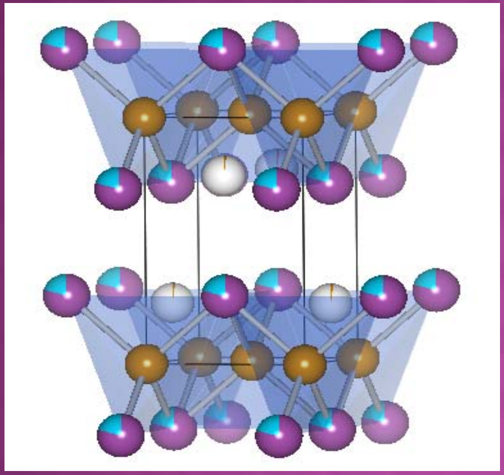
Neutron Diffraction Studies on the Structure and Chemical Composition of Superconducting Phases of $Fe_{1+y}Te_{1-x}Se_x$



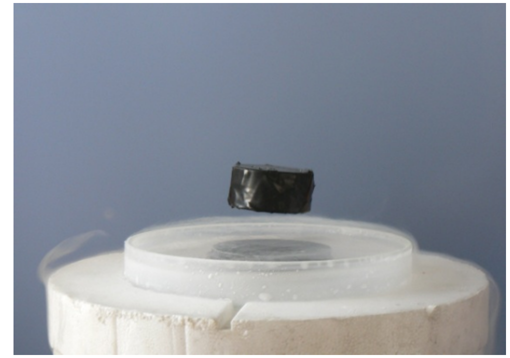
Vikas Bhatia*, Efrain Rodriguez
NIST Center For Neutron Research

Overview

- Introduction & Motivation
- X-Ray/Neutron Diffraction
- Synthesis Methods
- Rietveld (Structural) Analysis
- Future Work

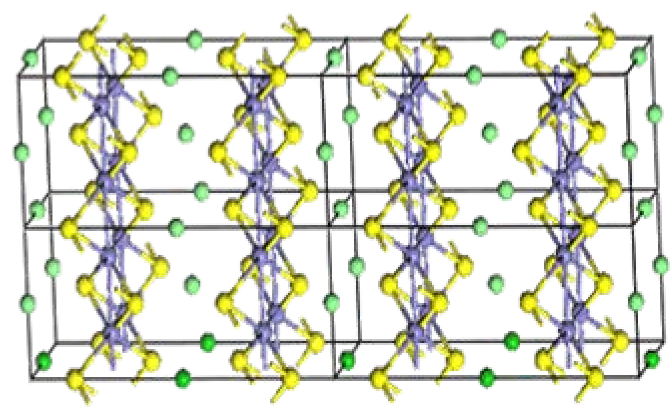
Introduction

- Superconductivity- Electrical Resistivity of Zero



Meissner Effect
Taken from http://faculty.nwacc.edu

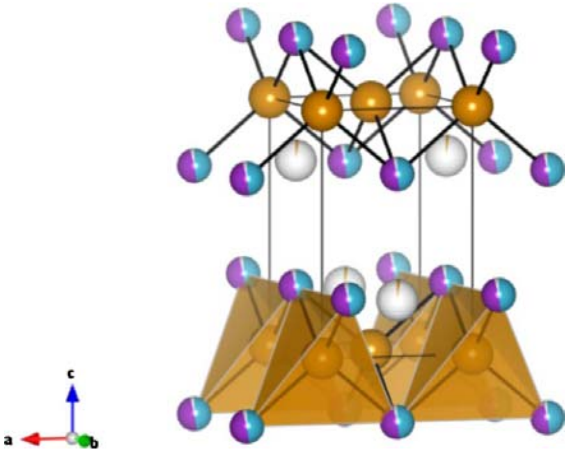
- Iron based compounds become superconducting at certain chemical compositions
  - Iron Pnticides



