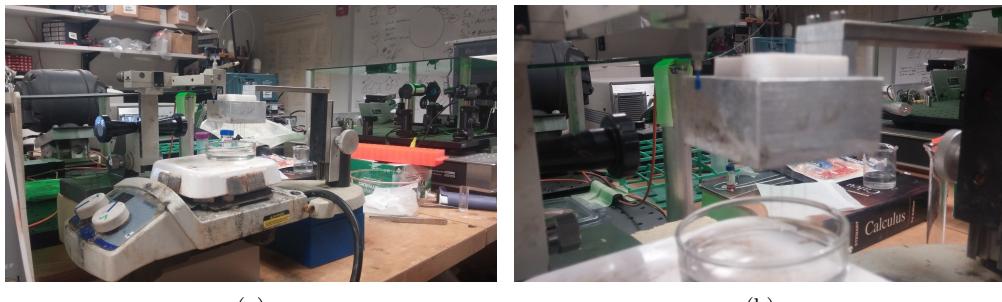


Figure 1: Holding fibers, 1 in setup, 5 in groove holder.

3 SEM

Date of Imaging	XX/XX/2013
# Fibers Imaged	6
Microscope Performance	great
Signs of contamination	No
Good cleaves	6/6

SEM WAS GREAT! Turned on 2-2.5 hours early for vacuum pump, and turned on beam 30 min early. Fresh tape as well. All images were spoton. **All cleaves were great, with a slight defect on the very edge where blade touches fibers.** Fiber 1, which sat out longer **was not clean**, but other fibers which went directly from ethanol to grid **were very clean**.

3.1 Coverage Analysis ¹

Image	NPS	single_eqvs	double_eqvs	flat_eqvs	super_eqvs	Diam Est(nm)	bw_nonoise(%)
f1_30000.tif	1.49e+06	53.47	35.77	10.76	0.0	20.57	25.49
f1_60000.tif	1.53e+06	67.98	28.91	3.11	0.0	21.5	18.4
f2_30000.tif	1.40e+06	48.92	29.54	21.53	0.0	21.0	30.03
f2_30000_2.tif	1.74e+06	50.13	31.35	17.74	0.79	20.86	28.63
f3_30000.tif	7.32e+05	78.9	19.23	1.87	0.0	22.12	10.28
f3_30000_2.tif	6.92e+05	76.79	21.0	2.22	0.0	22.15	9.7
f4_30000.tif	1.15e+06	65.42	28.13	6.45	0.0	22.12	18.08
f4_30000_2.tif	1.11e+06	53.65	36.36	9.99	0.0	20.73	20.45
f5_30000.tif	1.12e+06	60.31	29.34	10.35	0.0	20.02	18.85
f5_30000_2.tif	1.48e+06	41.61	35.78	22.61	0.0	20.95	27.17
f6_30000.tif	1.30e+06	56.86	28.49	8.13	6.52	21.03	20.26
f6_30000_2.tif	1.17e+06	50.62	35.36	14.02	0.0	21.08	25.66

Image	BSA	single_true	double_true	flat_true	super_true	corr_cov(%)	hex_ffrac(%)
f1_30000.tif	3.91e+07	70.39	23.54	6.07e+00	0.00e+00	22.04	29.08
f1_60000.tif	4.14e+07	81.09	17.24	1.67e+00	0.00e+00	21.13	20.74
f2_30000.tif	3.81e+07	67.83	20.48	1.17e+01	0.00e+00	25.26	33.72
f2_30000_2.tif	4.63e+07	68.79	21.51	9.69e+00	4.24e-03	29.67	31.85
f3_30000.tif	2.05e+07	88.31	10.76	9.27e-01	0.00e+00	9.42	12.05
f3_30000_2.tif	1.92e+07	86.99	11.89	1.11e+00	0.00e+00	9.13	11.05
f4_30000.tif	3.13e+07	79.49	17.09	3.43e+00	0.00e+00	16.87	20.04
f4_30000_2.tif	2.89e+07	70.52	23.9	5.58e+00	0.00e+00	16.67	22.85
f5_30000.tif	3.02e+07	76.02	18.49	5.49e+00	0.00e+00	17.26	20.99
f5_30000_2.tif	3.94e+07	60.56	26.04	1.34e+01	0.00e+00	26.81	30.34
f6_30000.tif	3.35e+07	76.26	19.1	4.63e+00	1.10e-02	18.33	23.38
f6_30000_2.tif	3.06e+07	68.3	23.85	7.85e+00	0.00e+00	18.31	28.7

SEM Analysis

Coverage estimates based on bf_nonoise(%).

- I'd estimate the coverage of fiber 1 between 18 and 22%
 - The 30k image over-estimates because it has more contamination, so I trust the 60k more
 - This fiber sat out the longest, **so only one that showed signs of contamination.**
- The range of coverages is about 10% for fiber 3 to 29% for fiber 2. In terms of hex fill frac, this is about 11% to 32%!

4 Results

4.1 Summary

- The range of coverages is about 10% for fiber 3 to 29% for fiber 2. In terms of hex fill frac, this is about 11% to 32%
 - Fibers 2-5 sat in AuNPs a few seconds longer, so might be slightly over-counting, but range is between fibers 2 and 3, **so does not affect this result.**
 - This seems consistent with functionalizing the fibers individually and **means that different rate of functionalizing fibers at different times in self assembly.**
 - Cleaves were also perfect**, so cleaves cannot explain this rate!

- 6 fibers all had good formation at least.
- Because the biggest variance was on fibers 2 and 3, *this can't be attributed to the distance from the stirrer in the solution as clay wisely pointed out.*

It turns out that within the six fibers, **a range of 20%** coverage variability was observed, with the least coated fiber being approximately 10% covered, and the most being about 30%. The fibers were all cleaved perfectly and all exhibited clean AuNP formation. All the images were clear except for fiber 1 perhaps, which had contamination likely due to sitting out on the grid for a while while I worked and soldered nearby. These results are at a glance commensurate with the variability in rates I typically observed functionalizing one at a time. This work is complimentary to some results last fall where we explored various ways to control formation upper limit, including PH changes and AuNP concentration. This is intended to be incorporated into a discussion section in an outlined paper skeleton which I'll distribute soon for feedback and brainstorming.

4.2 Follow-up

- Use ilastik to carefully threshold and see how close our estimates of BW coverage are.
- Could also use these images for comparison of flats, supers, but not sure if that's worth our time the moment.

5 Sectional Data Analysis

5.1 f1-aunps

See picture in main report for experimental setup!

Steps:

- Fiber sat in air in splice a while while we work out logistics, but **data was nice so didn't affect it.**
 - Only put the other 5 fibers nearby after logistics worked out so they were minimally affected; taking groove piece apart and back together also done with minimal hassle.
- Added 10mL AuNPs to 10mL DI water in clean pirhana dish.
 - Fibers had been placed in h2o prior to AuNP addition
 - Data was fast and good on AuNP formation
- Self-assembly was so quick that final data probably closer to 2700-3000 counts
- Transferred splice fiber into its own ethanol, and probably an extra 2-5 seconds passed before we pulled other fibers out. Additionally, **these fibers sat in air about 5 seconds longer than other fiber.** Therefore, **Should look for similarities within these 5 fibers that are possibly not in fiber 1 that would explain this extra air and AuNP time.** Overall, the handling was good and this is as good as we could do this run. I don't think the air or other effects will be first-order affects.

