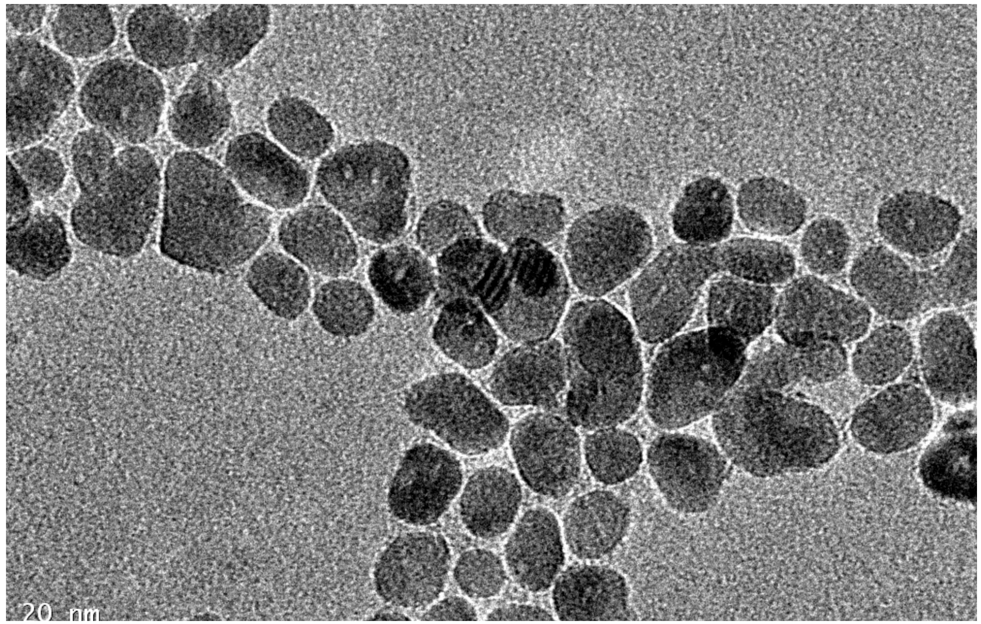
Pyrite Collaborative Update

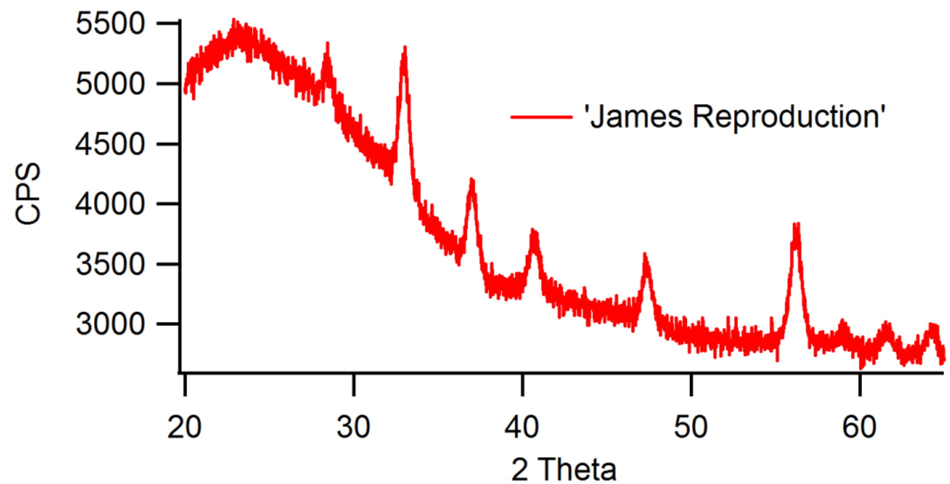
Nanocrystal Synthesis and Thin Films from Molecular Inks

Goals For Pyrite Research

- Scalable Synthesis of Pyrite Nanocrystals
  - Gram Scale Synthesis of Pyrite NCs
  - Non-Chlorinated Solvent
  - Halogen-Free Synthesis
  - Transition to film production using scaled Inks
- Molecular Films
  - High density, connected films with large grains and variable thickness

Standard Law Group FeS_2 NC Synthesis





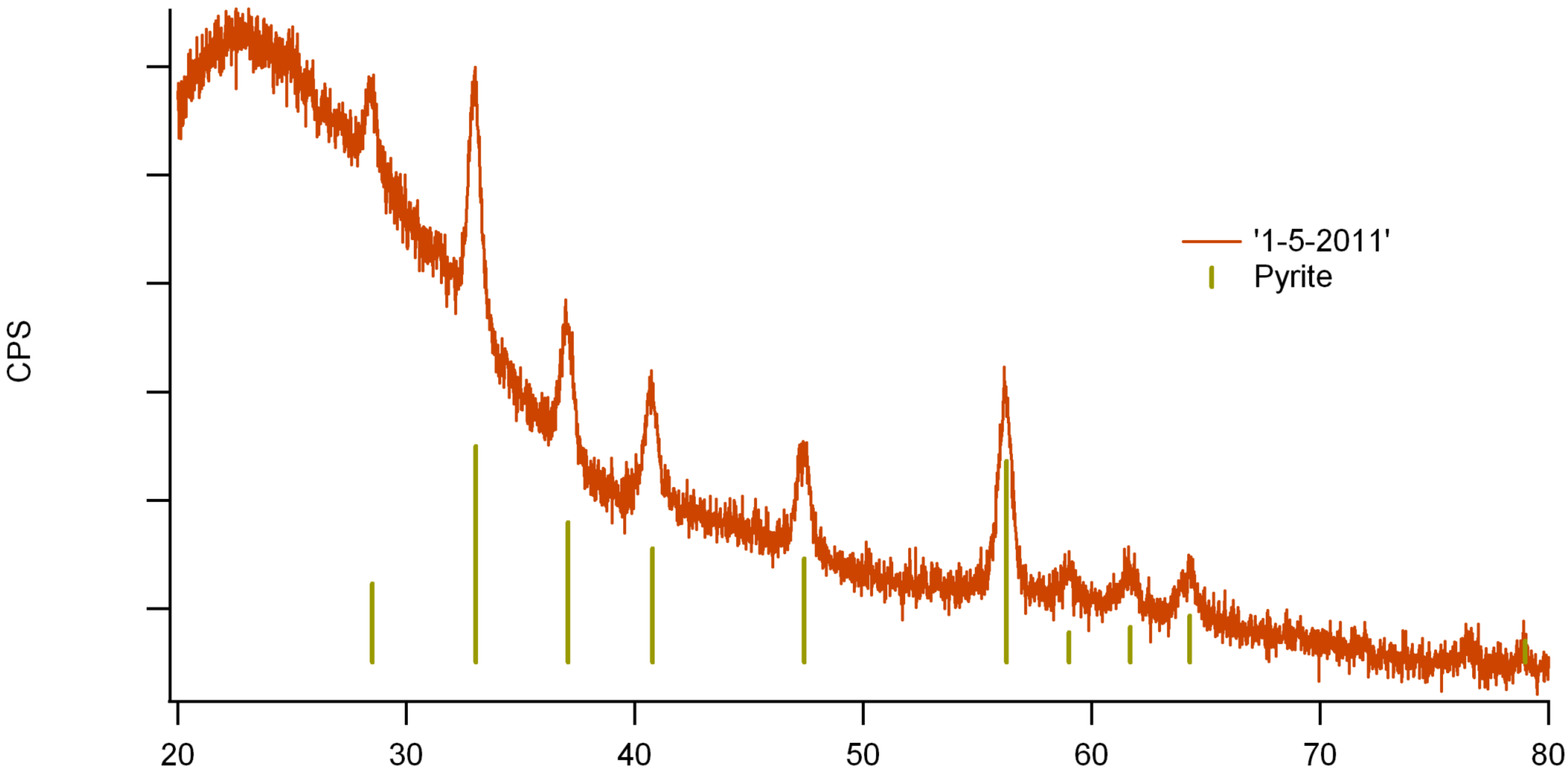
Standard Law Group FeS_2 NC Synthesis

- 2.5mmol FeCl2*4H20 in 25mL DDA
- (Verified with FeCl2 anhydrous)
- 400mg S in 10ml PE
- Dg @ 75C 1hr Inject Sulfur to Iron flask @220C and cook for 4 hours.
- Crash with ethanol redisperse with Chloroform and sit overnight.
- Next Day add xanthate and wash like normal after 45 minutes.