Iron Pnticide -**$CaFe_2As_2$**
Taner Yildirum

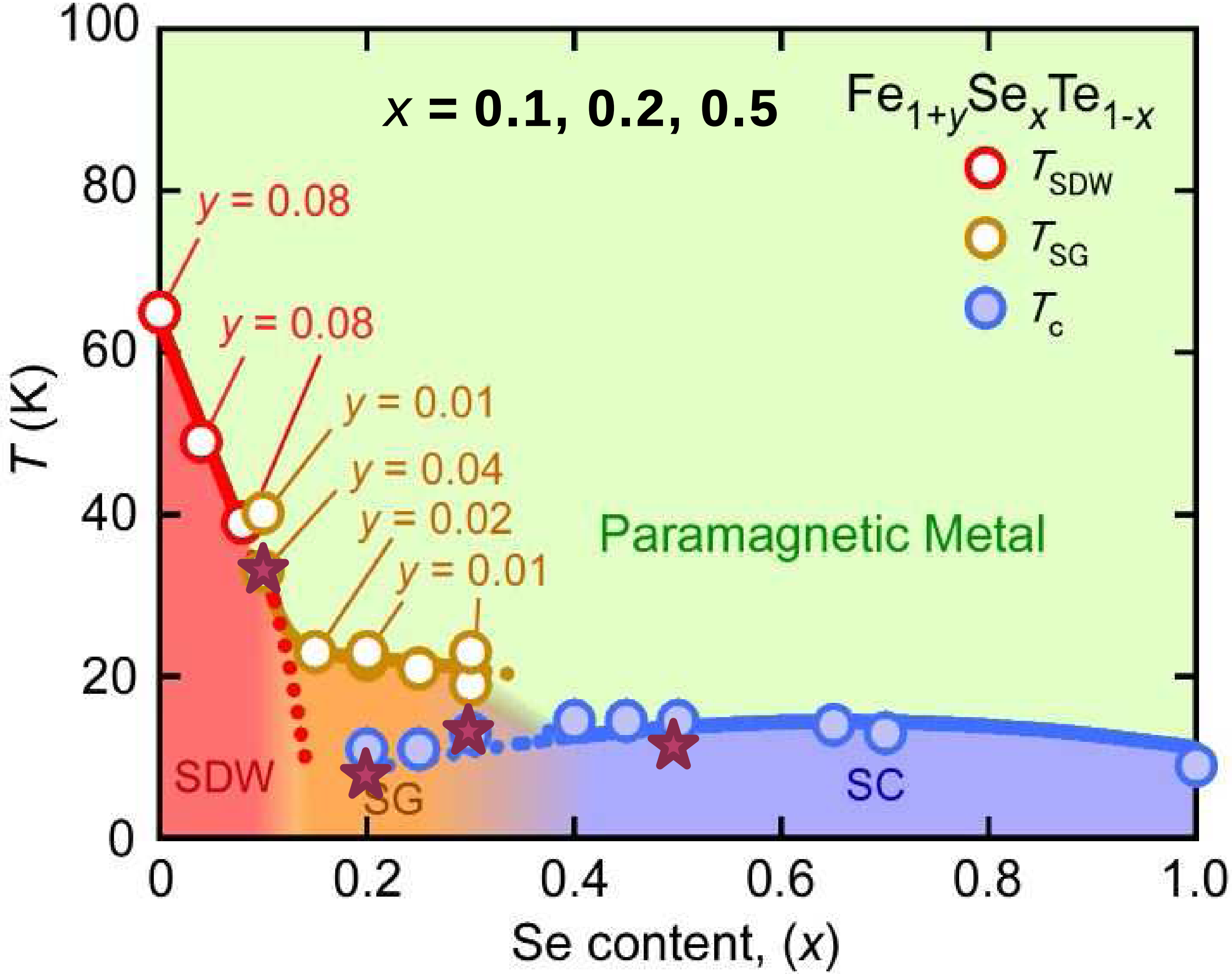
Motivation

- Iron Chalcogens have been found to superconduct
- FeTe becomes superconducting with the doping of Se at certain stoichiometries
  - $Fe_{1+y}Te$ – monoclinic or orthorhombic
  - $Fe_{1+y}Te_{1-x}Se_x$- tetragonal
- Fe(Te,Se) system is nonconventional
  - Offer the opportunity to study chemical composition, magnetism, and superconductivity