What went well

- Nothing to remark

What went poorly

- Nothing to remark

File path: [1] "/home/lab3/Dropbox/FiberData/2014/7_10_14_BATCHVARIETY/Datafiles/Fiber1/NPSAM/"

```
[1] "Integration Time (usec): 63000 (USB2E7196)"  
[1] "Boxcar Smoothing: 10 (USB2E7196)"  
[1] "Spectra Averaged: 50 (USB2E7196)"  
[1] "Date: Thu Jul 10 18:00:30 EDT 2014"  
[1] "total elapsed time = 134s"
```

Warning: Parameters shown from first saved datafile; may have been changed during the run.

```
## Warning:  cannot open file './CSV_Files/RawData.csv':  No such file or directory  
## Error:  cannot open the connection
```

```
## Warning:  cannot open file './CSV_Files/DarkCorrData.csv':  No such file or directory  
## Error:  cannot open the connection
```

```
## Warning:  cannot open file './CSV_Files/RefCorrData.csv':  No such file or directory  
## Error:  cannot open the connection
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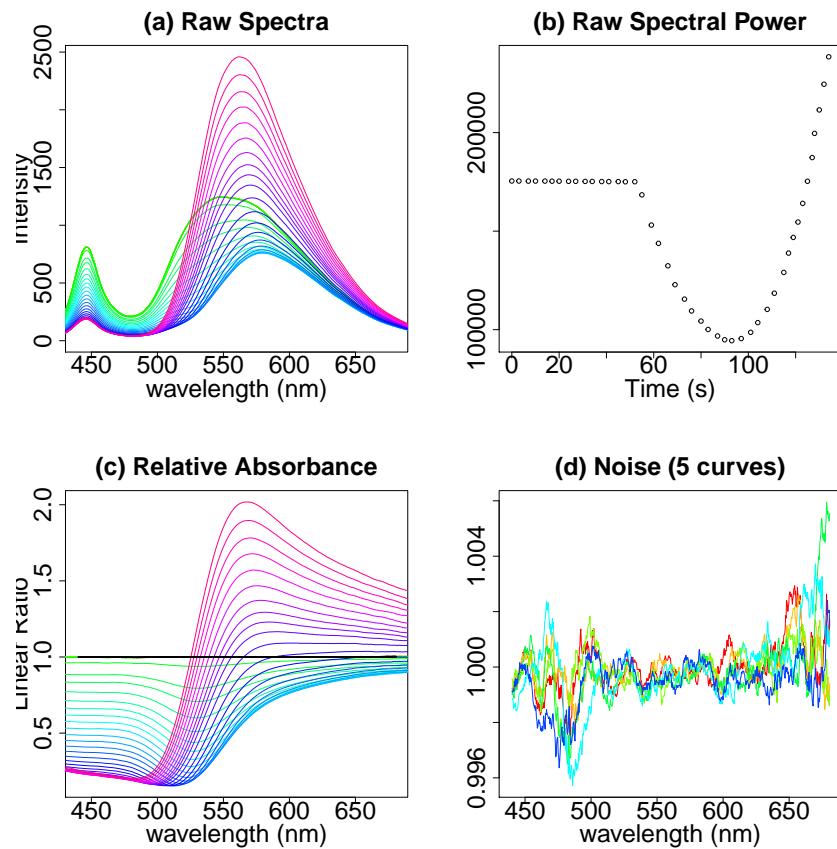


