

The Formation of Tin Chalcogenide Nanomaterials

Thao H. Nguyen, Dr. Bernadette A. Hernandez-Sanchez,
Andrew A. Golembeski, Chris T. Stevens, Dr. Khalid M.
Hattar



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Sandia National Laboratories
Advanced Materials Laboratory

1001 University Blvd SE
Albuquerque, NM 87106

huongthao44@gmail.com



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Safety In The Lab

Glove Box



Schlenk Line



Goggles



Safety Training Courses

- New Employee Orientation
- Chemical Safety Training
- Site -Specific Chemical Safety Training
- Cryogen Safety
- Pressure Safety Orientation
- Neo200
- ES&H Awareness
- Hazardous Waste & Environment Management Training.

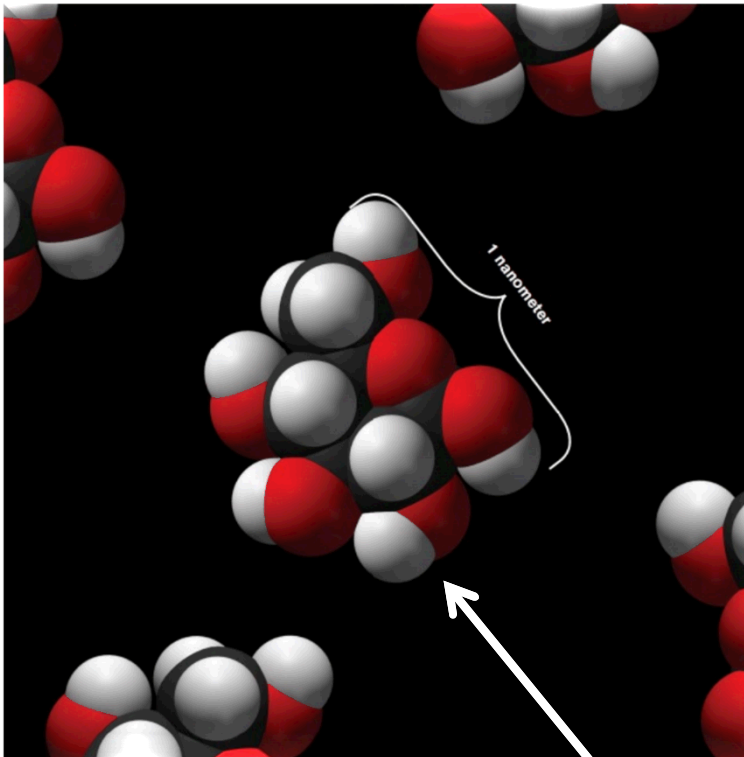
Lab Coat

Gloves



What is Nano?

One nanometer is a **BILLIONTH** of a meter.



$$\begin{array}{r}
 1 \times 10^{-9} \text{ of a meter} \\
 = \\
 1 \\
 \hline
 1,000,000,000
 \end{array}$$

A sugar molecule is about 1 nanometer wide.

Periodic Table of the Elements																		18 VIIIA 8A	
1 IA 1A																	2 He Helium 4.003		
3 Li Lithium 6.941	4 Be Beryllium 9.012											5 B Boron 10.811	6 C Carbon 12.011	7 N Nitrogen 14.007	8 O Oxygen 15.999	9 F Fluorine 18.998	10 Ne Neon 20		
11 Na Sodium 22.990	12 Mg Magnesium 24.305	3 IIIB 3B	4 IVB 4B	5 VB 5B	6 VIB 6B	7 VIIB 7B	8 VIII 8	9 VIII 8	10 VIII 8	11 IB 1B	12 IIB 2B	13 Al Aluminum 26.982	14 Si Silicon 28.086	15 P Phosphorus 30.974	16 S Sulfur 32.066	17 Cl Chlorine 35.453	18 Ar Argon 39.948		
19 K Potassium 39.098	20 Ca Calcium 40.078	21 Sc Scandium 44.956	22 Ti Titanium 47.88	23 V Vanadium 50.942	24 Cr Chromium 51.996	25 Mn Manganese 54.938	26 Fe Iron 55.933	27 Co Cobalt 58.933	28 Ni Nickel 58.693	29 Cu Copper 63.546	30 Zn Zinc 65.39	31 Ga Gallium 69.723	32 Ge Germanium 72.61	33 As Arsenic 74.922	34 Se Selenium 78.09	35 Br Bromine 79.904	36 Kr Krypton 84.80		
37 Rb Rubidium 84.468	38 Sr Strontium 87.62	39 Y Yttrium 88.906	40 Zr Zirconium 91.224	41 Nb Niobium 92.906	42 Mo Molybdenum 95.94	43 Tc Technetium 98.907	44 Ru Ruthenium 101.07	45 Rh Rhodium 102.906	46 Pd Palladium 106.42	47 Ag Silver 107.868	48 Cd Cadmium 112.411	49 In Indium 114.818	50 Sn Tin 118.71	51 Sb Antimony 121.760	52 Te Tellurium 127.6	53 I Iodine 126.904	54 Xe Xenon 131.29		
55 Cs Cesium 132.905	56 Ba Barium 137.327	57-71 Lanthanide Series	72 Hf Hafnium 178.49	73 Ta Tantalum 180.948	74 W Tungsten 183.85	75 Re Rhenium 186.207	76 Os Osmium 190.23	77 Ir Iridium 192.22	78 Pt Platinum 195.08	79 Au Gold 196.967	80 Hg Mercury 200.59	81 Tl Thallium 204.383	82 Pb Lead 207.2	83 Bi Bismuth 208.980	84 Po Polonium [209.962]	85 At Astatine 209.987	86 Rn Radon 222.018		
87 Fr Francium 223.020	88 Ra Radium 226.025	89-103 Actinide Series	104 Rf Rutherfordium [261]	105 Db Dubnium [262]	106 Sg Seaborgium [266]	107 Bh Bohrium [264]	108 Hs Hassium [269]	109 Mt Meitnerium [268]	110 Ds Darmstadtium [269]	111 Rg Roentgenium [272]	112 Cn Copernicium [277]	113 Uut Ununtrium unknown	114 Fl Flerovium [289]	115 Uup Ununpentium unknown	116 Lv Livermorium [298]	117 Uus Ununseptium unknown	118 Uuo Ununoctium unknown		
Lanthanide Series			57 La Lanthanum 138.906	58 Ce Cerium 140.115	59 Pr Praseodymium 140.908	60 Nd Neodymium 144.24	61 Pm Promethium 144.913	62 Sm Samarium 151.966	63 Eu Europium 157.25	64 Gd Gadolinium 157.25	65 Tb Terbium 158.925	66 Dy Dysprosium 162.50	67 Ho Holmium 164.930	68 Er Erbium 167.26	69 Tm Thulium 168.934	70 Yb Ytterbium 173.04	71 Lu Lutetium 174.967		
Actinide Series			89 Ac Actinium 227.028	90 Th Thorium 232.038	91 Pa Protactinium 231.036	92 U Uranium 238.029	93 Np Neptunium 237.048	94 Pu Plutonium 244.064	95 Am Americium 243.061	96 Cm Curium 247.070	97 Bk Berkelium 247.070	98 Cf Californium 251.080	99 Es Einsteinium [254]	100 Fm Fermium 257.095	101 Md Mendelevium 258.1	102 No Nobelium 259.101	103 Lr Lawrencium [262]		