D. Louca, *et al.*, *Phys Rev B*. **2010,** 81

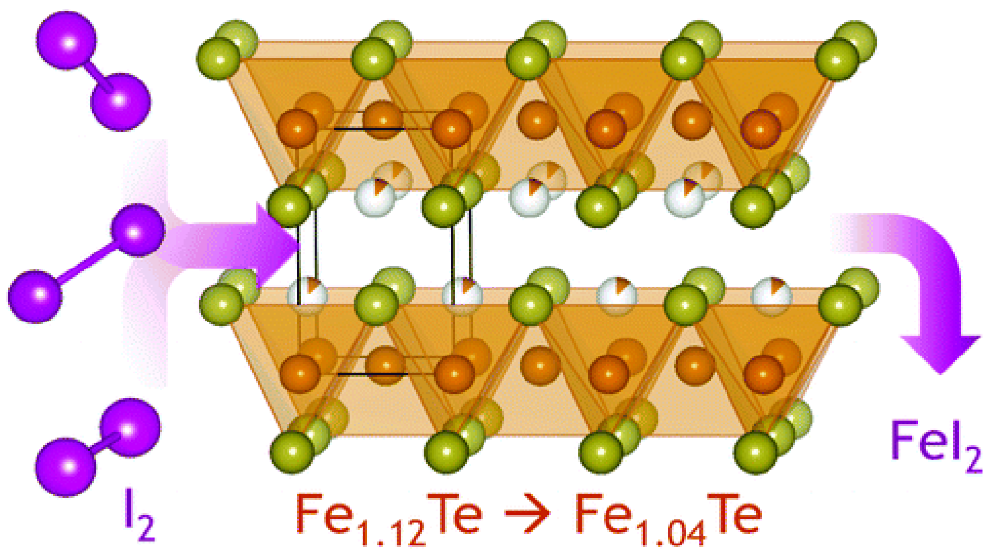
$Fe_{1+y}(Te_{1-x}Se_x)$ phase diagram- What are we studying?



N. Katayuma, *et al*., *Arxiv* 1003.4525v1

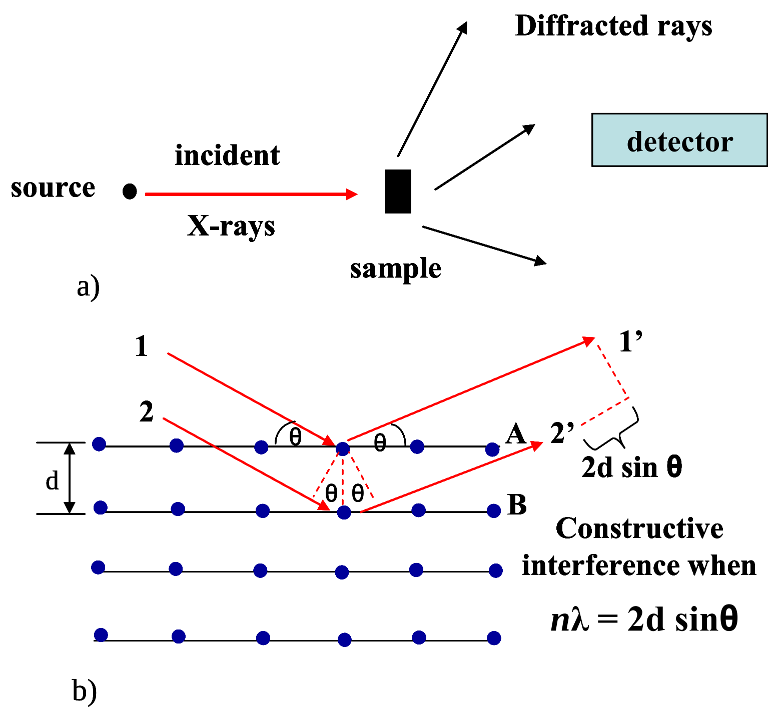
Soft Chemistry Route- Iodine used to remove excess Iron

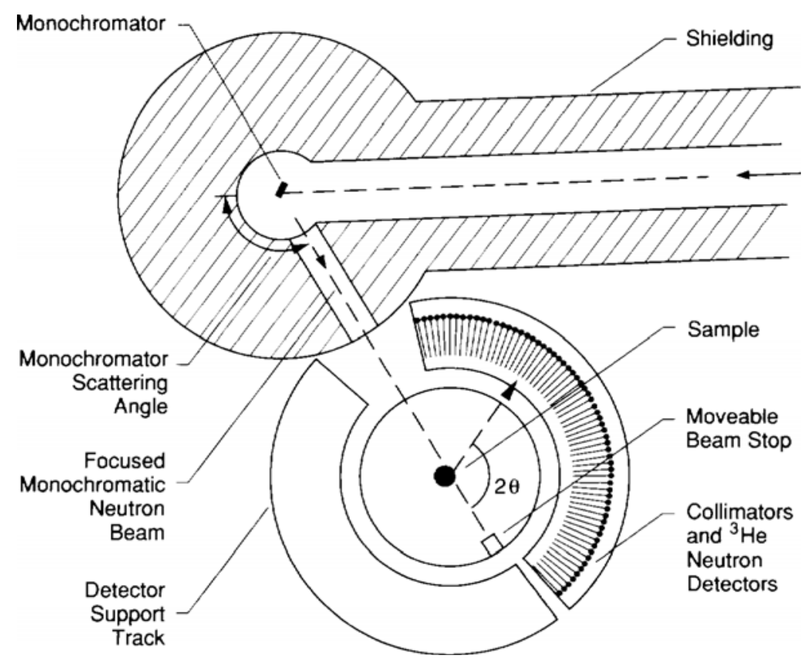
Iodine deintercalation to help study the effects of excess iron



