Iron Sulfur Molecular Inks for Pyrite Thin-Films

Amanda Weber
Pyrite Collaboration Meeting
12-7-2010
Outline

- Motivation
- Synthesis
- H₂S annealing
- S₂ annealing
- Future Work
Motivation for Molecular Inks

- **Goal:** Find a convenient method for film deposition that has the correct composition, phase purity, and grain structure

- Benefits of Using Molecular Ink approach:
  - Solution based approaches are easier to scale up
  - Well controlled stoichiometry and minimal to no contamination (elemental precursors)
  - Simple film deposition (spin casting, doctor blading, drop casting)
Using dimethyl sulfoxide (DMSO) and ethanolamine as a solvent mixture to dissolve elemental precursors

**Motivation:** Paper by Mitzi stating that a mixture of DMSO and ethanolamine was an effective wetting agent for thermally oxidized silicon substrates; less toxic than hydrazine

**Previous molecular ink approaches:**
- CZTS (copper zinc tin sulfur/selenide) films: Dissolved elemental precursors in hydrazine and obtained devices with over 9.6% efficiency (as opposed to previously obtained efficiencies of 3.2% and 6.7%)
- CIGS (copper indium gallium sulfur/selenide): "" in hydrazine and obtained efficiencies of 10.1%
Synthesis Process Inside N$_2$ Glovebox

- Each layer is roughly 150 nm
- 200 °C is ideal pre-baking temperature since the boiling points of DMSO and ethanolamine are 189 °C and 170 °C respectively; by FT-IR all organics seem to be removed

Form mixture of DMSO and ethanolamine (6.5:1)

Dissolve S (1 M) in solution and stir for >6 hours (at room temp)

Add Fe (0.33 M) and stir for >24 hrs.

Spin coat at 1500 rpm for 60s

Pre-bake on hotplate at 200 °C for 5 min.

Anneal in S (25 mg) at 550 °C for 6 hours
H₂S 450 °C, 15hrs
H$_2$S, 500°C, 15hrs
H₂S, 550°C, 15hrs
S$_2$ Annealing X-Ray Diffraction Patterns
500 °C, 6hrs, 25 mg S

- Yields pure pyrite, but morphology is not ideal
  - Little grain growth
  - Not well connected grains
550°C, 6hrs, 25mg S, 7 layers
$600^\circ C, 6h, 25mg S$
15mg S, 550 °C, 6hr, 7layers
550 °C, 6hrs, 100 mg S, 7 layers
Future Work

- Determining chemical composition
  - Thermogravimetric Analysis
  - Differential Scanning Calorimetry
  - Mass Spectrometry
  - Powder X-Ray Diffraction
  - Nuclear Magnetic Resonance
  - XPS – look for contaminants (O, C, N, Na)
- $S_2$ annealing: vary S content, temperature, total cook time
- $H_2S$ annealing: time, temperature
- Exchanging ethanolamine with ethylene glycol (less toxic)
- Alloying with Zn