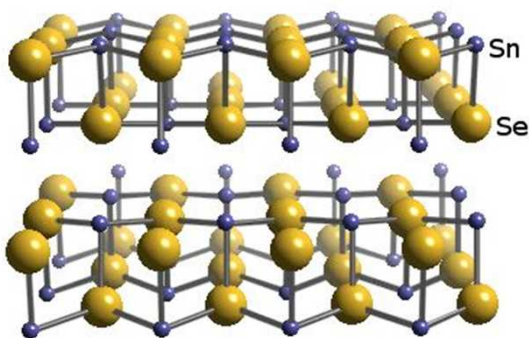
- 4

Tin Chalcogenides

Tin Selenide

- $E_g = 0.2\text{eV}$ (Bulk)
- Narrow band gap semiconductor
- Orthorhombic
- CELL: $a=4.313\text{\AA}$ $b=11.703\text{\AA}$
 $c=4.313\text{\AA}$ $\langle 90.0^\circ \times 90.0^\circ \times 90.0^\circ \rangle$
- $\text{Vol} = 217.7$
- $\text{Density(c)} = 6.03$

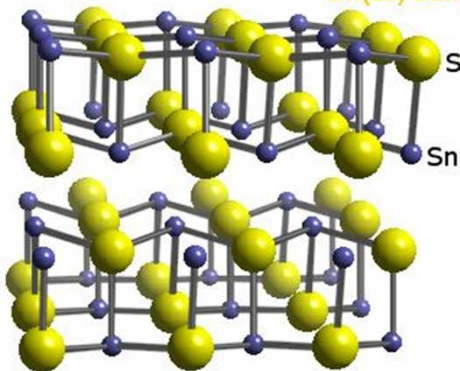
tin(II) selenide



Tin Sulfide

- $E_g = 1.0\text{-}2.3\text{ eV}$ (Bulk)
- Semiconductor
- Orthorhombic
- CELL: $a=4.175\text{\AA}$ $c=11.42\text{\AA}$
 $c=4.125\text{\AA}$ $\langle 90.0^\circ \times 90.0^\circ \times 90.0^\circ \rangle$
- $\text{Vol} = 253.3$
- $\text{Density(c)} = 6.46$

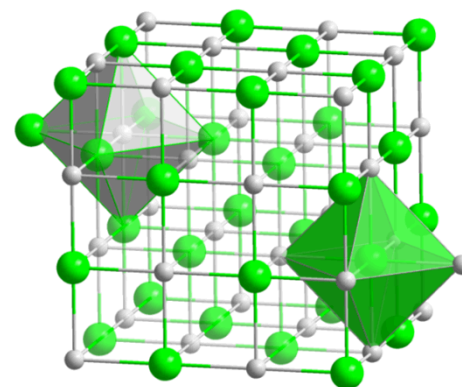
tin(II) sulfide



NaCl Crystal Structure

Tin Telluride

- $E_g = 0.18\text{eV}$ (Bulk)
- Narrow band gap semiconductor
- Cubic
- CELL: $a=6.327\text{\AA}$ $b=6.327\text{\AA}$
 $c=6.32751\text{\AA}$ $\langle 90.0^\circ \times 90.0^\circ \times 90.0^\circ \rangle$
- $\text{Vol} = 196.7$
- $\text{Density(c)} = 5.064$



Applications

$*E = Se, S, Te$

SnE is a potential product that can be use for...