Figure 1: Dark corrected (a) raw spectra (b) raw spectral area (c) relative absorbance (linear ratio) (d) first five relative absorbance curves. **Note:** the black lines in (c) correspond to (d).

5.2 Script Parameters

PyUvVis Parameters

- bline_fit: True
- specunit: nm
- sub_base: True
- reference: 0
- _fit_regions: ((345.0, 395.0), (900.0, 1000.0))
- t_start: None
- intvlunit: s
- git: False
- _valid_minmax: (339.0, 1024.0)
- x_max: 680.0
- x_min: 430.0
- uv_ranges: 8
- iunits: [None, 'r', 'a']
- t_end: None

Analysis Parameters

- plot_dpi: None
- trace: False
- verbosity: warning
- sweep: True
- analysis: ['1d']
- inroot: Datafiles/
- fontsize: 18
- plot_dim: width=6cm
- outroot: Analysis/
- overwrite: True

5.3 IPython Notebooks

Links will not work until git pushed.

APPENDIX

A Related

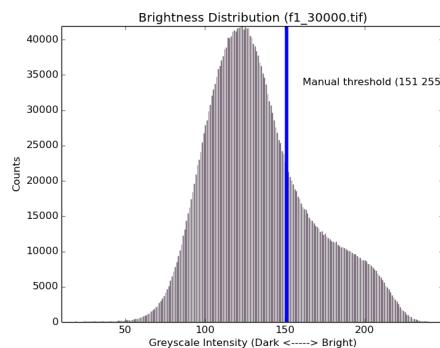
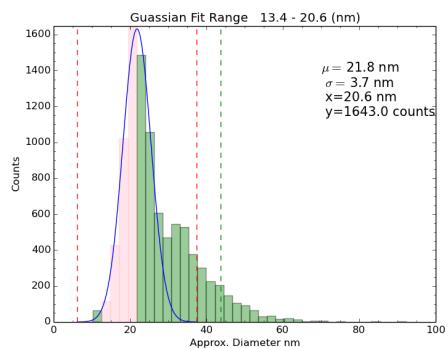
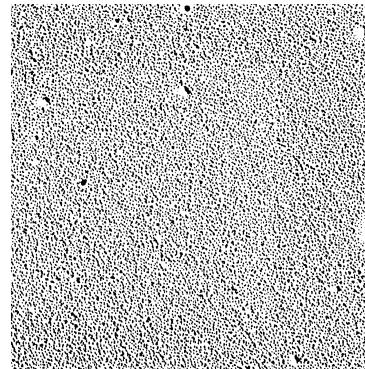
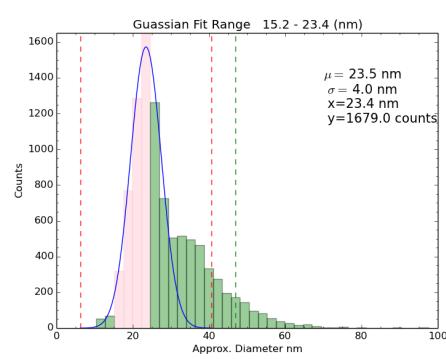
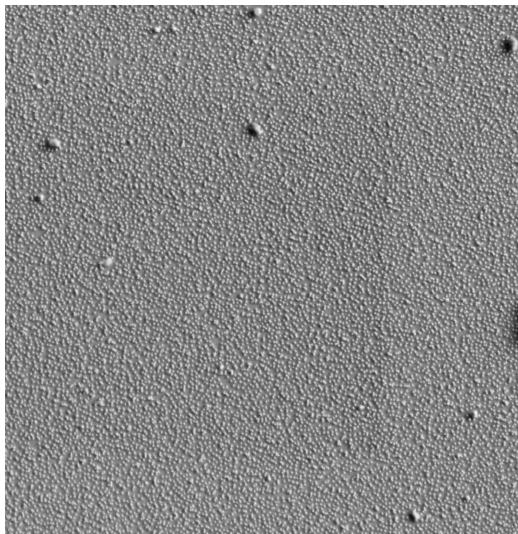
B Acronyms and Terminology

- **AuNP:** Gold nanoparticle