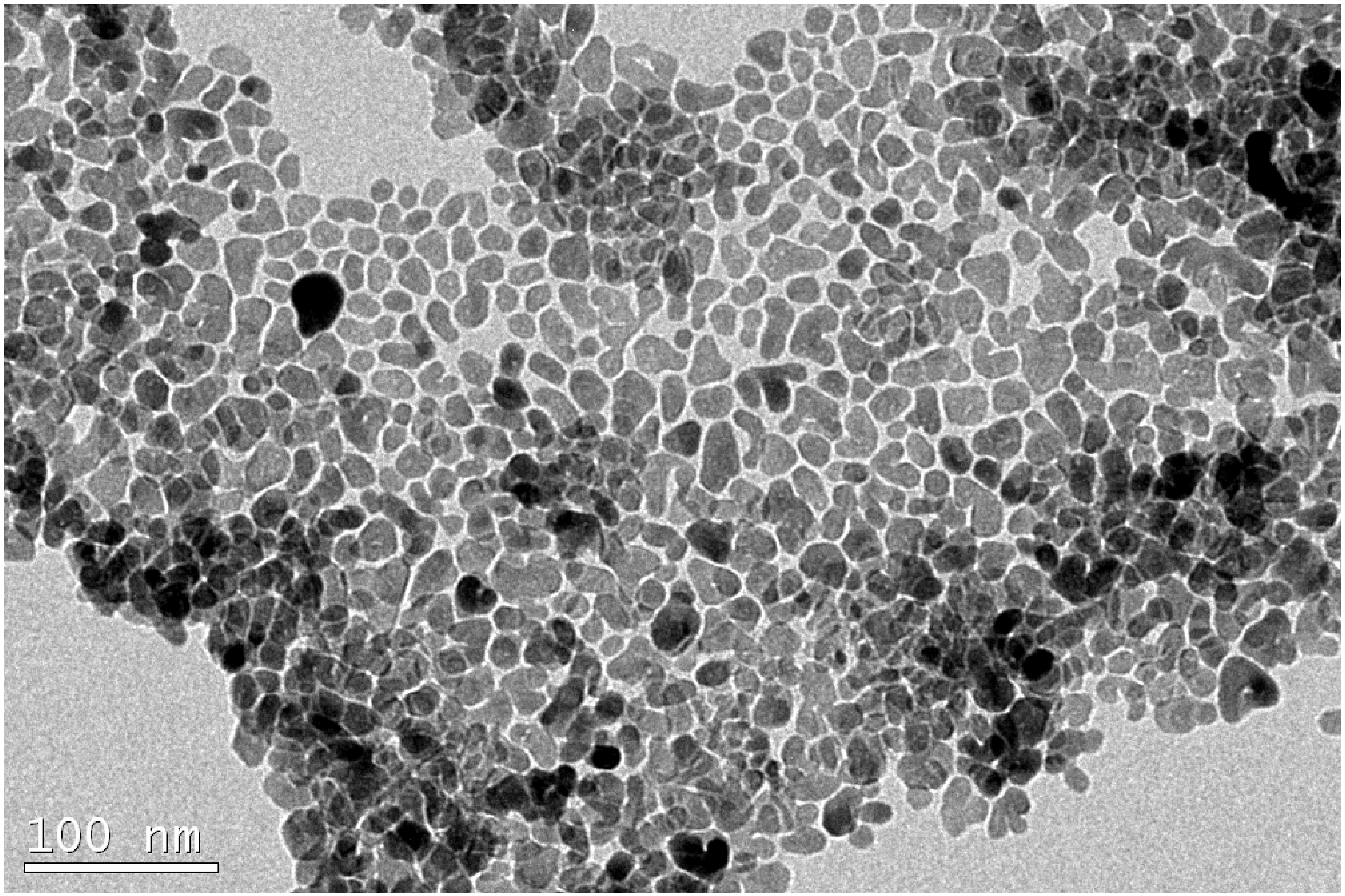
Pyrite NC Scale Up Reaction

- (1.2g $FeCl_2$ $4H_20$ / 86g ODA) (1.6g S/40ml PE)
- Dg @ 110 C / 85C
- Yield of Synthesis
  - 676mg Soluble
  - 74mg Insoluble
    - Soluble defined as still in solution after 5mins @ 4.4krpm
  - 90.1% Soluble Yield