Thermoelectrics

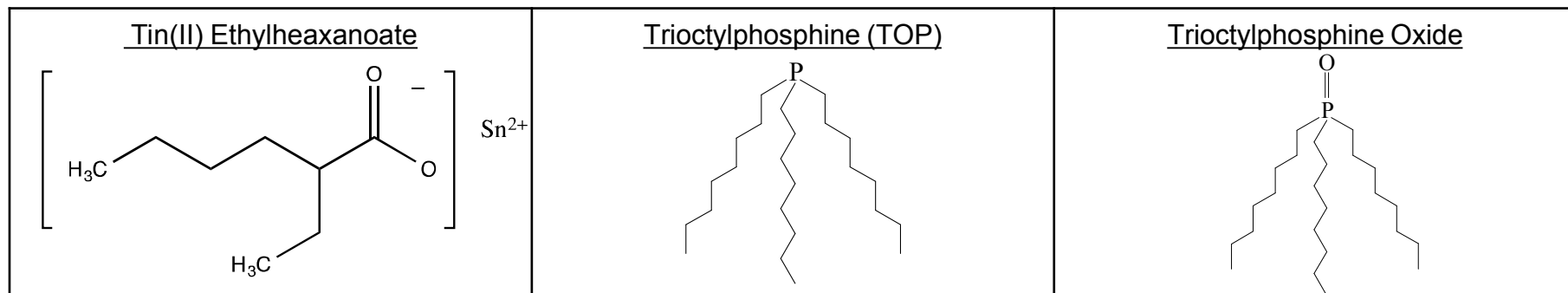


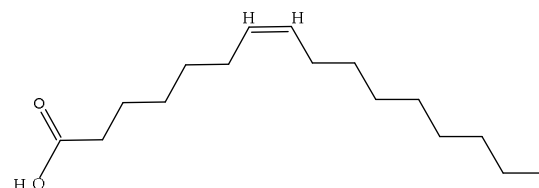
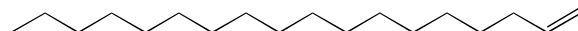
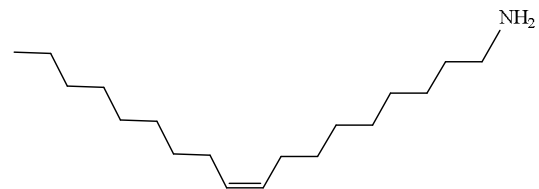
Solar Panels



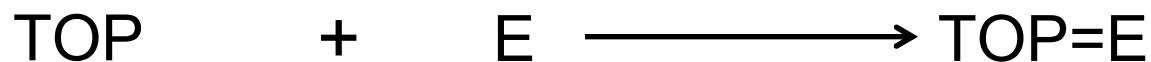
Infrared

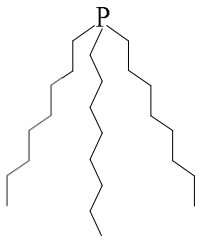
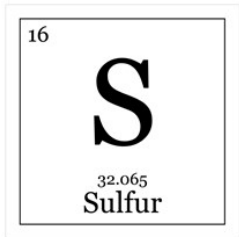
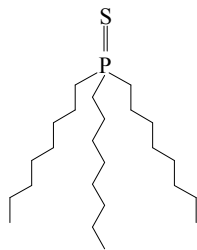
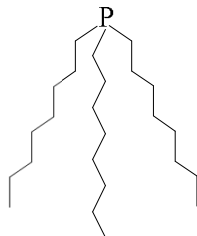
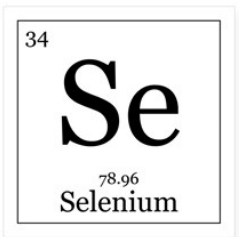
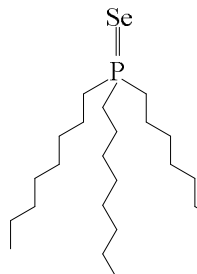
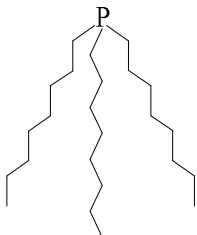
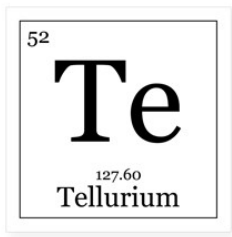
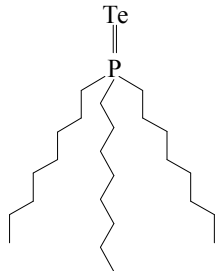
Precursors