- **BSA:** Bovine Serum Albumin
- **PBS:** Phosphate Buffered Saline- 0.01M pH 7.4 from Sigma Aldrich powder packet.
- **TMSDE:** (3-Trimethoxysilylpropyl)-diethylenetriamine
- **MPTMS:** (3-Mercaptopropyl)trimethoxysilane
- **PTMS:** Propyltrimethoxysilane
- **ETMS:** Ethyltrimethoxysilane
- **DTSSP:** Water soluble DSP (Dithiobis[succinimidyl propionate])

C SEM Images and Histograms

5(4,1.0) july_10_14

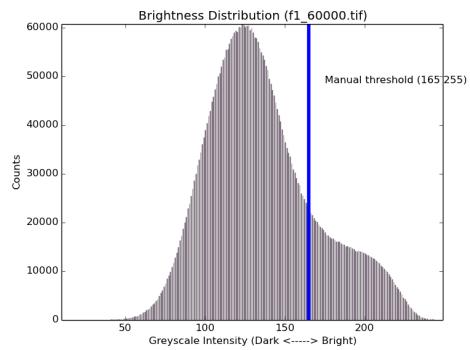
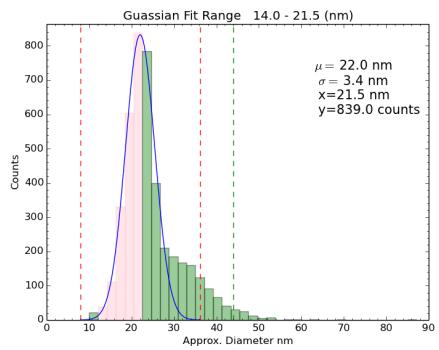
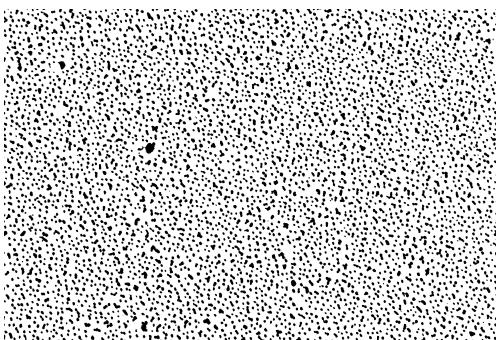
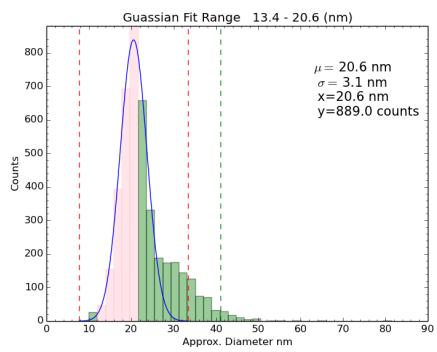
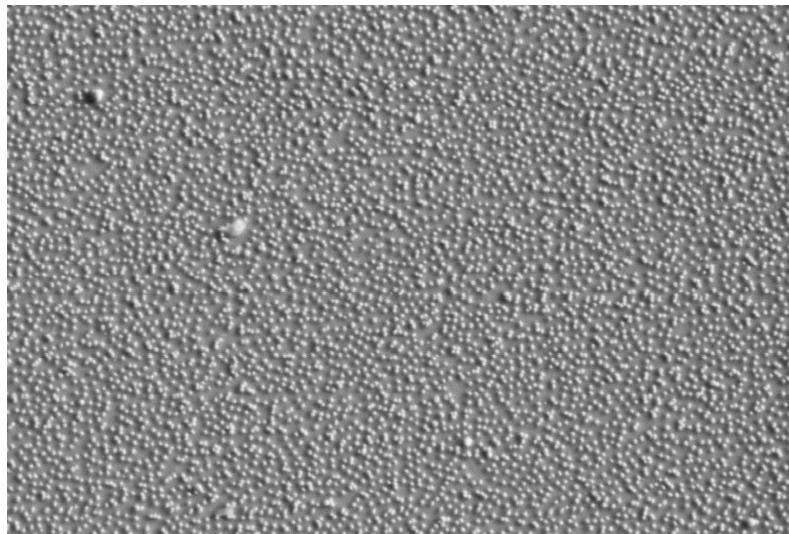
f1_30000



f1_30000: SEM image, raw (top)/size-corrected (bottom), diam histograms, binary, grayscale.
Cropped: **True** BW coverage: **25.49** corr coverage: **22.04** hex fillfrac: **29.08** man-adjustment: **Yes**

5(4,1.0) **july_10_14**

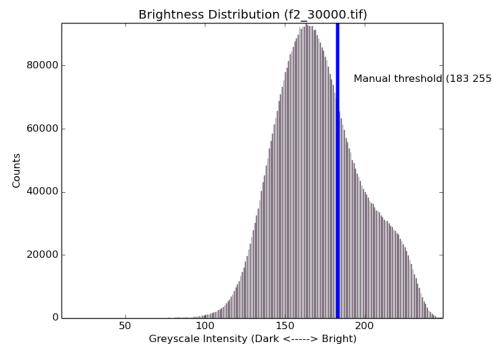
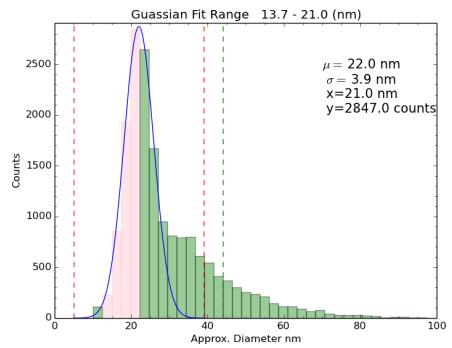
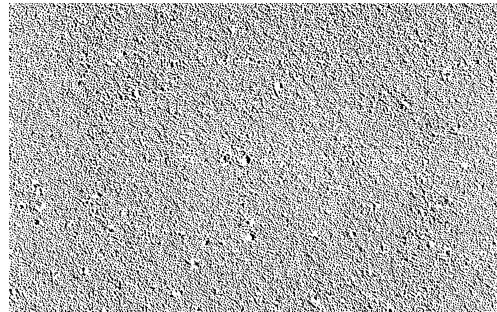
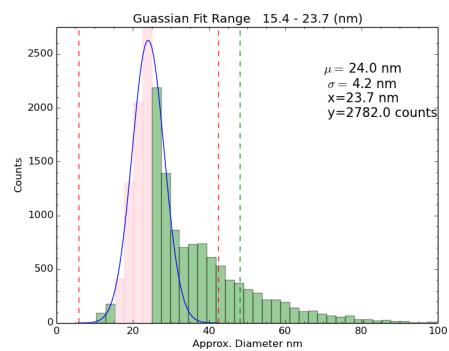
