

CVD and Annealing

Nick Berry

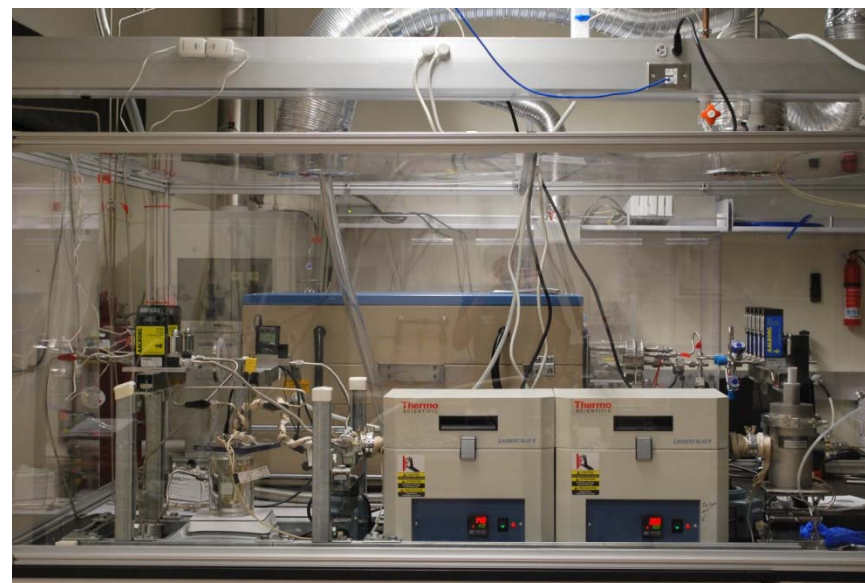
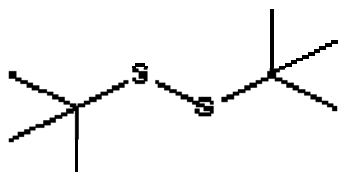
NSF Meeting #1

8/3/2010

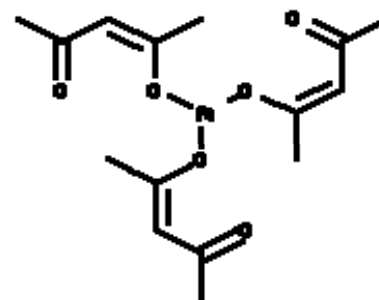
CVD Setup

- Ar Carrier Gas
 - Or Ar/H₂ mixtures
- Temperatures
 - Reaction: 300-350C
- Flow Rates
- Precursors