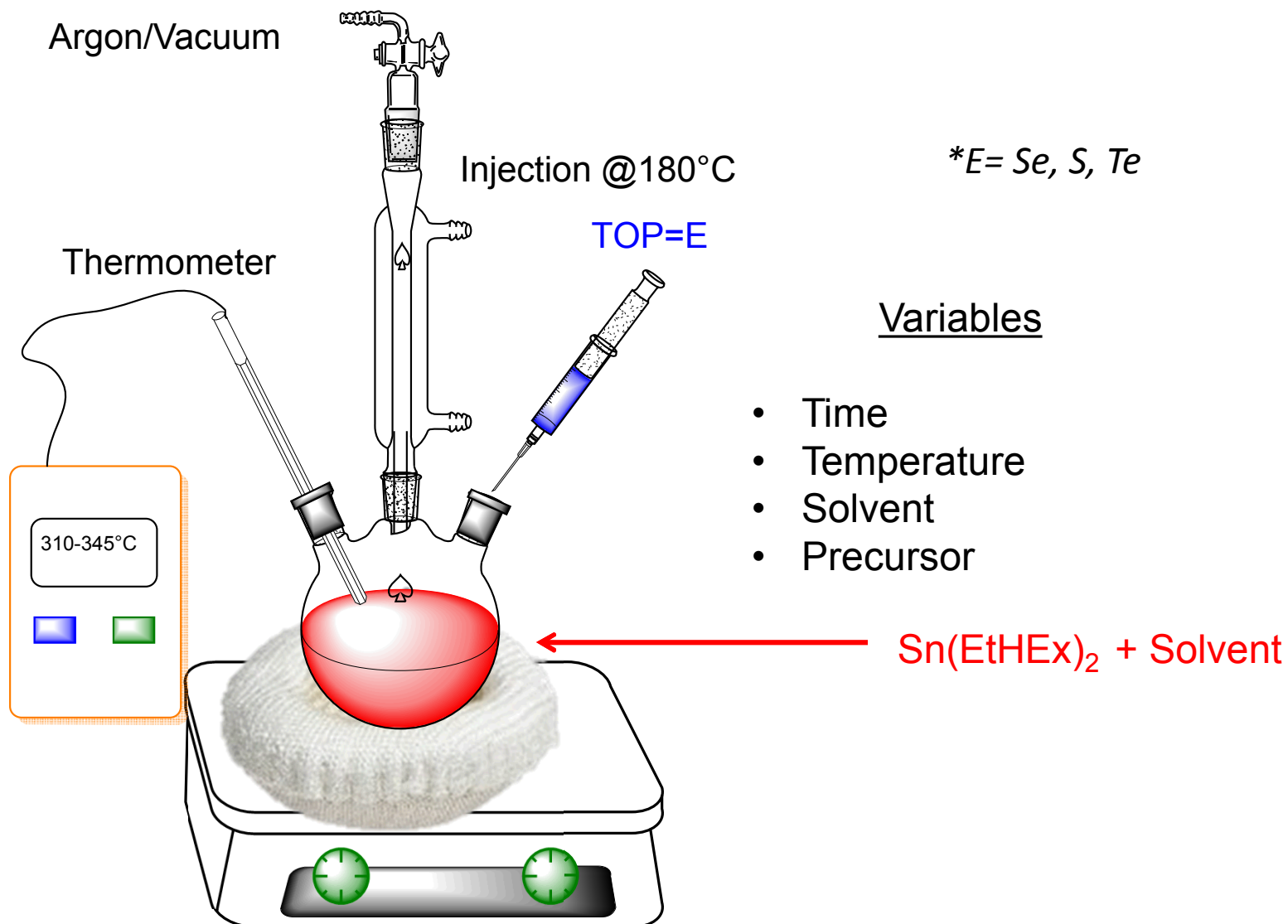
Coordinating Solvents:	NON-Coordinating Solvent:
<u>Oleic Acid</u> 	<u>Octadecene</u> 
<u>Oleylamine</u> 	Precursors explored for shape control

Preparation of TOP=E Precursors



Trioctylphosphine 	Sulfur 	Trioctylphosphine Sulfide 
Trioctylphosphine 	Selenium 	Trioctylphosphine Selenide 
Trioctylphosphine 	Tellurium 	Trioctylphosphine Telluride 

How To Synthesize SnE Nanomaterials



Time/Temperature

- Injection at 180 °C with TOP=E
- Max Temps between 340–350 °C
- Total Time (1hr 30 minutes)