E. E. Rodriguez, *et al*., *J. Am Chem. Soc*. **2010,** 132

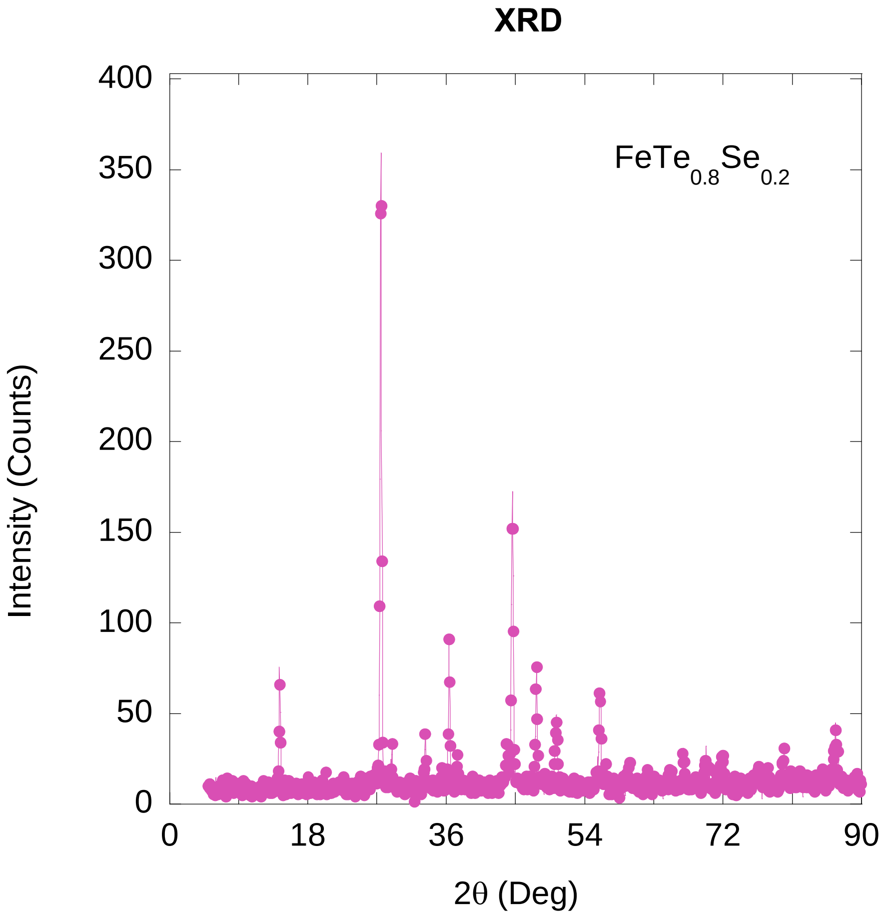
X-Ray vs. Neutrons

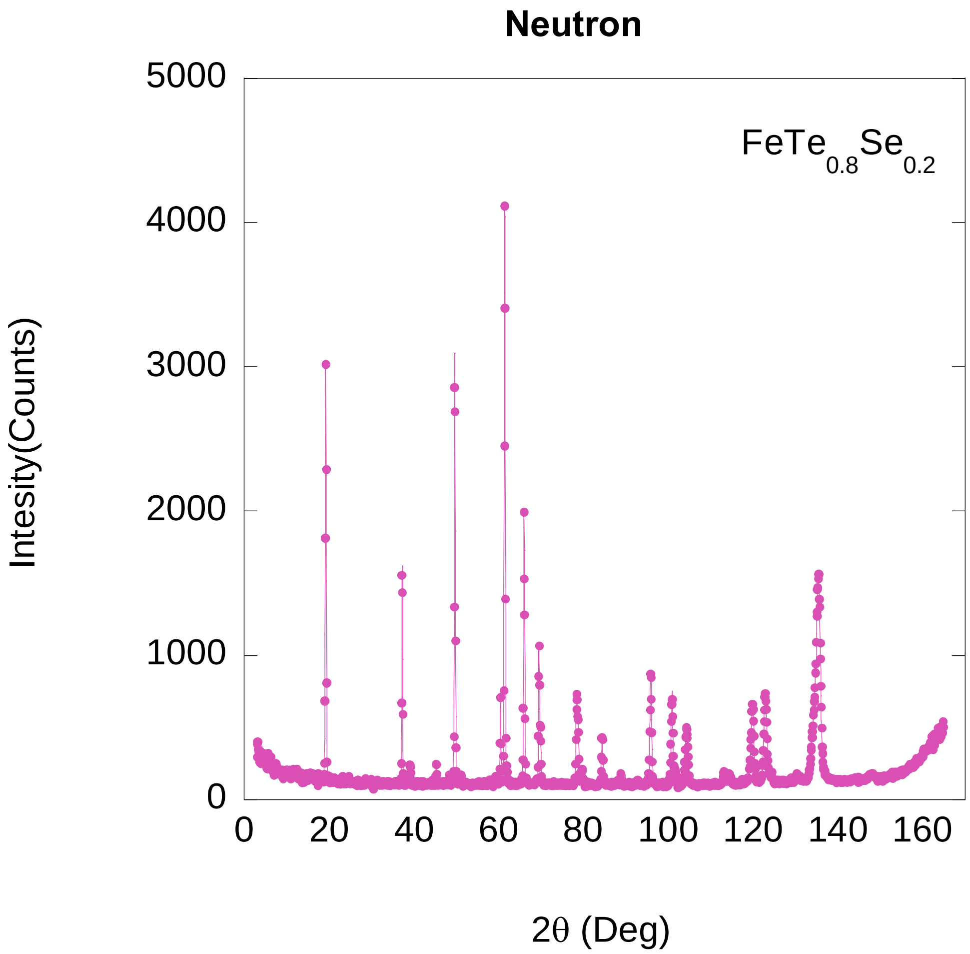




Roger Pynn, "Neutron Scattering: A Primer"

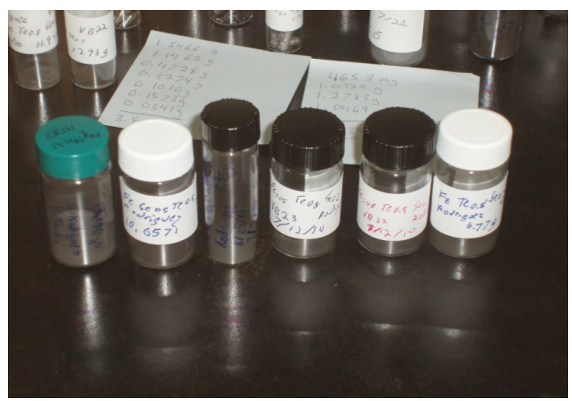
Adapted from: West, A. R., *Basic Solid State Chemistry*. 2nd ed.; John Wiley & Sons: New York, 1999, pp. 134-135.


XRD
FeTe0.8Se0.2
Intensity (Counts)
2θ (Deg)
0
50
100
150
200
250
300
350
400
0
18
36
54
72
90


Neutron