Tert-butyl disulfide(TBDS) – liquid

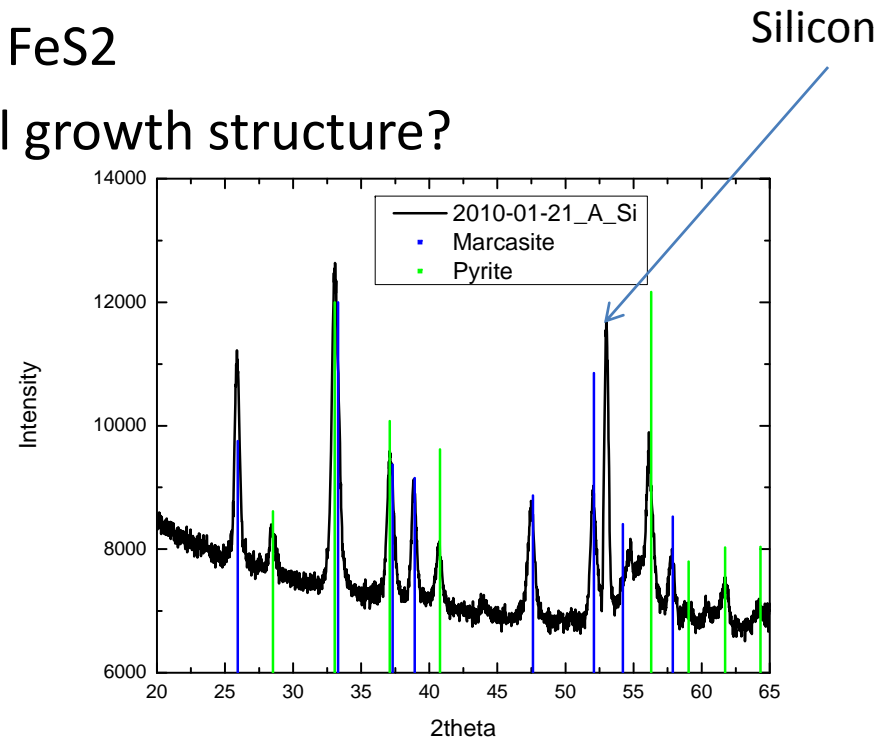
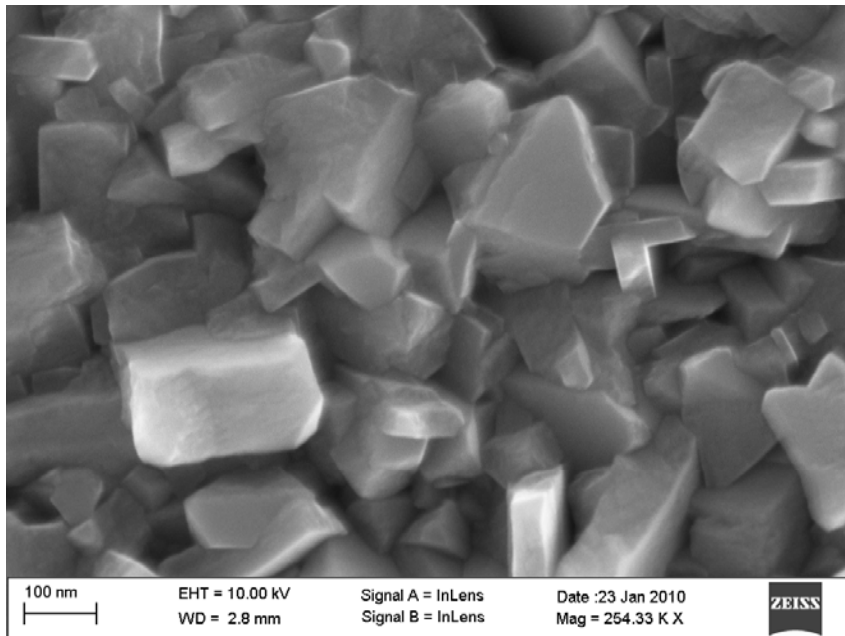