Characterization



PXRD (Powder X-ray Diffraction):
Identifies crystal structure & phase

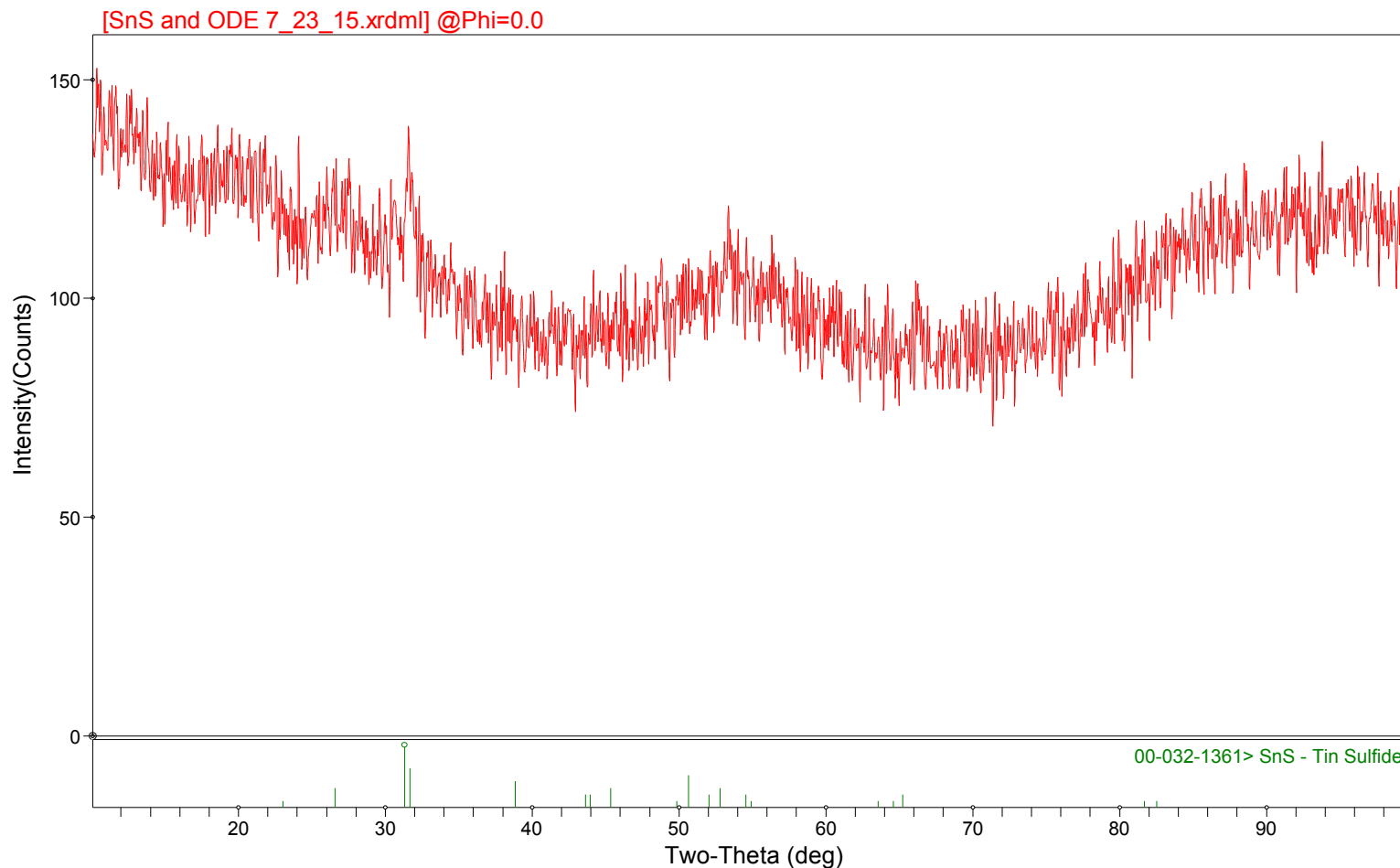
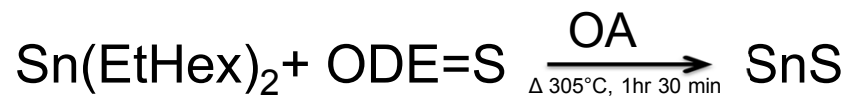


TEM (Transmission Electron Microscopy): Shows nanoparticle size & shape

SnE Reactions

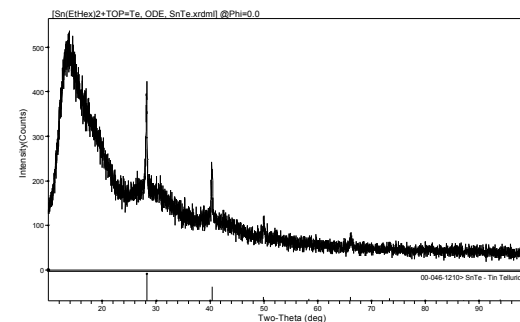
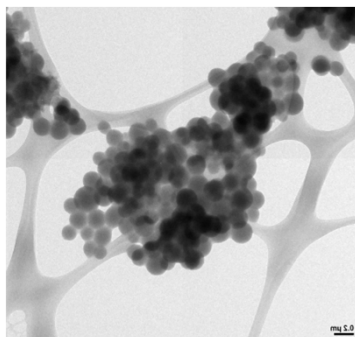
Tin Precursor	Chalcogen Precursor	Reaction Time	Solvents	Max. Temp. (°C)	XRD Phase	TEM	
Sn(EtHex) ₂	TOP=Se	1 hr. 30 min.	OA	315	SnSe	Mixed Morph. (Sheets, organic)	✓
Sn(EtHex) ₂ (Temporal Study)	TOP=Se	1 hr. 30 min.	OA	331	SnSe	---	✓
Sn(EtHex) ₂	TOP=Te	1 hr. 5 min.	OA	270	SnTe	Organic Shape	✓
Sn(EtHex) ₂ (Temporal Study)	TOP=Te	1 hr. 35 min.	OA	338	SnTe	---	✓
Sn(EtHex) ₂	TOP=S	1 hr. 30 min.	OA	345	Mixed SnS	---	
Sn(EtHex) ₂	ODE + S	60 min.	OA	330	Amorphous	---	