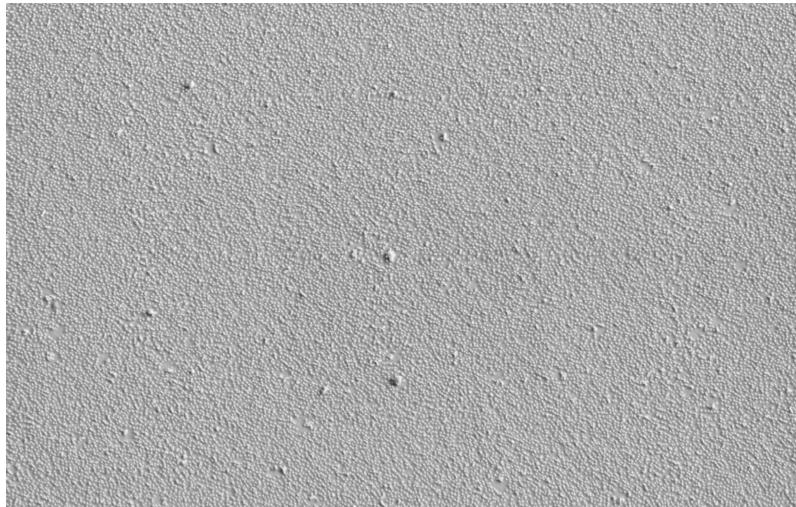
f1_60000



f1_60000: SEM image, raw (top)/size-corrected (bottom), diam histograms, binary, grayscale.
Cropped: **True** BW coverage: **18.4** corr coverage: **21.13** hex fillfrac: **20.74** man-adjustment: **Yes**

5(4,1.0) july_10_14

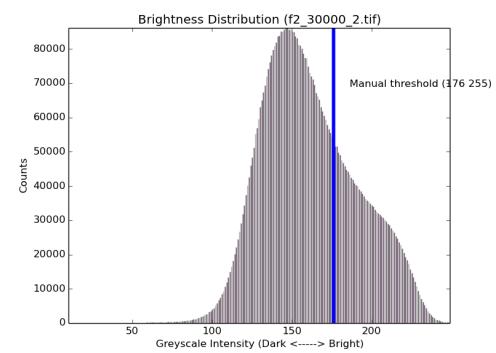
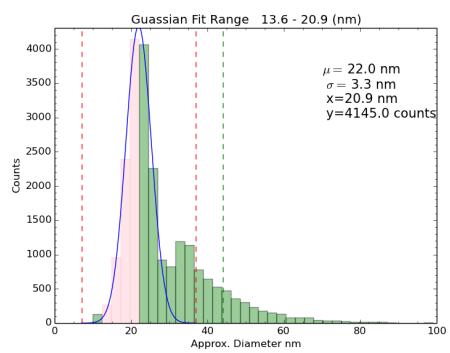
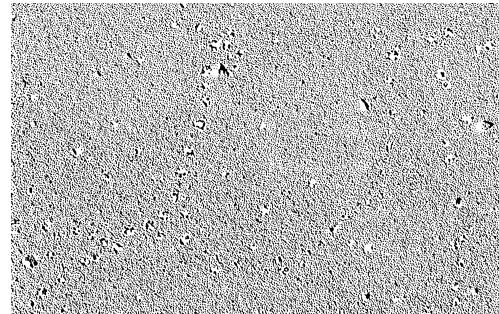
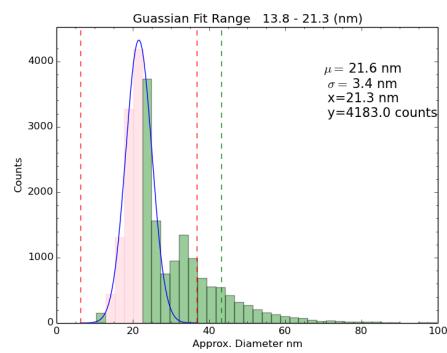
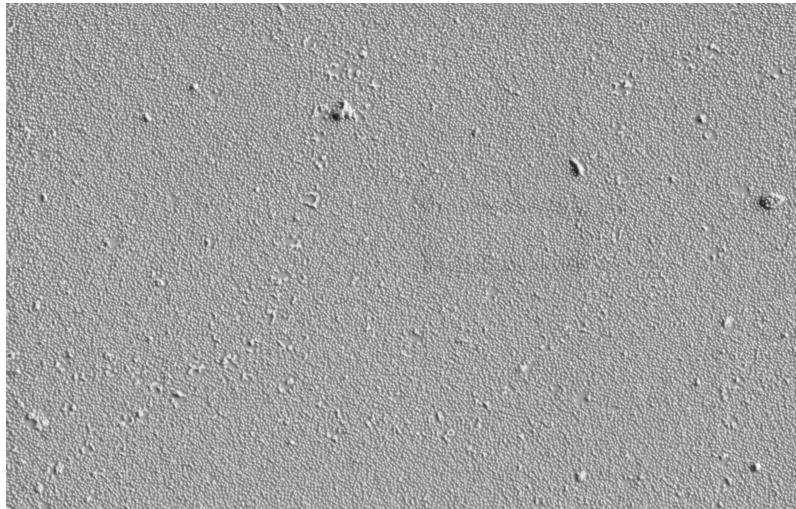
f2_30000



f2_30000: SEM image, raw (top)/size-corrected (bottom), diam histograms, binary, grayscale.
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5(4,1.0) july_10_14

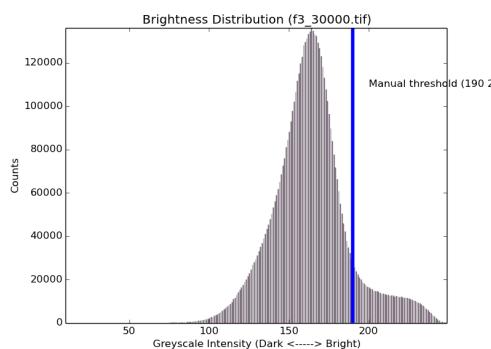
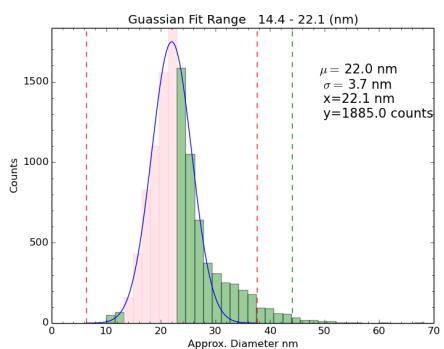
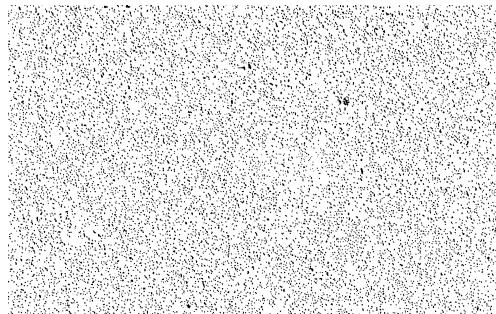
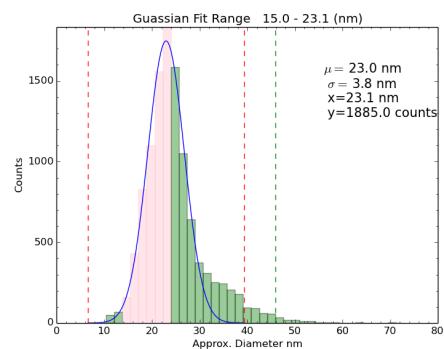
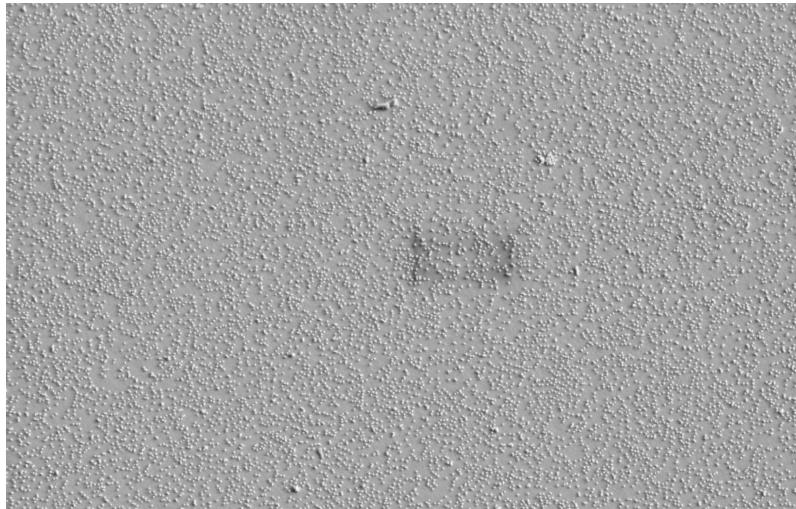
f2_30000_2



f2_30000_2: SEM image, raw (top)/size-corrected (bottom), diam histograms, binary, grayscale.