Iron(III) Acetylacetonate - solid



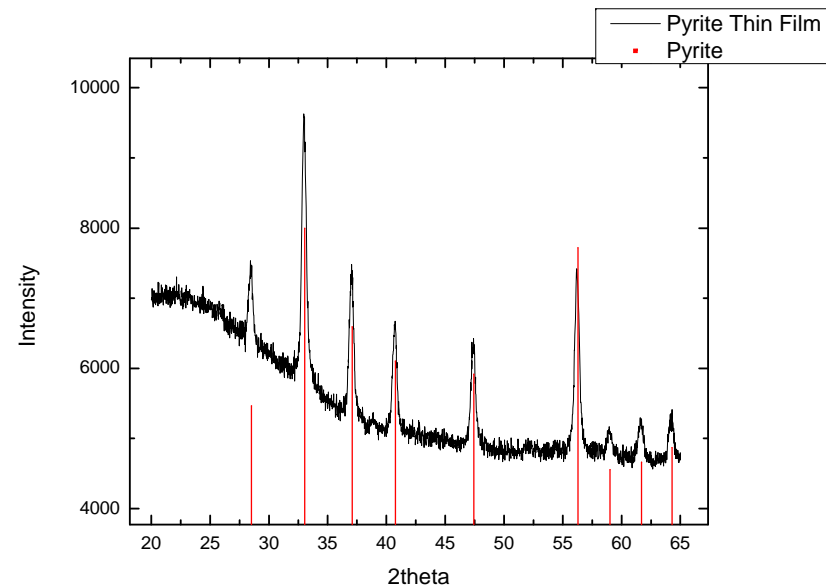
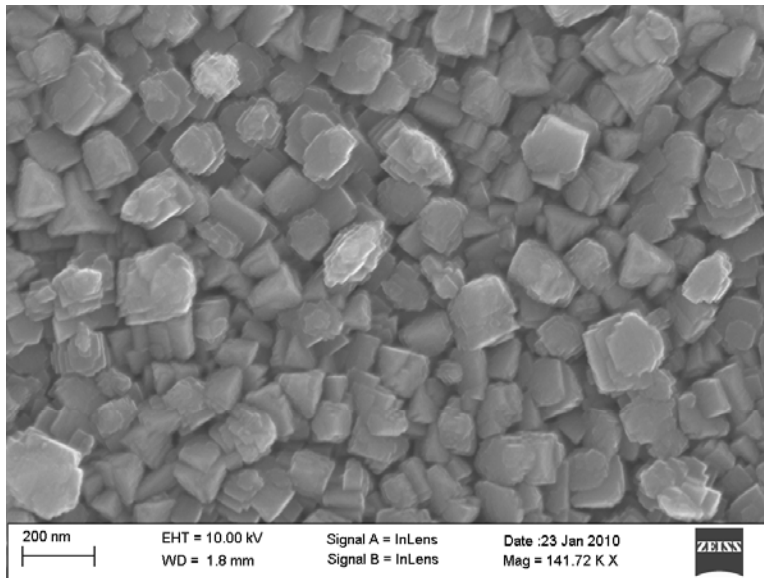
Typical Surface Growth

- Good coverage of surface growth over large area
- On quartz and silicon
 - Mostly Marcasite, with some pyrite
 - Marcasite: Orthorhombic FeS₂
- Why is marcasite the preferential growth structure?



Growth on Glass

- Same conditions as previous reactions on quartz
- Pure pyrite only on glass
 - Many other substrates tried
- XPS indicates lots of sodium leaching into film, which we believe to be the cause.
 - How does this happen?



Various Substrates

- Attempting to find best substrate
 - Ti, Cr, Moly, Si, Quartz, Glass, Ni, Stainless steel alloys, Al, Al alloy...
- Silicon has atomic distances that match to pyrite, yet marcasite still forms
- How does it nucleate? Ideal surface?
- So far Moly and pure Aluminum will survive conditions, but many more tests are needed.
 - Moly will form thin MoS₂ layer

Annealing in Elemental Sulfur

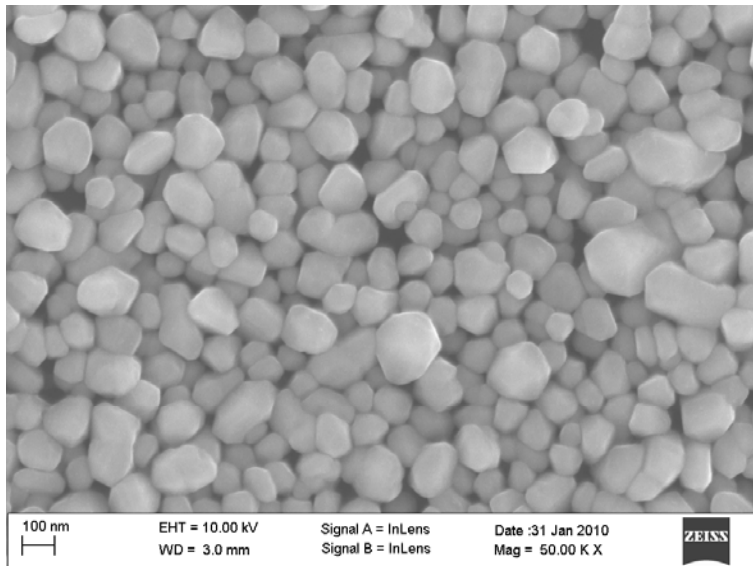
- Conversion from Marcasite to Pyrite above $\sim 450\text{C}$
 - Dependent upon Time, Temperature, and Sulfur concentration
 - Dependence on Sulfur concentration
 - More sulfur = faster conversion
 - Why?
- Must anneal in Sulfur to prevent sulfur loss. Above 700, still get conversion to pyrrhotite(FeS_{1-x})
- Testing $\text{H}_2\text{S}/\text{H}_2$ Annealing furnace