Tin Sulfide Reaction with Octadecene

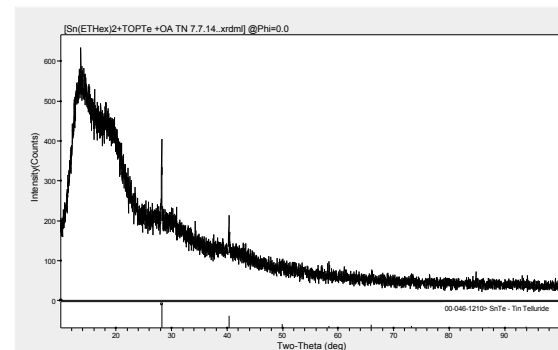
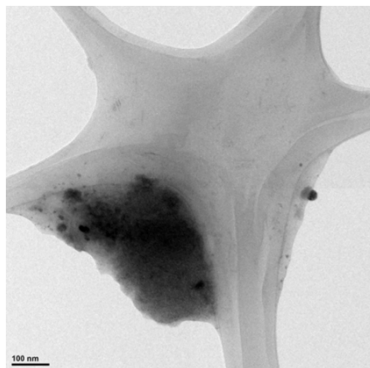


Tin Telluride Morphology

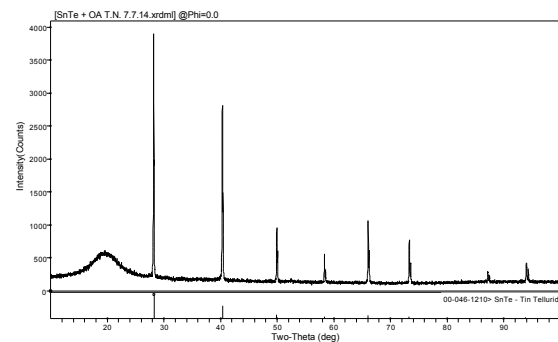
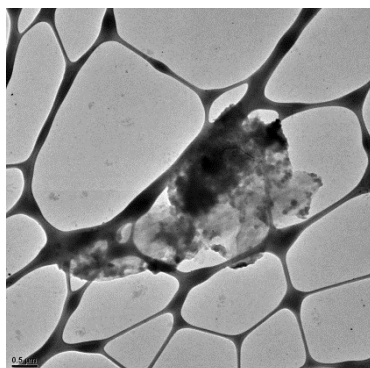
Octadecene