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5(4,1.0) july_10_14

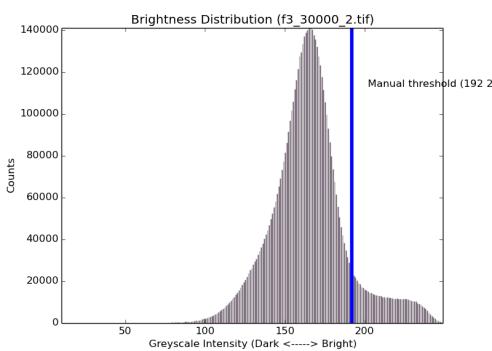
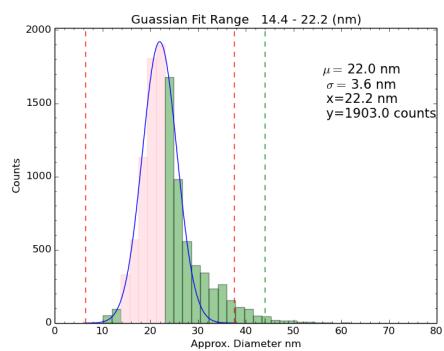
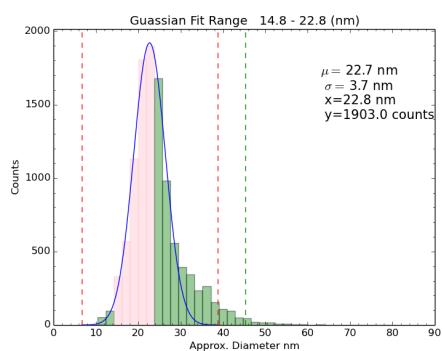
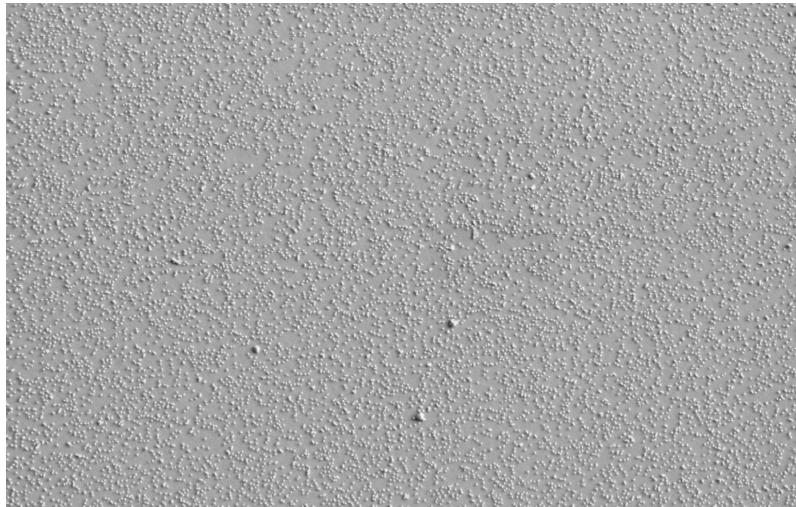
f3_30000



f3_30000: SEM image, raw (top)/size-corrected (bottom), diam histograms, binary, grayscale.
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5(4,1.0) july_10_14

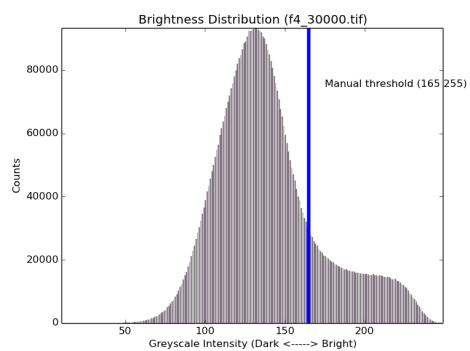
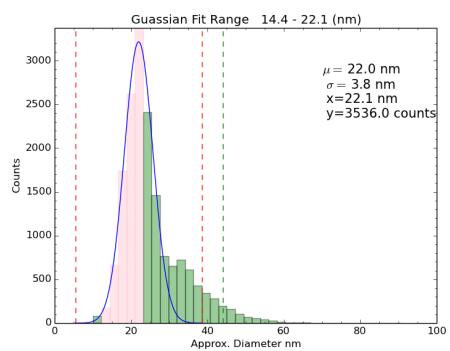
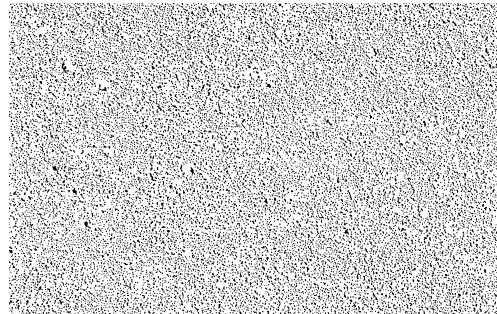
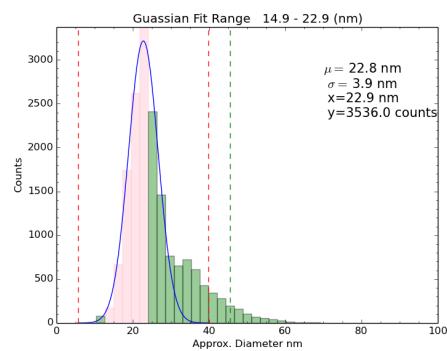
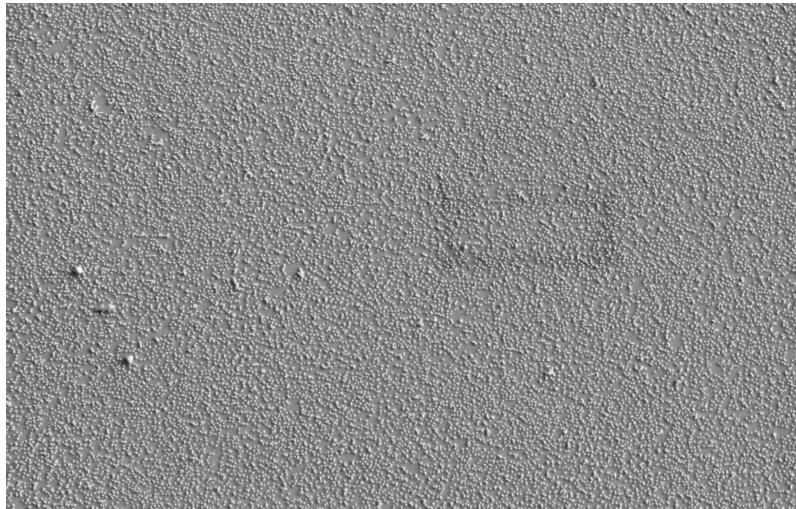
f3_30000_2



f3_30000_2: SEM image, raw (top)/size-corrected (bottom), diam histograms, binary, grayscale.
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5(4,1.0) july_10_14

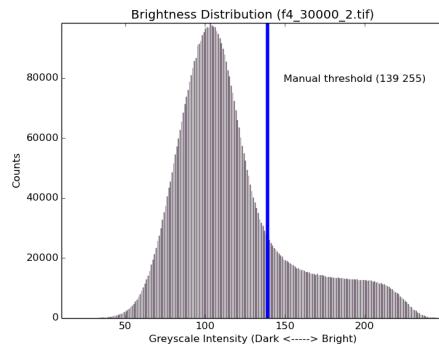
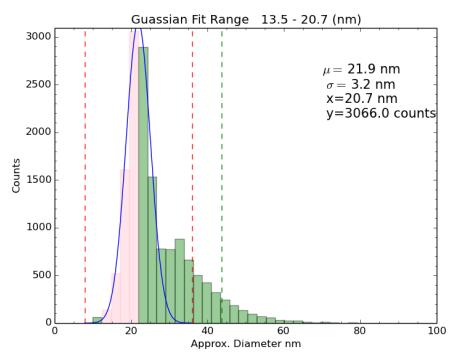
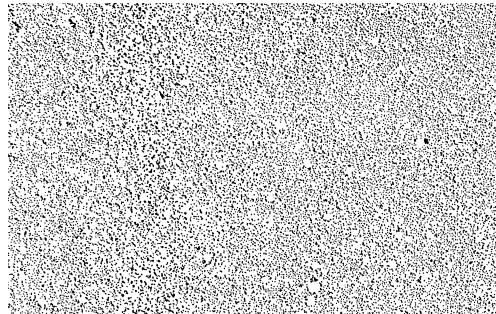
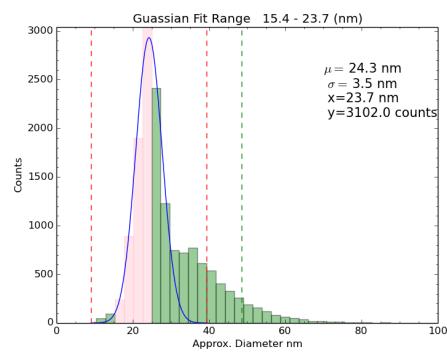
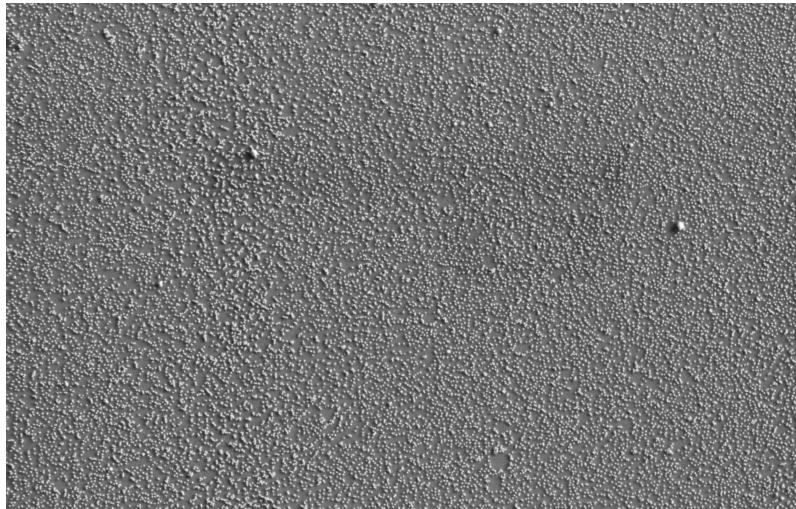
f4_30000