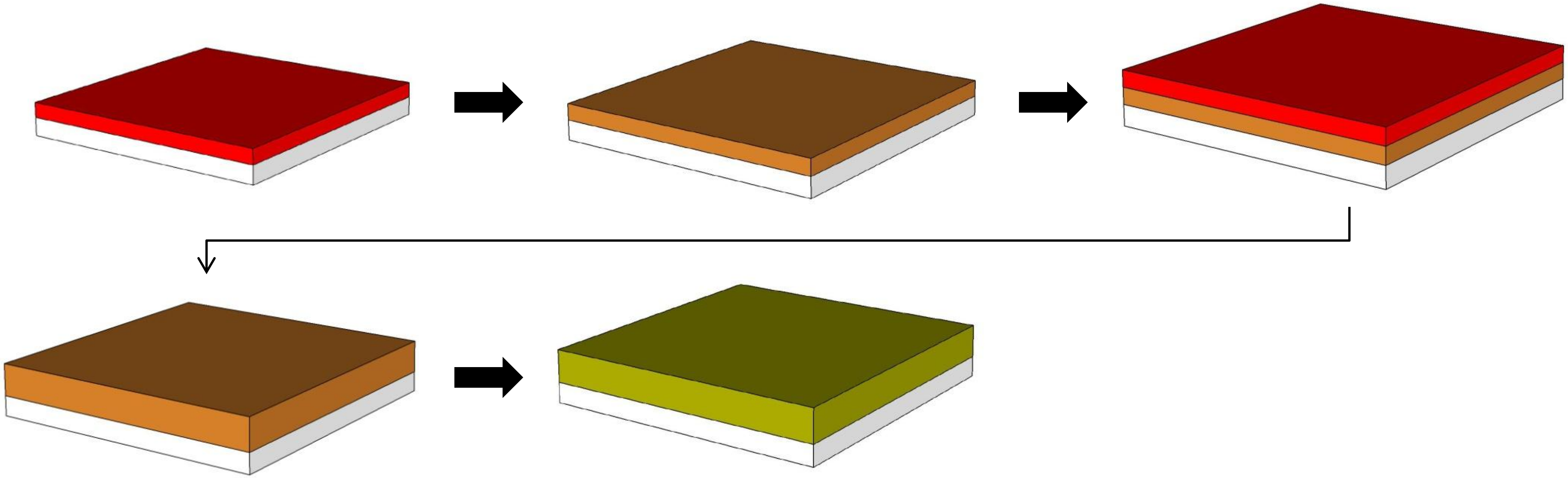
Pyrite Scale Up Synthesis



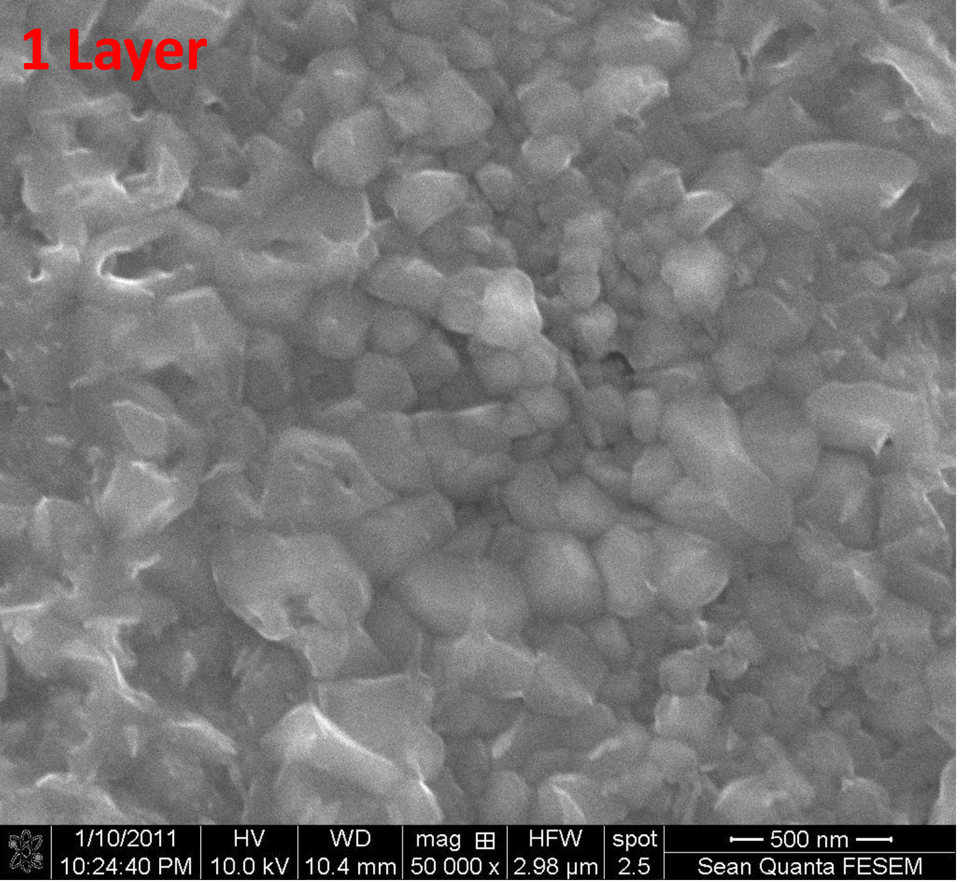
Pyrite Scale Up Synthesis



Pyrite Thin Films via Molecular Inks



- Iron (III) Acetylacetonate molecular ink is coated onto a glass slide
- The layer is then baked in air at 350 C for 30 minutes to form an amorphous iron oxide layer
- This process can be repeated to build varying thicknesses of films