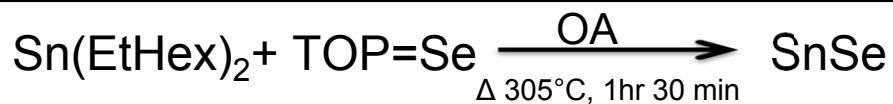
Oleylamine



Oleic Acid

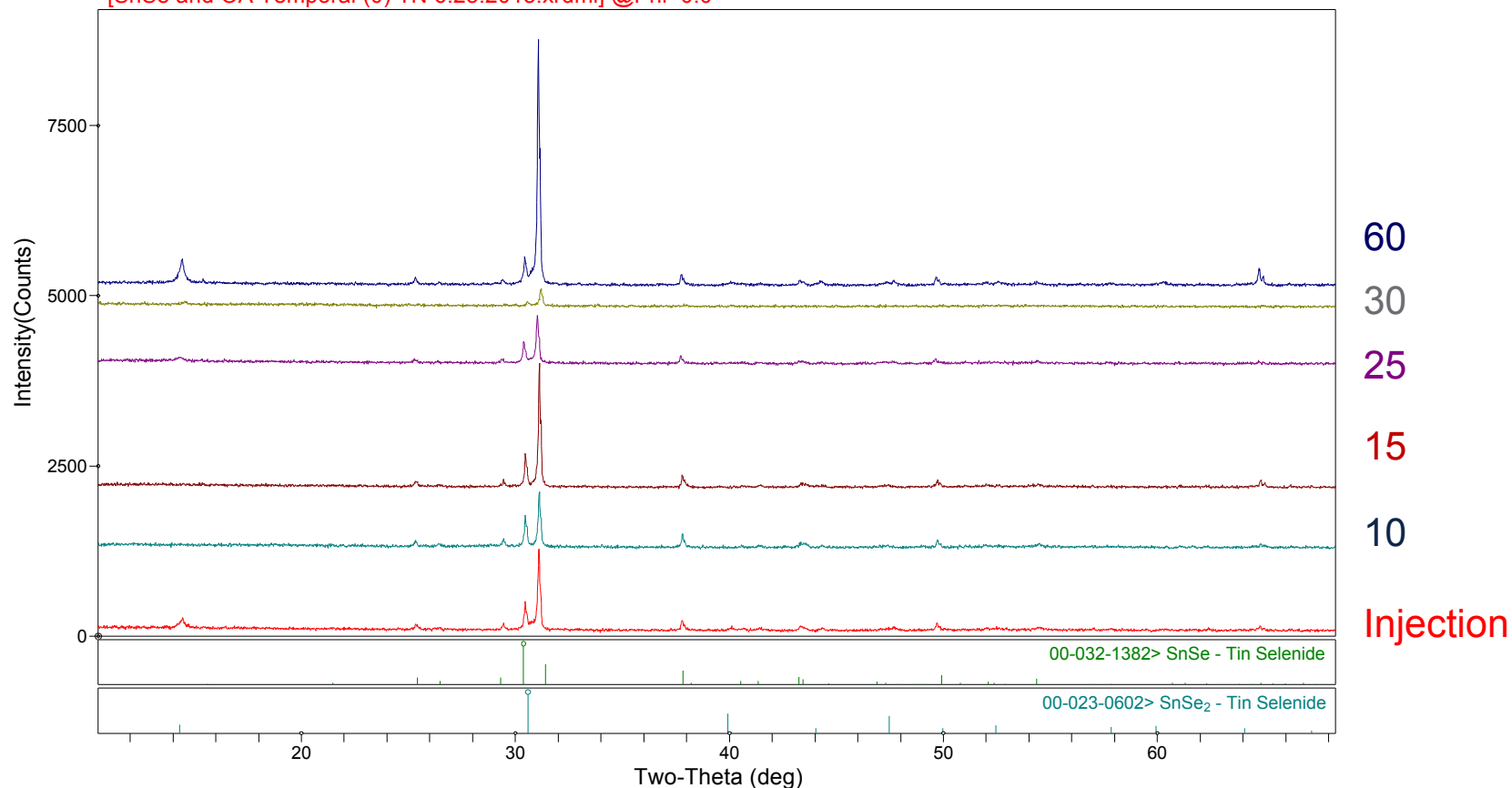


Temporal Study

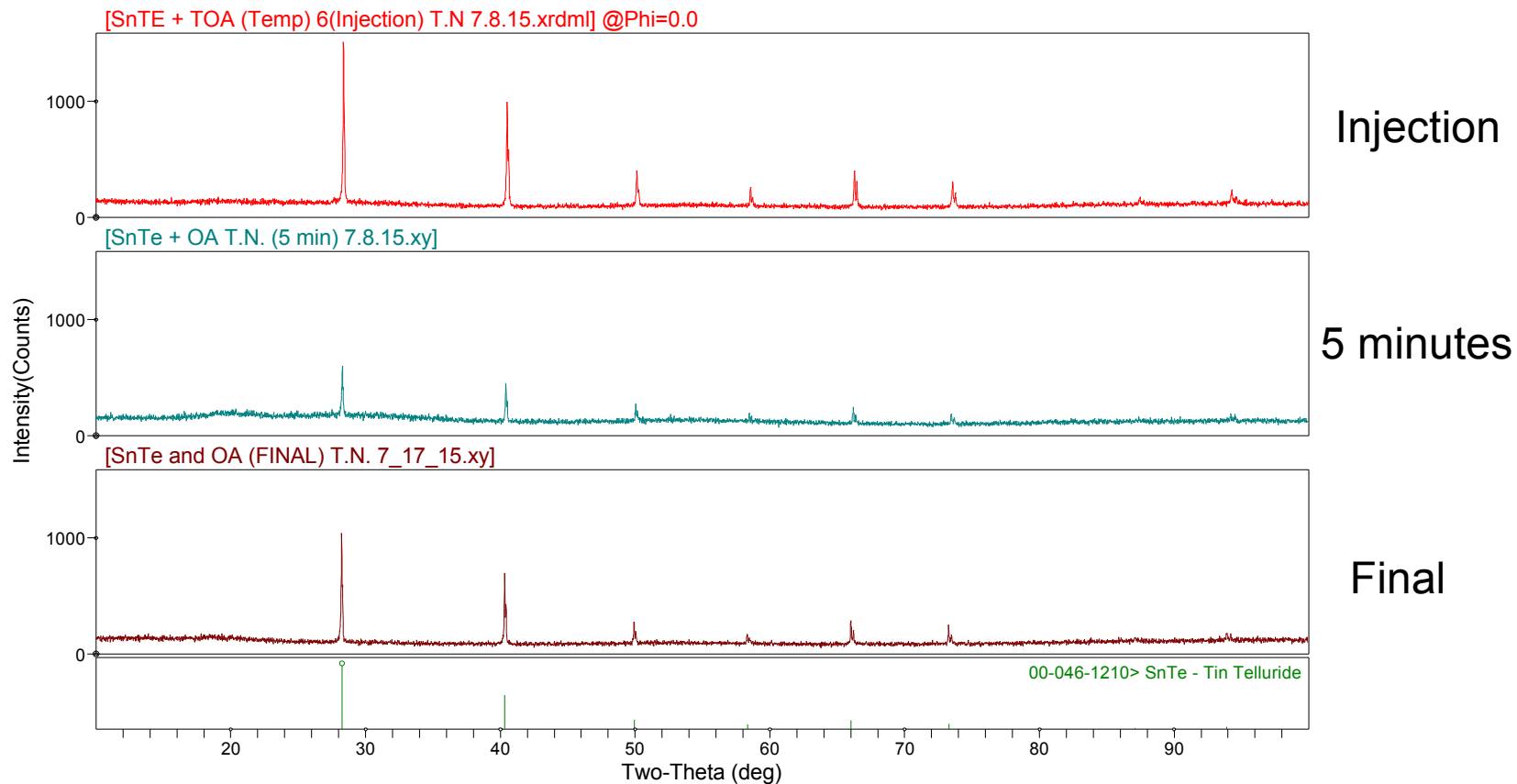
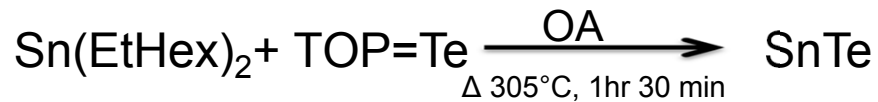