f4_30000: SEM image, raw (top)/size-corrected (bottom), diam histograms, binary, grayscale.
Cropped: **True** BW coverage: **18.08** corr coverage: **16.87** hex fillfrac: **20.04** man-adjustment: **Yes**

5(4,1.0) july_10_14

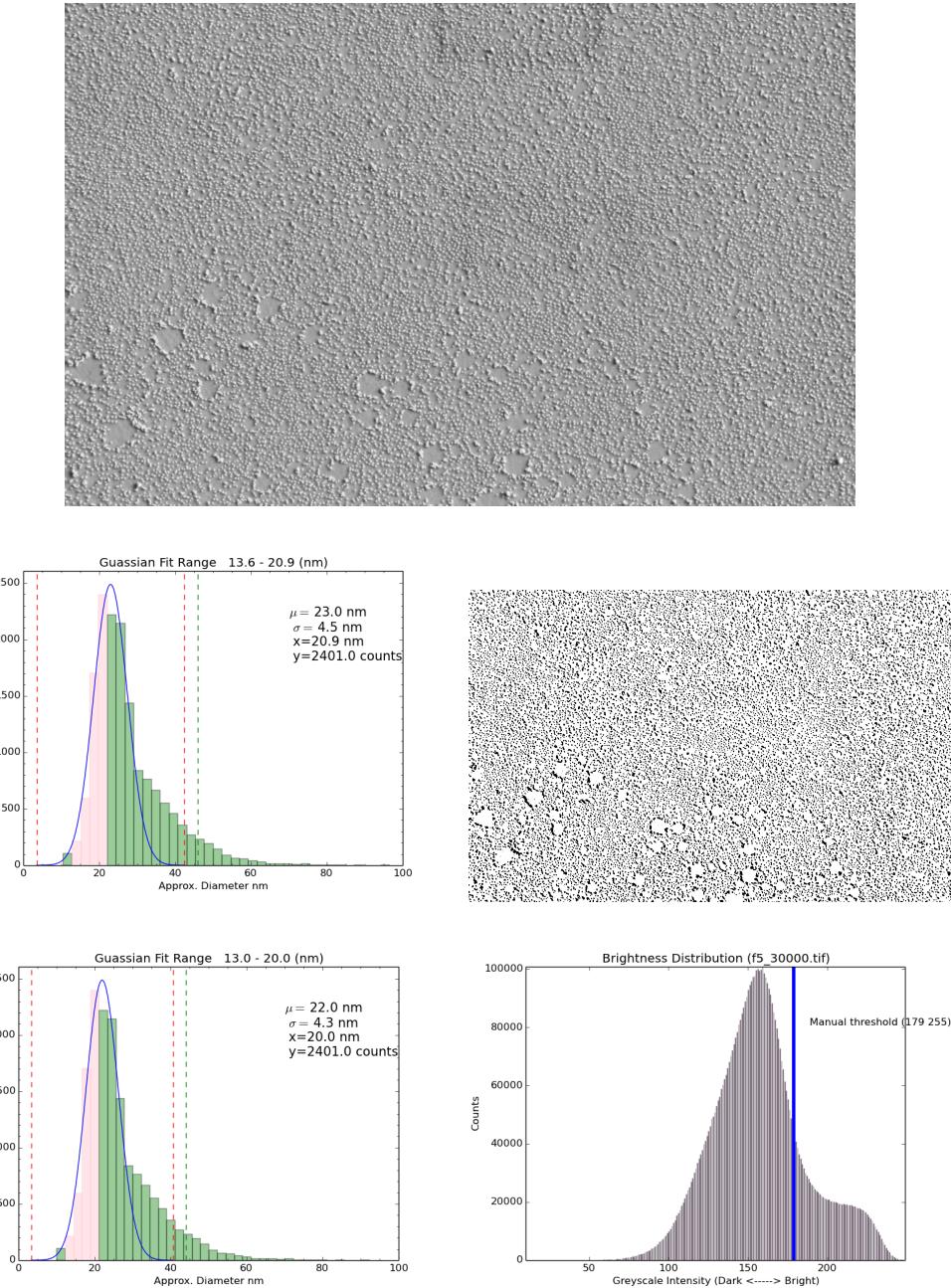
f4_30000_2



f4_30000_2: SEM image, raw (top)/size-corrected (bottom), diam histograms, binary, grayscale.
Cropped: **True** BW coverage: **20.45** corr coverage: **16.67** hex fillfrac: **22.85** man-adjustment: **Yes**

5(4,1.0) july_10_14

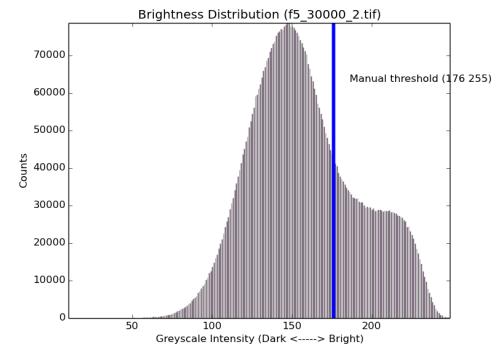
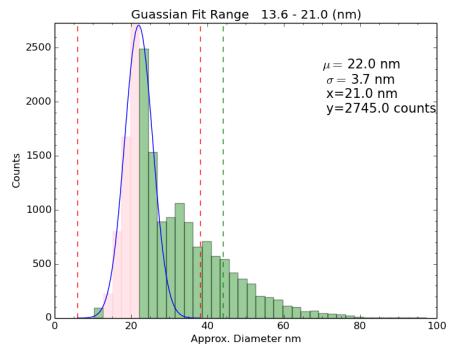
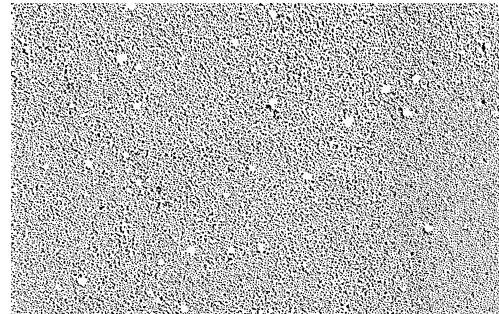
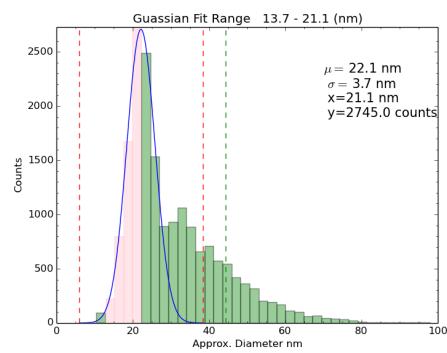
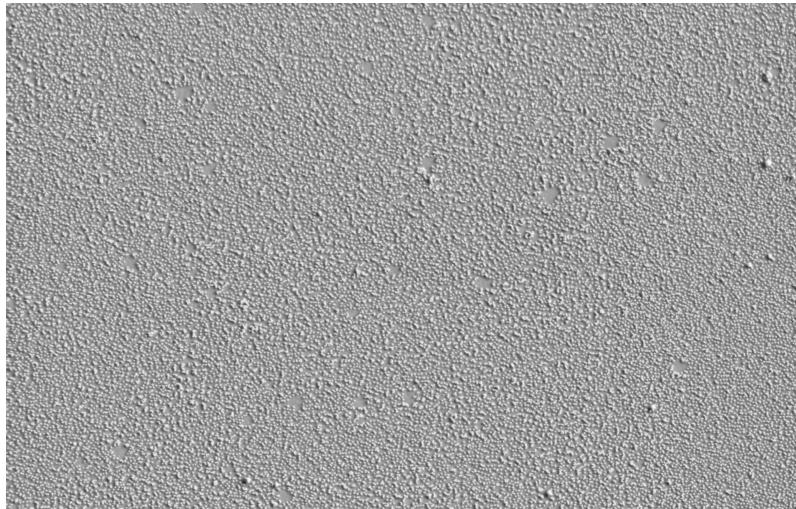
f5_30000



f5_30000: SEM image, raw (top)/size-corrected (bottom), diam histograms, binary, grayscale.
Cropped: **True** BW coverage: **18.85** corr coverage: **17.26** hex fillfrac: **20.99** man-adjustment: **Yes**

5(4,1.0) july_10_14

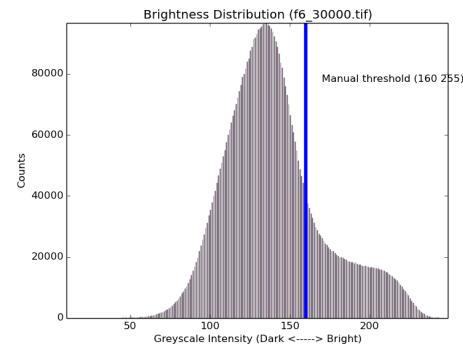
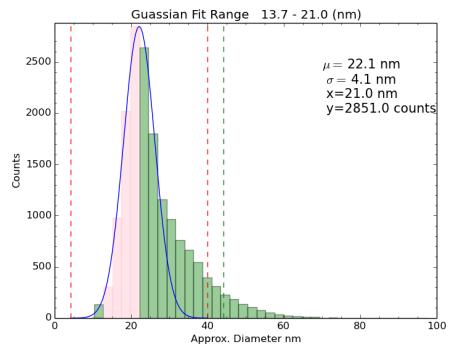