- The final film is then converted to pyrite with either H_2S or S_8 annealing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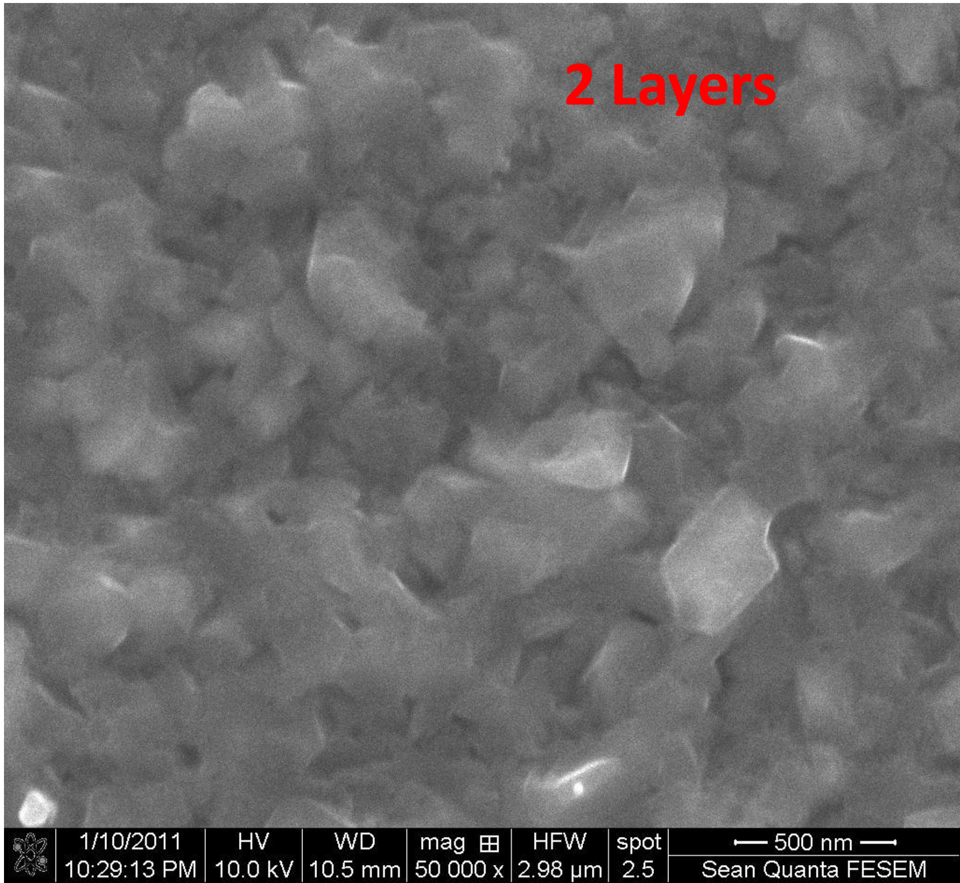
1/10/2011
10:24:40 PM
HV
10.0 kV
WD
10.4 mm
mag
50 000 x
HFW
2.98 µm
spot
2.5
500 nm
Sean Quanta FESEM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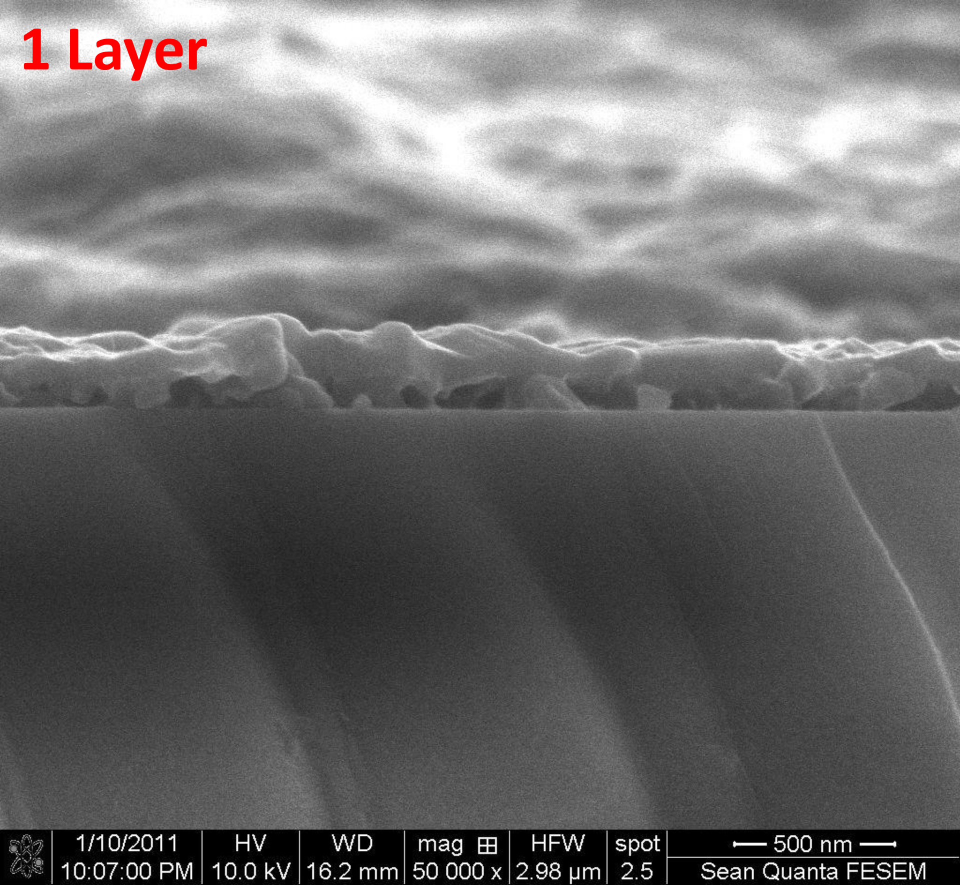
Sean Quanta FESEM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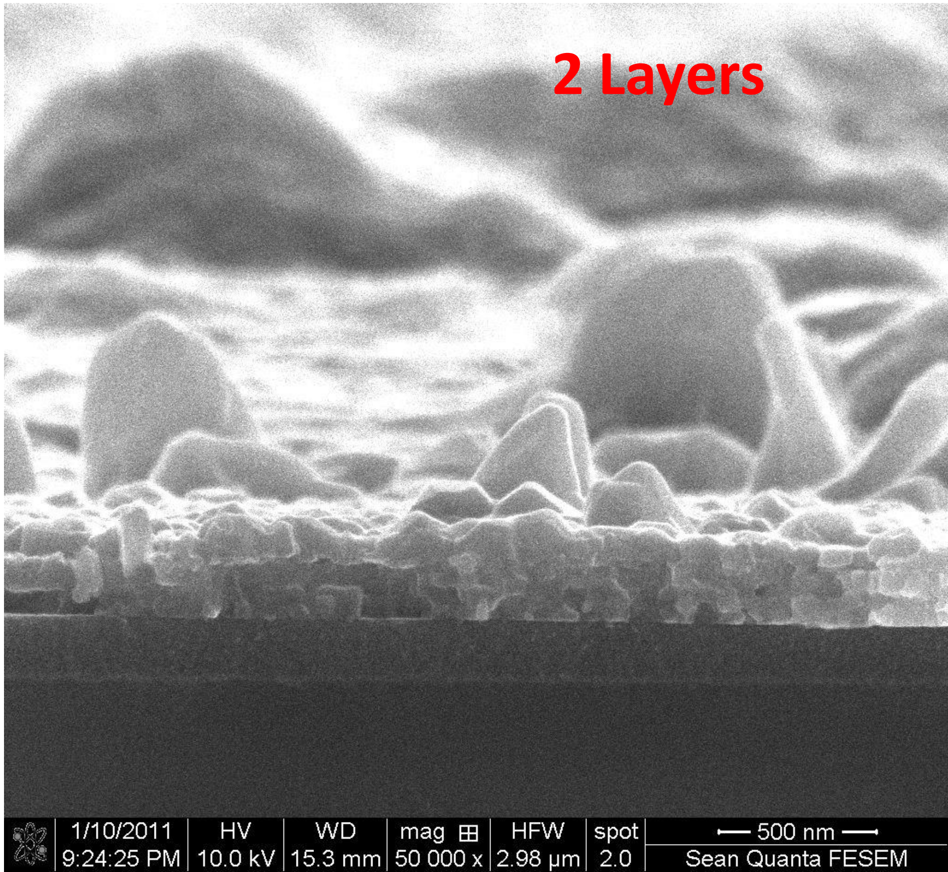
spot
2.5
500 nm