[SnSe and OA Temporal (10) T.N 6.23.15.xrdml] @Phi=0.0

[SnSe and OA Temporal (0) TN 6.25.2015.xrdml] @Phi=0.0

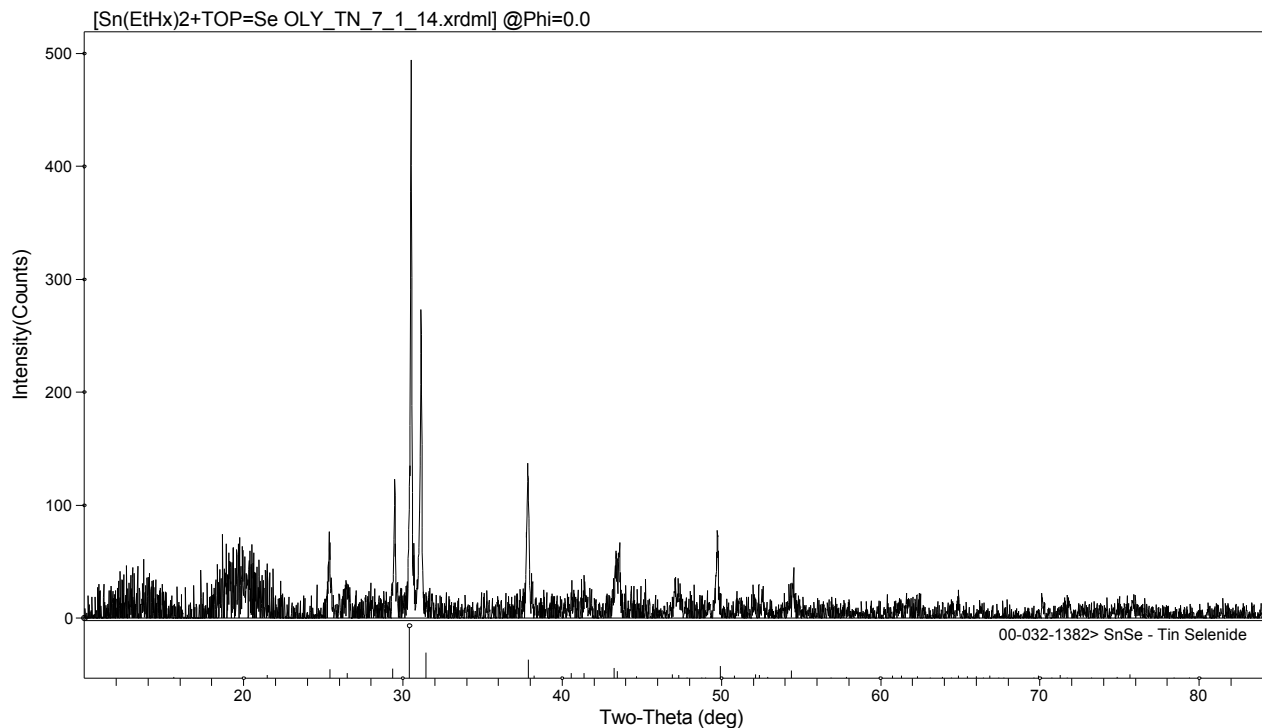
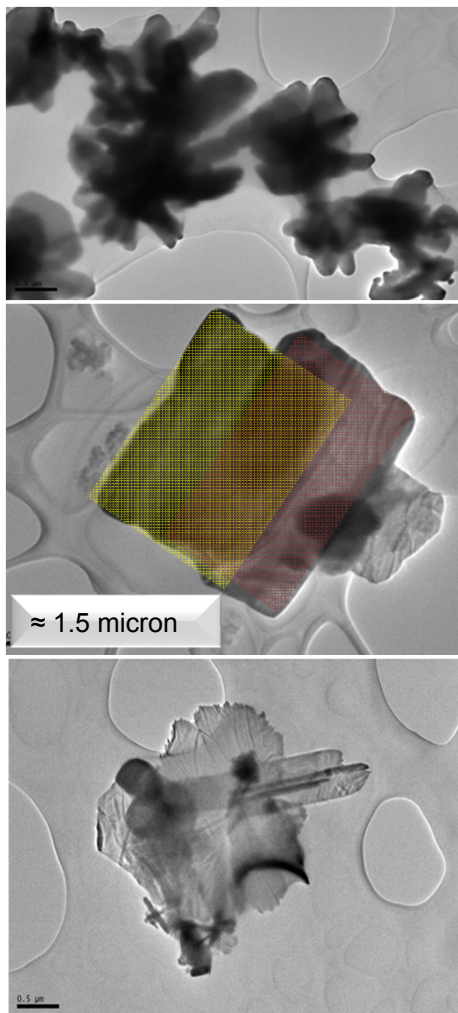


Temporal Study



XRD and TEM

Different morphologies observed
for the Same Rxn Condition, SnSe



What I Learned

- Bulk tin chalcogenide nanomaterial have a band gap of 0.2-2.3 eV and the products can be used as thin film coating for the panel for antireflection.
- Tin Telluride temporal study revealed that product formed right when the chalcogenide was injected.
- Different morphologies can be seen for the same product.