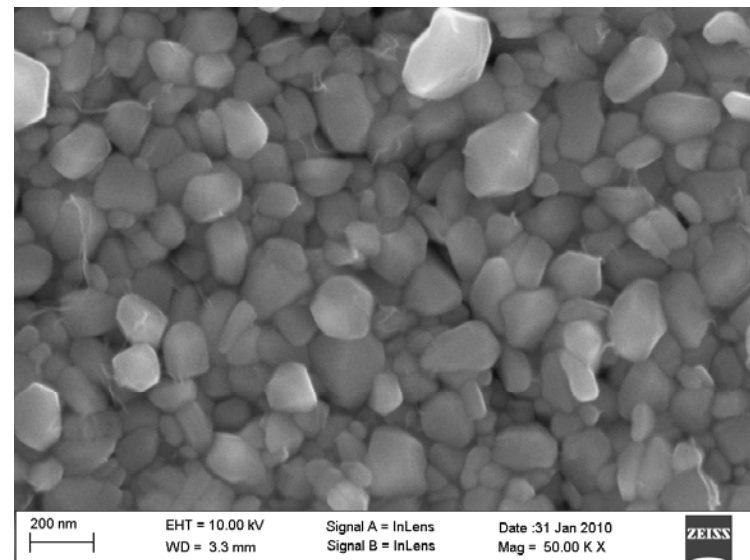
Pinhole/Crack Formation

- Increase in Grain Size during annealing
- However decreases connectivity forming pinholes or cracks
- How do grains decide to form, why do they pull apart?
- Hall Effect setup being tested to determine mobility

500 on Glass



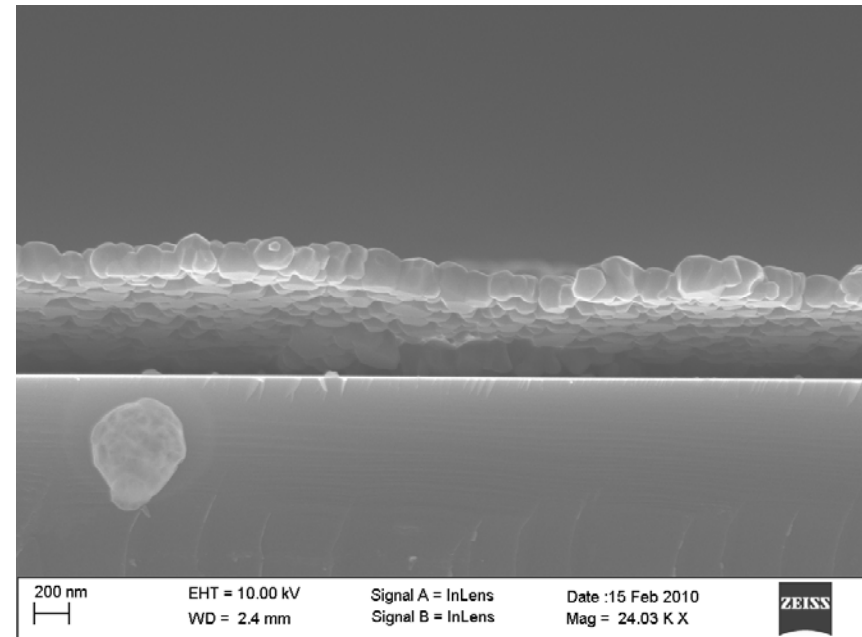
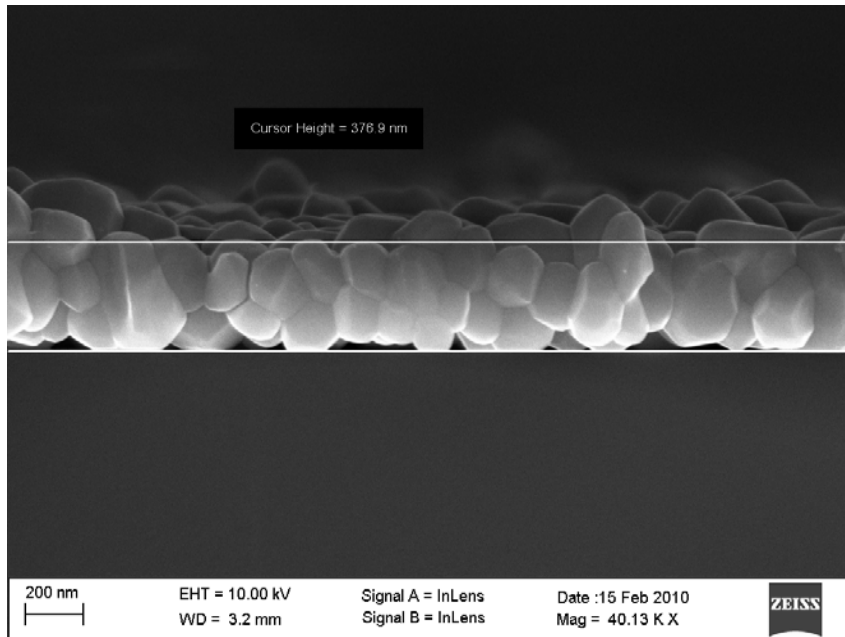
500 on Si