Sean Quanta FESEM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HFW
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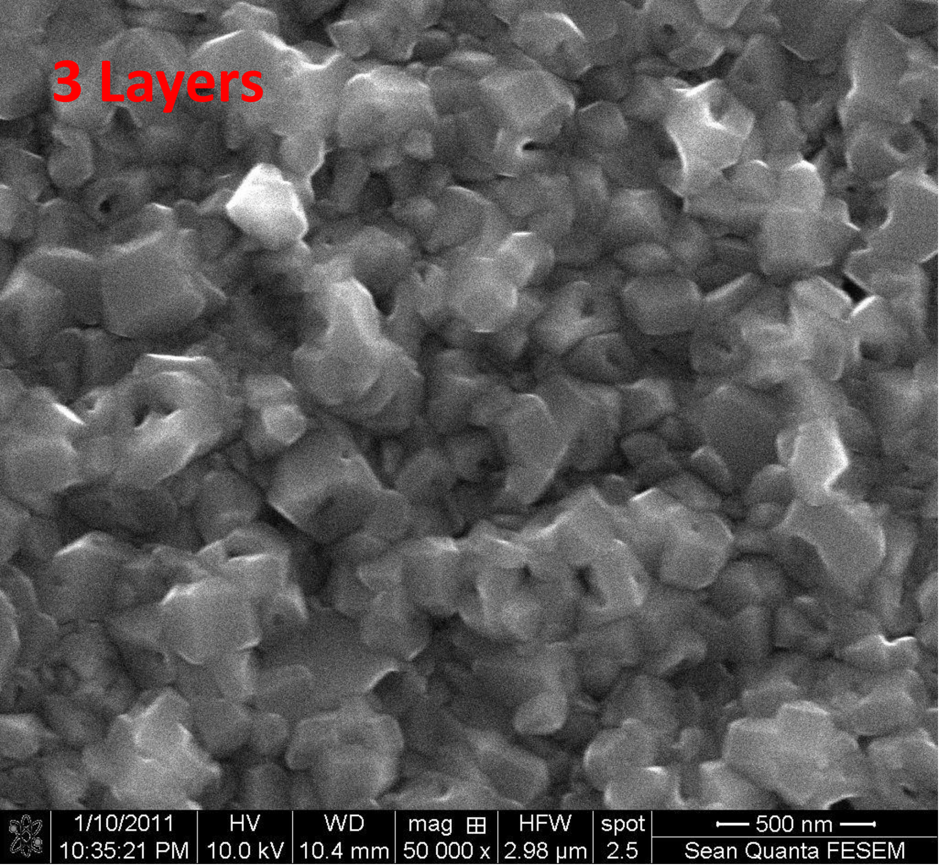
500 nm
Sean Quanta FESEM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HFW
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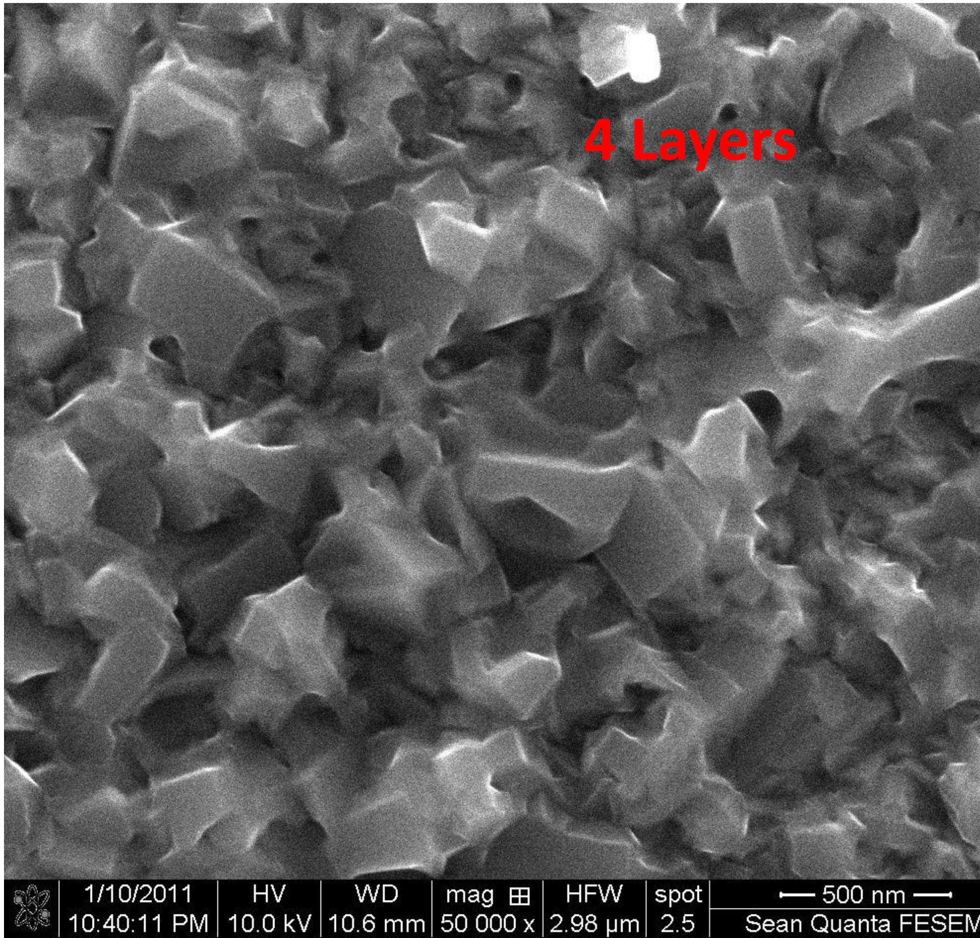
spot
2.5
500 nm
Sean Quanta FESEM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mag
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HFW