FeTe0.8Se0.2
Intesity(Counts)
2θ (Deg)
0
1000
2000
3000
4000
5000
0
20
40
60
80
100
120
140
160

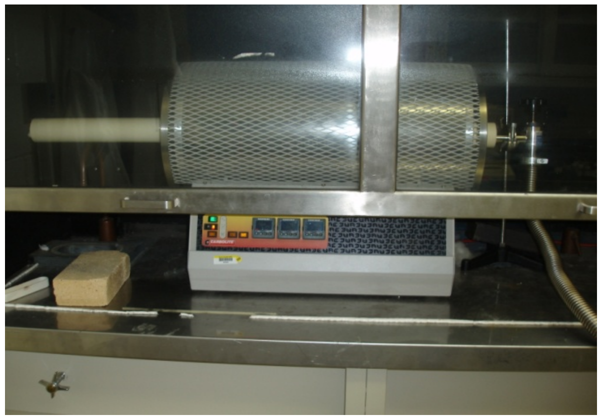
Synthesis — solid state method

- Calculate the amounts of reactants
  - $(1+y)Fe + (x)Se + (1-x)Te \rightarrow Fe_{1+y}Te_{1-x}Se_x$
- Solid State Reaction
- Samples are reacted with pure Iodine vapor to remove excess Iron
  - $Fe_{1+y}Te_{1-x}Se_x + (y)I_2 \rightarrow FeTe_{1-x}Se_x + FeI_2$
- Sample washed with methanol to remove FeI_2