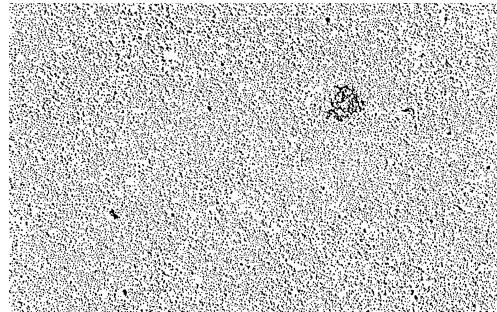
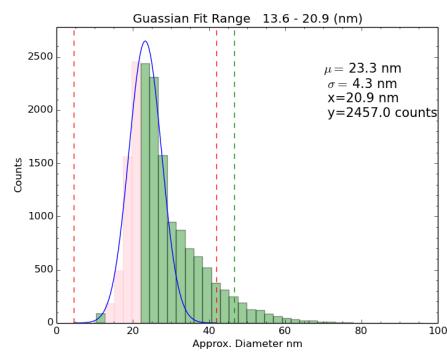
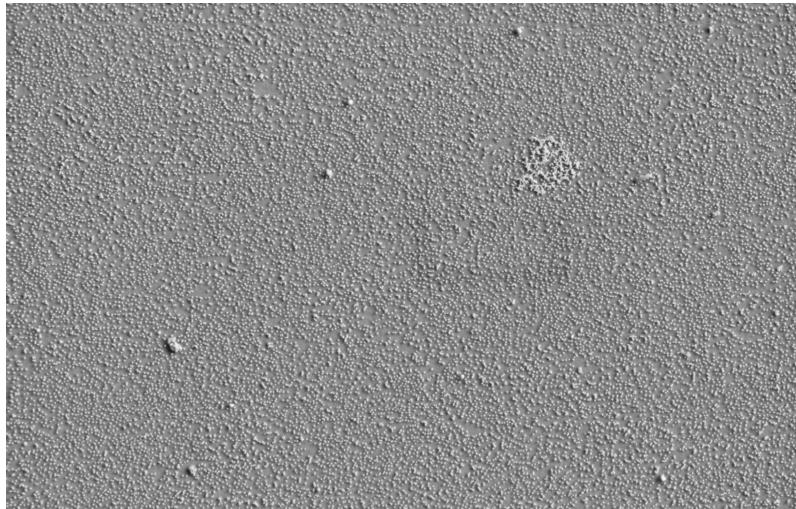
f5_30000_2



f5_30000_2: SEM image, raw (top)/size-corrected (bottom), diam histograms, binary, grayscale.
Cropped: **True** BW coverage: **27.17** corr coverage: **26.81** hex fillfrac: **30.34** man-adjustment: **Yes**

5(4,1.0) july_10_14

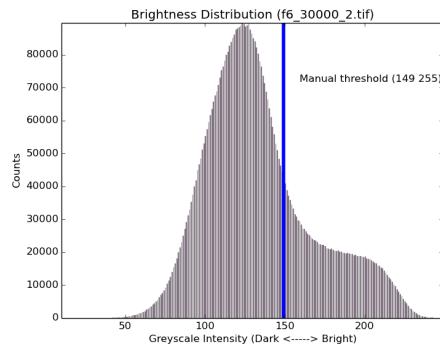
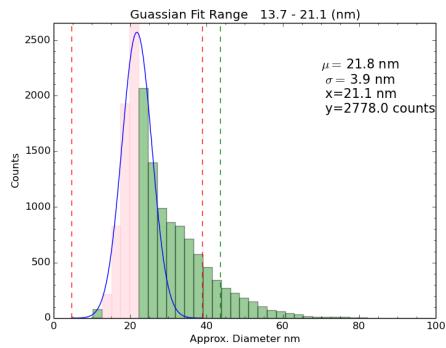
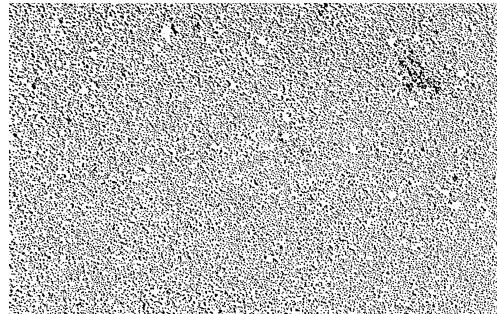
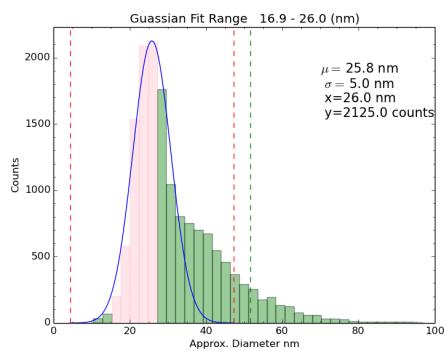
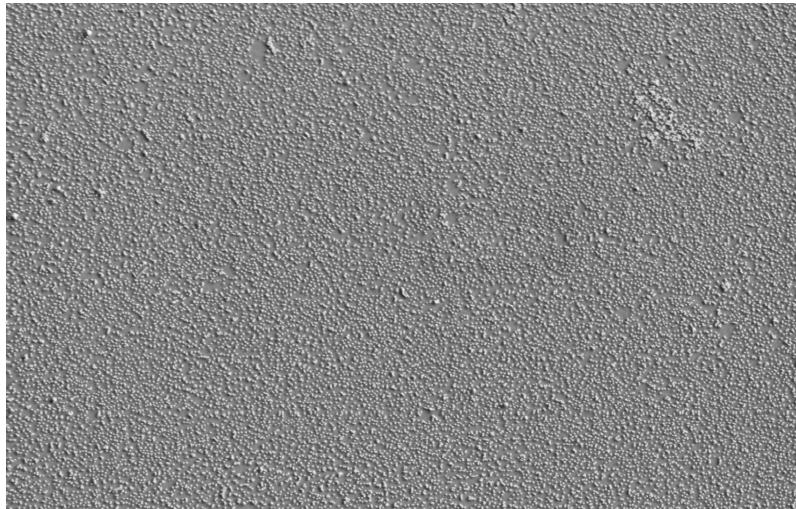
f6_30000



f6_30000: SEM image, raw (top)/size-corrected (bottom), diam histograms, binary, grayscale.
Cropped: **True** BW coverage: **20.26** corr coverage: **18.33** hex fillfrac: **23.38** man-adjustment: **Yes**

5(4,1.0) july_10_14

f6_30000_2



f6_30000_2: SEM image, raw (top)/size-corrected (bottom), diam histograms, binary, grayscale.
Cropped: **True** BW coverage: **25.66** corr coverage: **18.31** hex fillfrac: **28.7** man-adjustment: **Yes**