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spot
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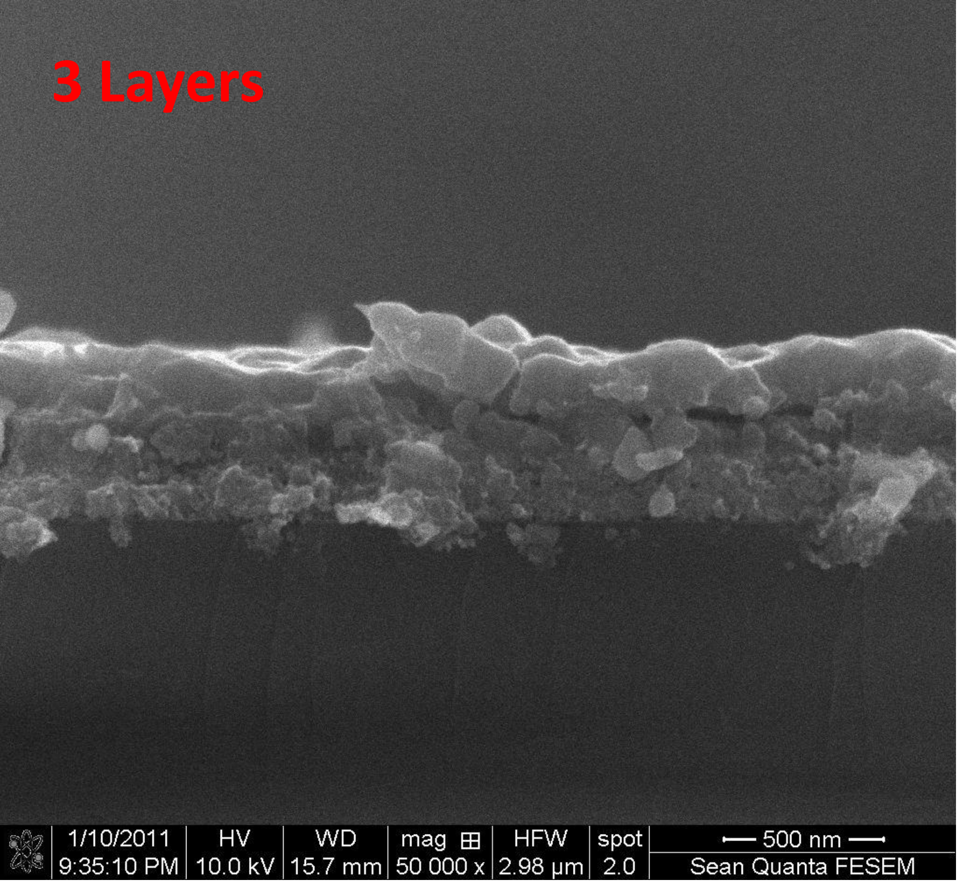
500 nm
Sean Quanta FESEM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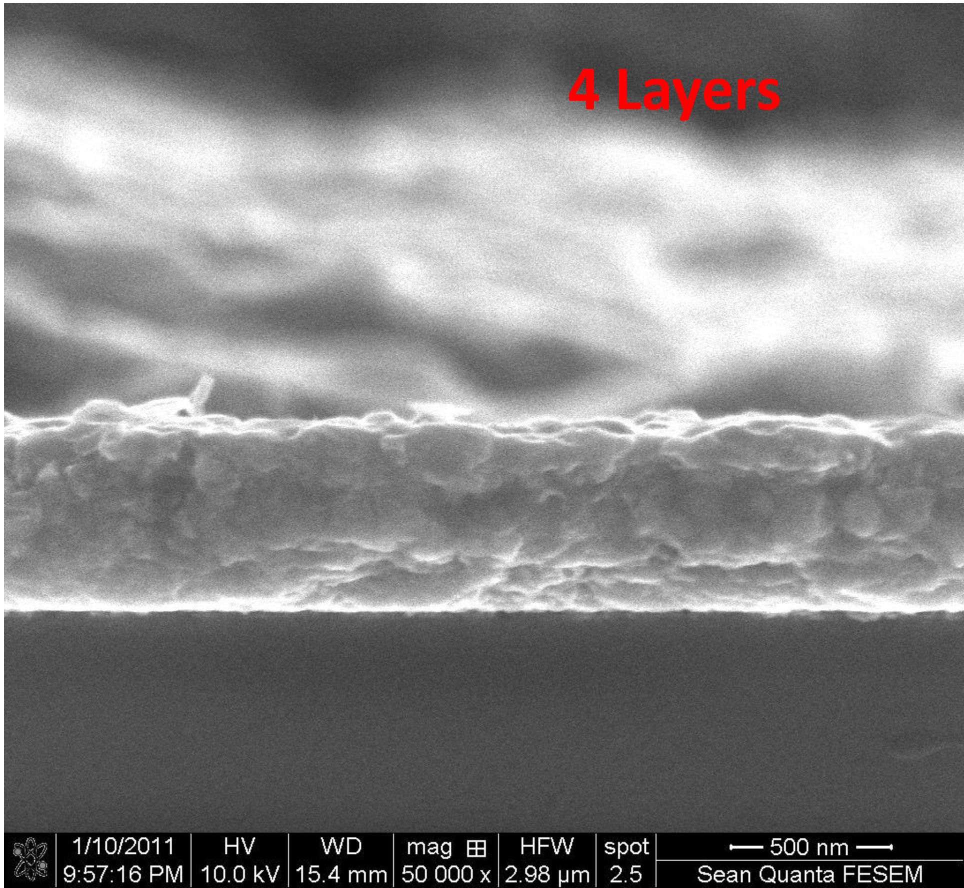
Sean Quanta FESEM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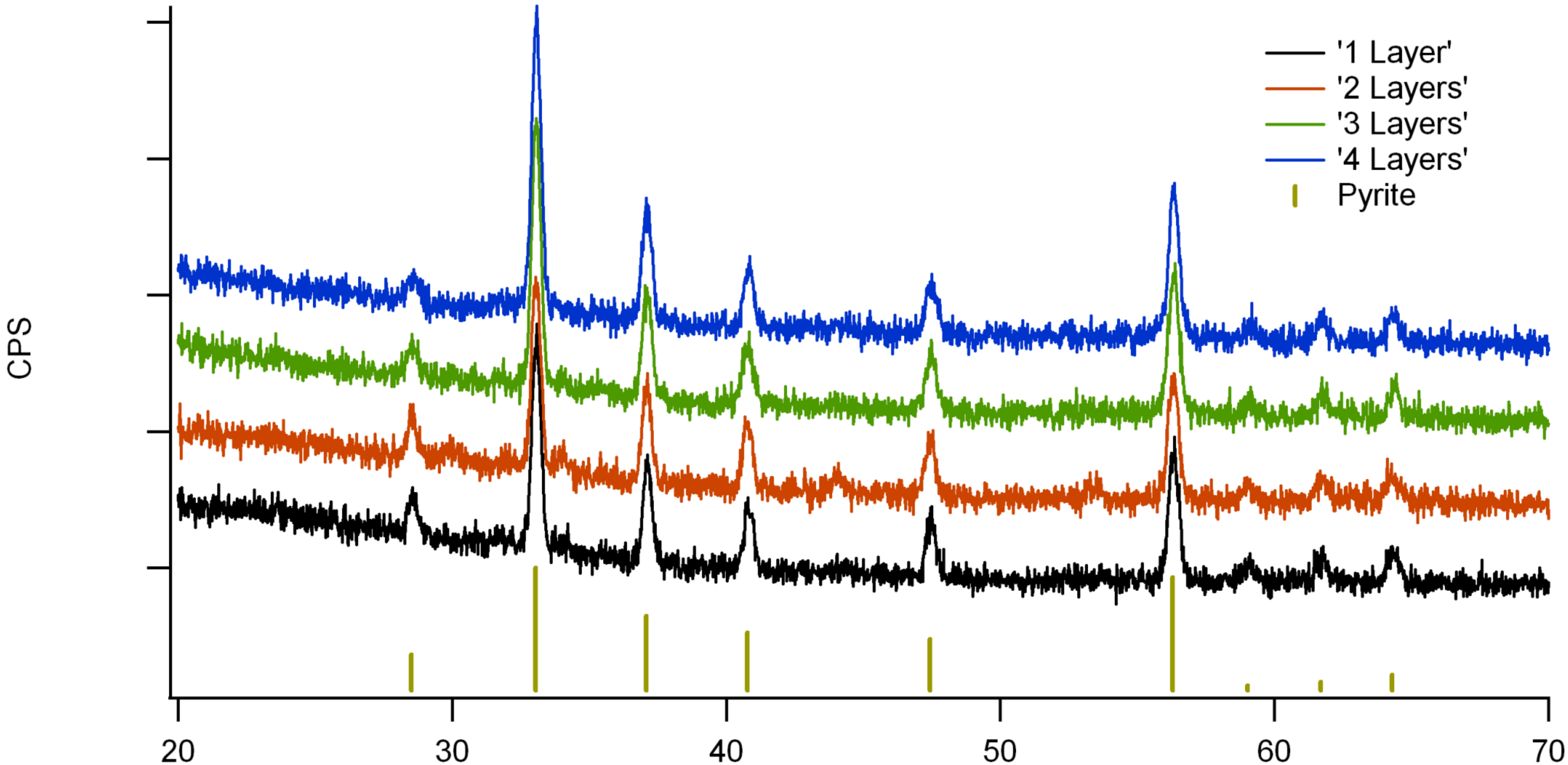
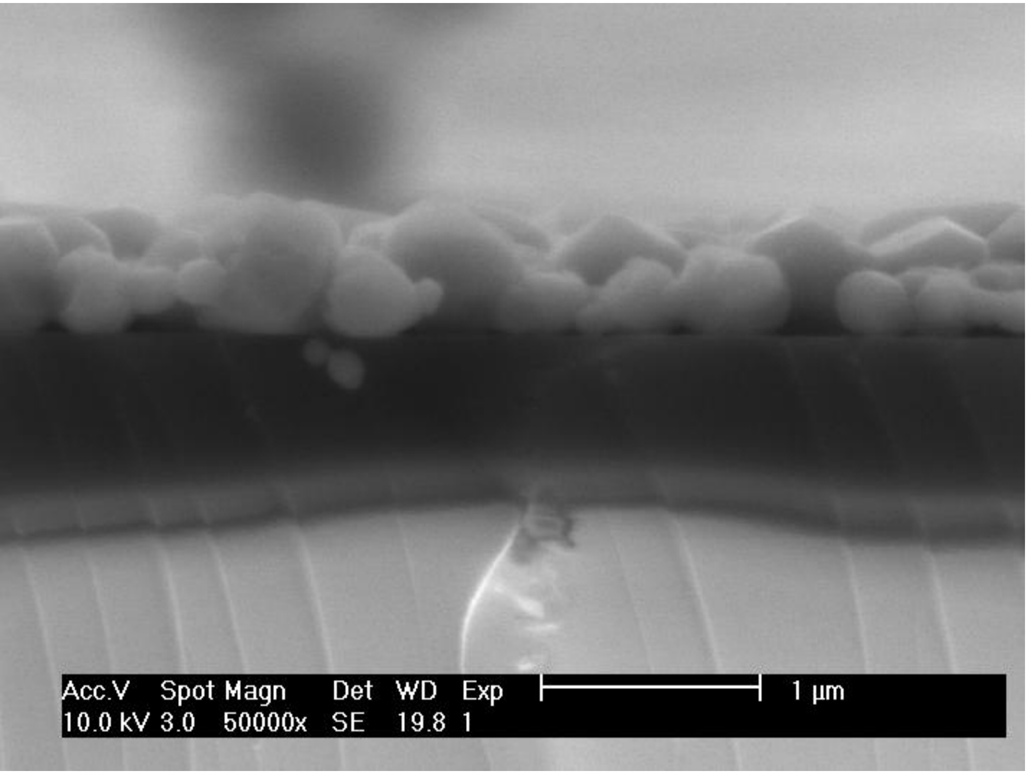
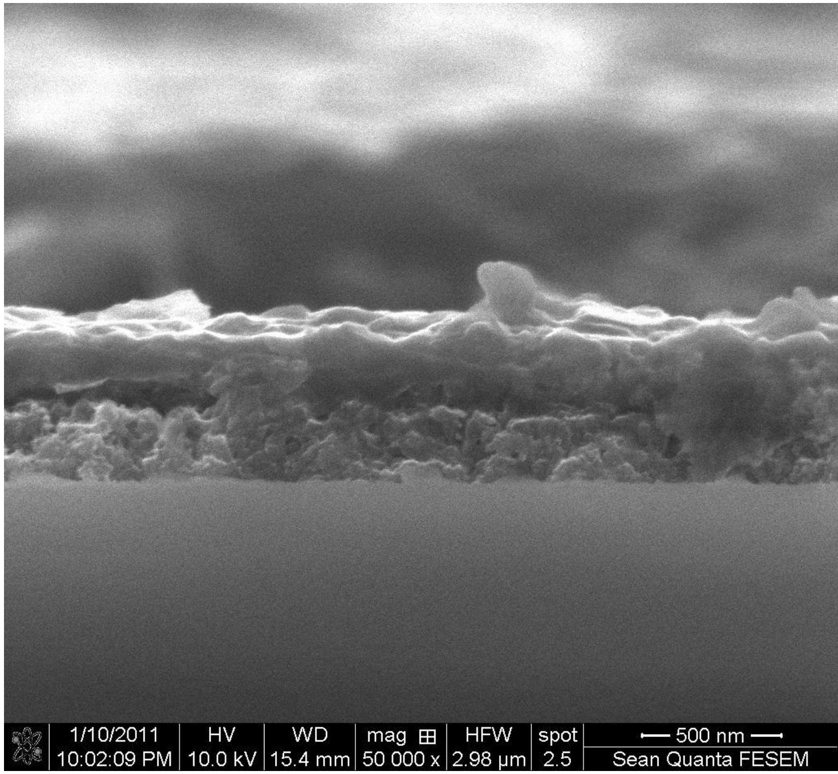
Sean Quanta FESEM