De-wetting

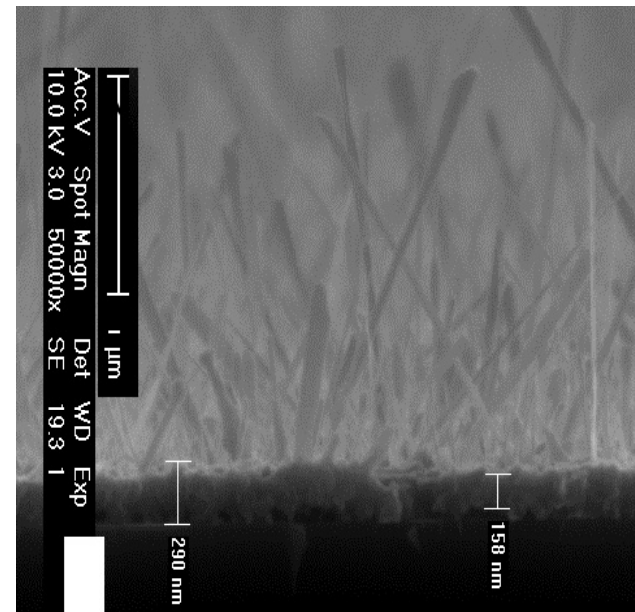
- Very well connected to surface initially out of CVD
- Films tend to de-wet from surfaces when they are annealed
- More experiments needed to find an ideal substrate, and how de-wetting varies

650 on Silicon



Ar+H₂ mixture as carrier gas

- Mostly pyrite instead of only marcasite
 - Mechanism previously investigated in literature
- Unexpected Nanowire Formation
 - Not mentioned in any literature
 - More research needs to be done to investigate what they are and how they form
- Not currently desired



Conclusion

- Surface growth of pyrite thin films possible via CVD and annealing in a sulfur atmosphere
- Good grain growth and impurity phase reduction can be seen upon annealing
- Many questions remain
 - Growth mechanisms for pyrite/marcasite
 - Substrate dependence of growth
 - Ideal conditions for marcasite conversion
 - How to prevent pinhole/crack formation while maintaining well connected large crystalline pyrite grains.