H_2S Annealing

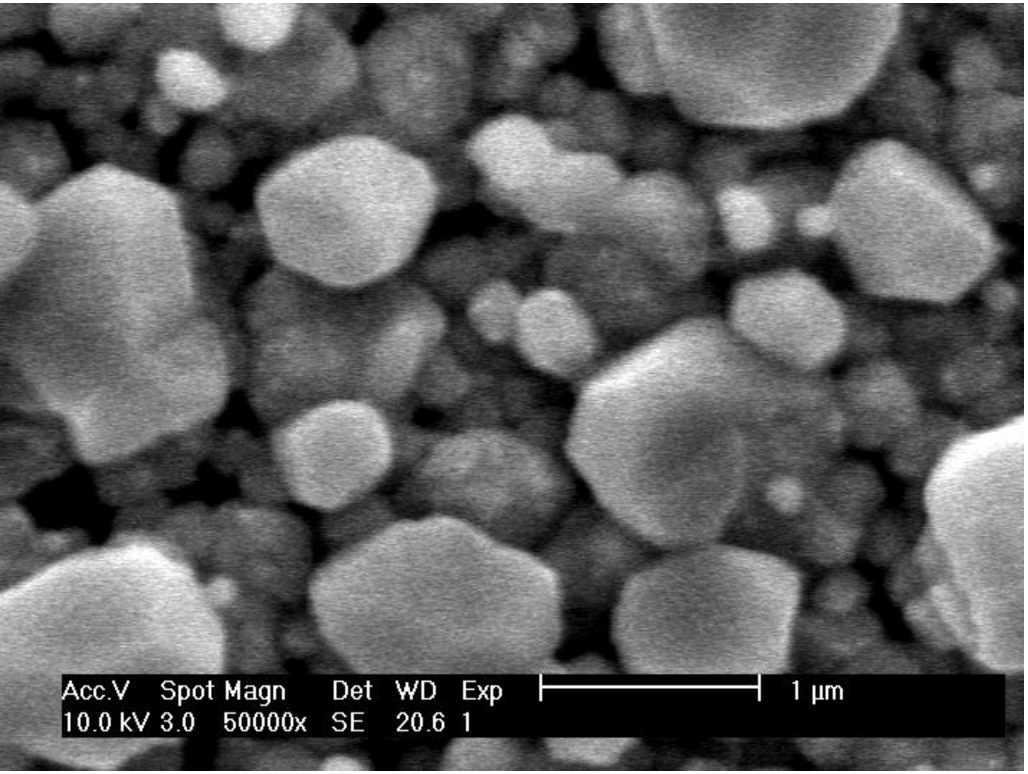


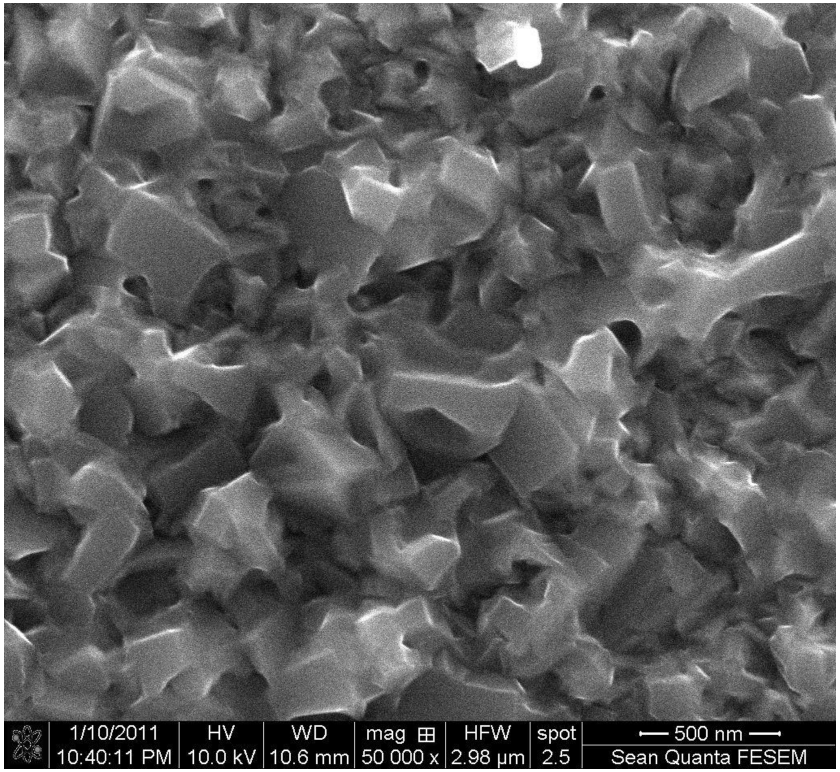
S_8 vs H_2S Annealing

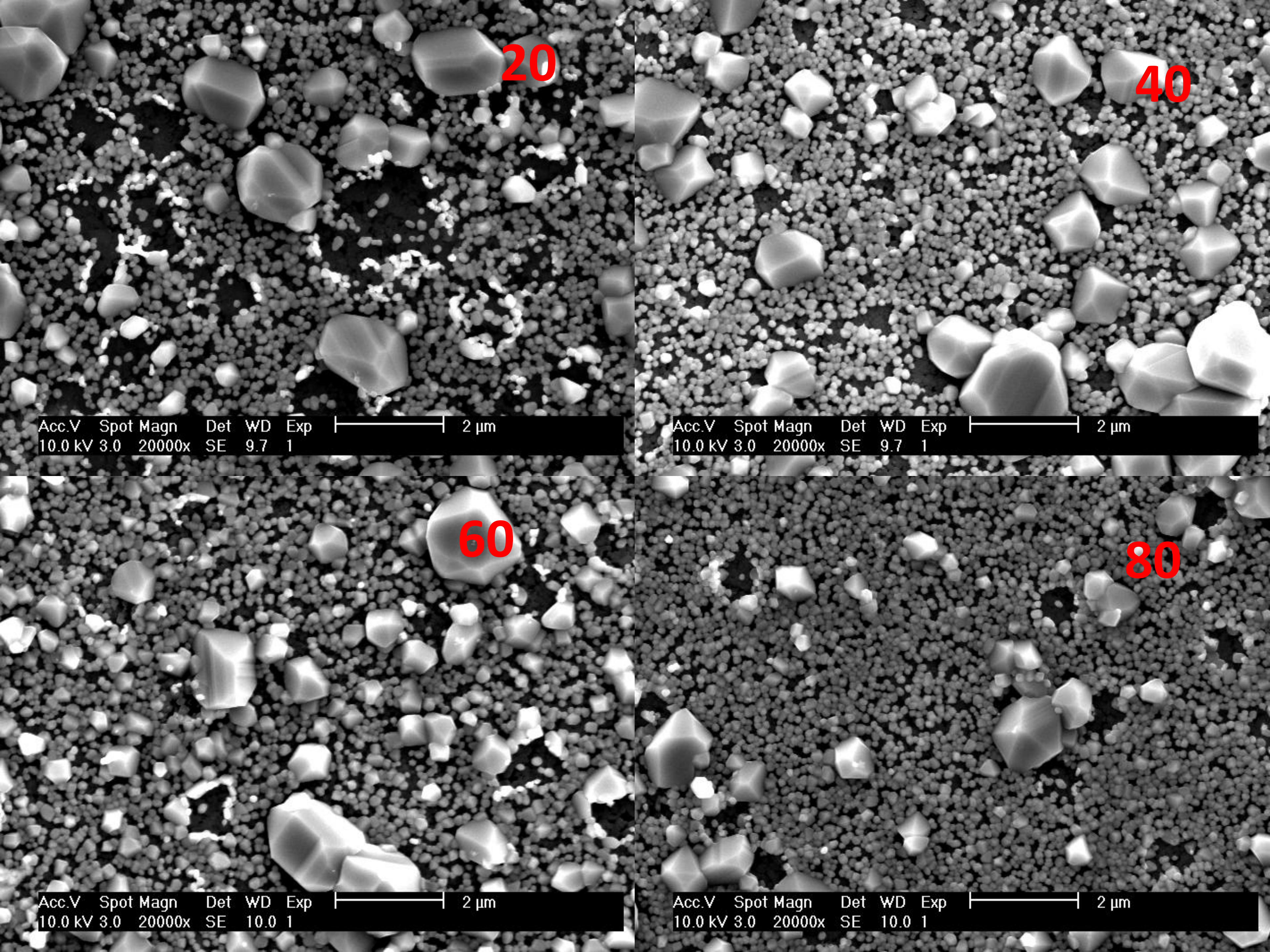


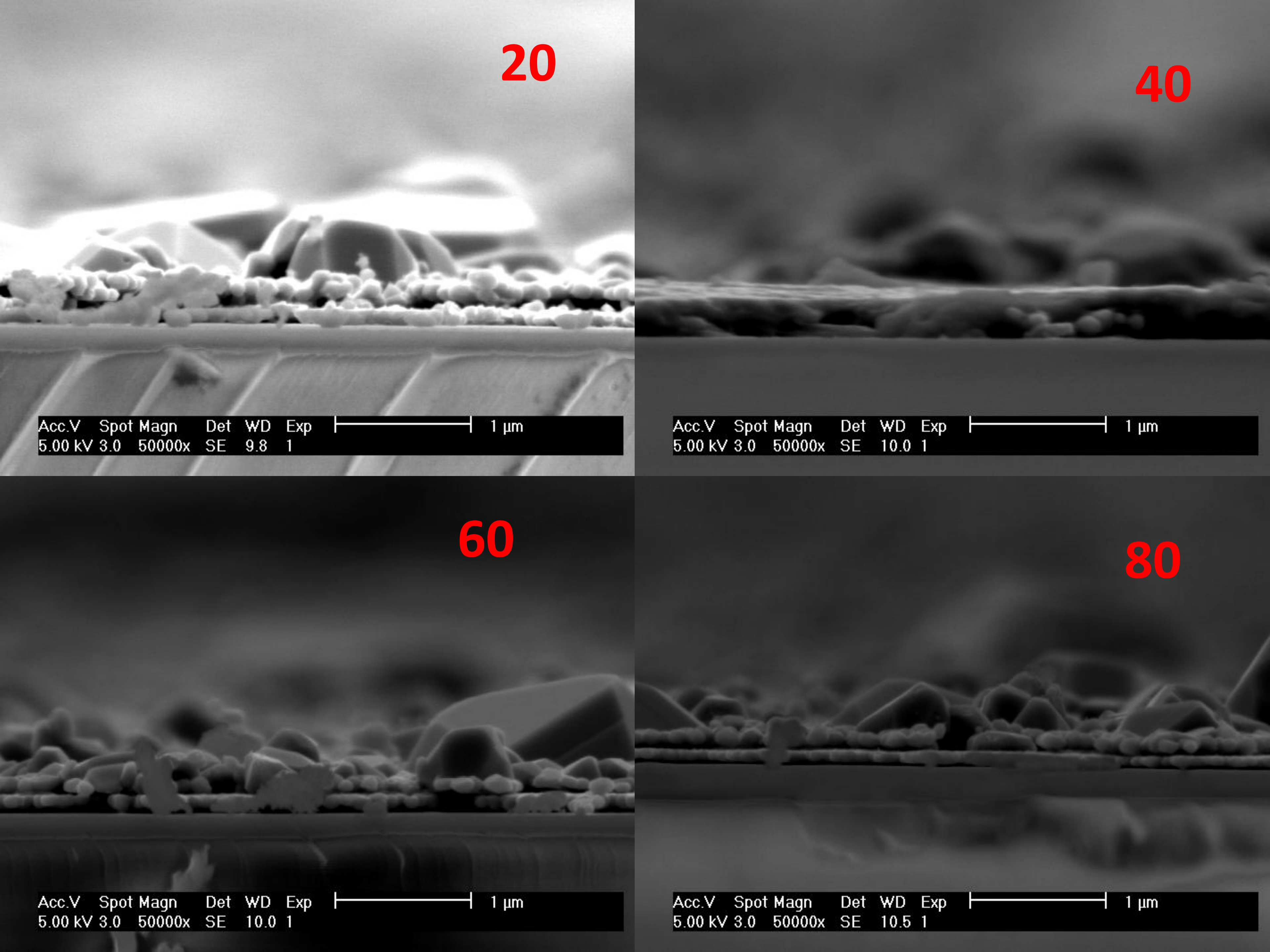


S_8 vs H_2S Annealing






20
Acc.V Spot Magn Det WD Exp 2 µm
10.0 kV 3.0 20000x SE 9.7 1
40
Acc.V Spot Magn Det WD Exp 2 µm
10.0 kV 3.0 20000x SE 9.7 1
60
Acc.V Spot Magn Det WD Exp 2 µm
10.0 kV 3.0 20000x SE 10.0 1
80
Acc.V Spot Magn Det WD Exp 2 µm
10.0 kV 3.0 20000x SE 10.0 1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5.00 kV 3.0 50000x SE 9.8 1
40
Acc.V Spot Magn Det WD Exp 1 µm
5.00 kV 3.0 50000x SE 10.0 1
60
Acc.V Spot Magn Det WD Exp 1 µm
5.00 kV 3.0 50000x SE 10.0 1
80
Acc.V Spot Magn Det WD Exp 1 µm
5.00 kV 3.0 50000x SE 10.5 1

Future Work

- Pyrite NCs soluble in non-chlorinated solvent.
  - Eventually move away from synthesis involving halogen precursor.
  - Proceed to film and device work once metrics are met to use the NC inks.
- Molecular Ink films need electrical and optical characterization for multiple thicknesses.
  - Devices will ultimately determine optimal thickness for molecular ink thin films.