Synthesis — new methods

[1]



Horizontal Bridgman Technique

"Vertical Bridgman Technique"

- New procedure for Iodine reactions was devised
  - Deintercalation of Iron with Iodine/Acetonitrile
- Single crystal growth
  - Horizontal and "Vertical" Bridgeman Technique

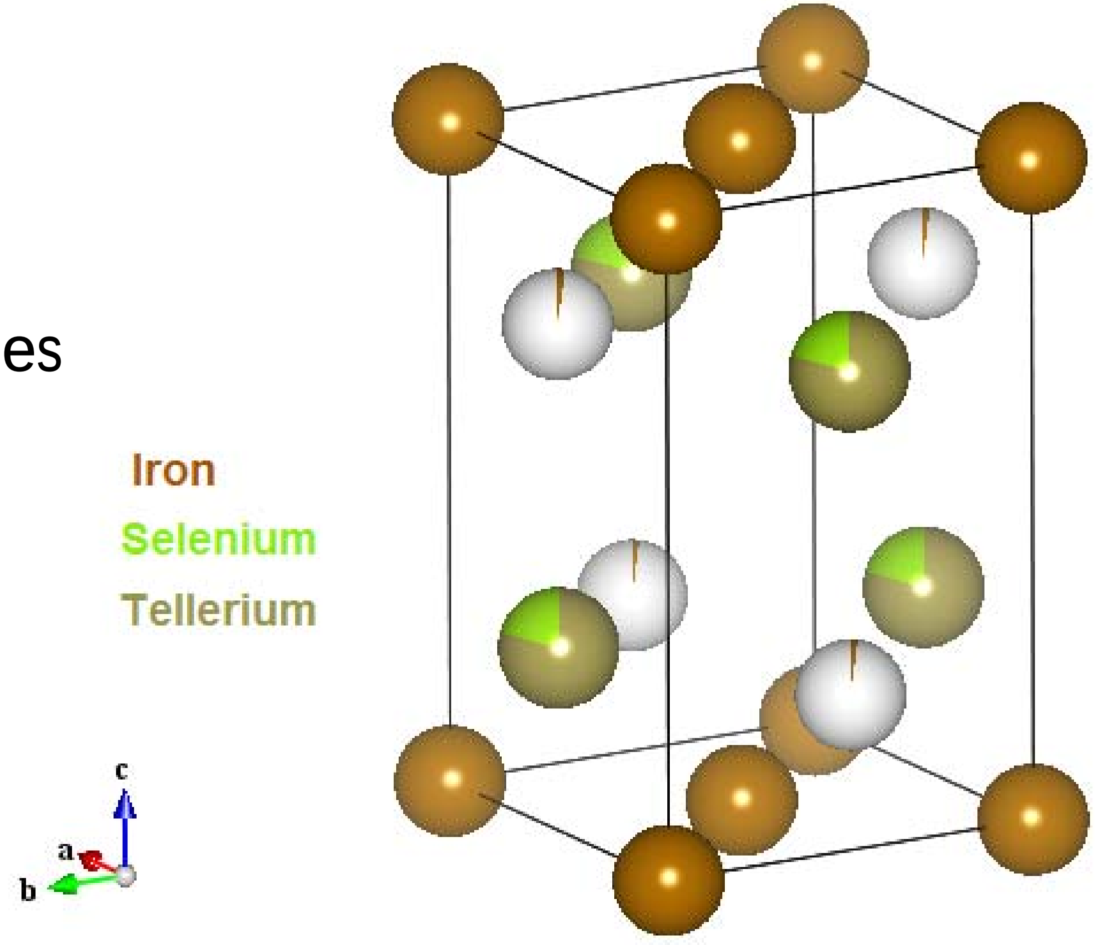
[1]. *Taken from stirplates.com*

After Samples Are Prepared…

- X-Ray Diffraction
- BT-1
- Rietveld Refinement
- Sent for magnetic measurements

Rietveld Refinement

- Lattice Parameters
- Occupancies
- Positions
- Bond Distances/ Angles

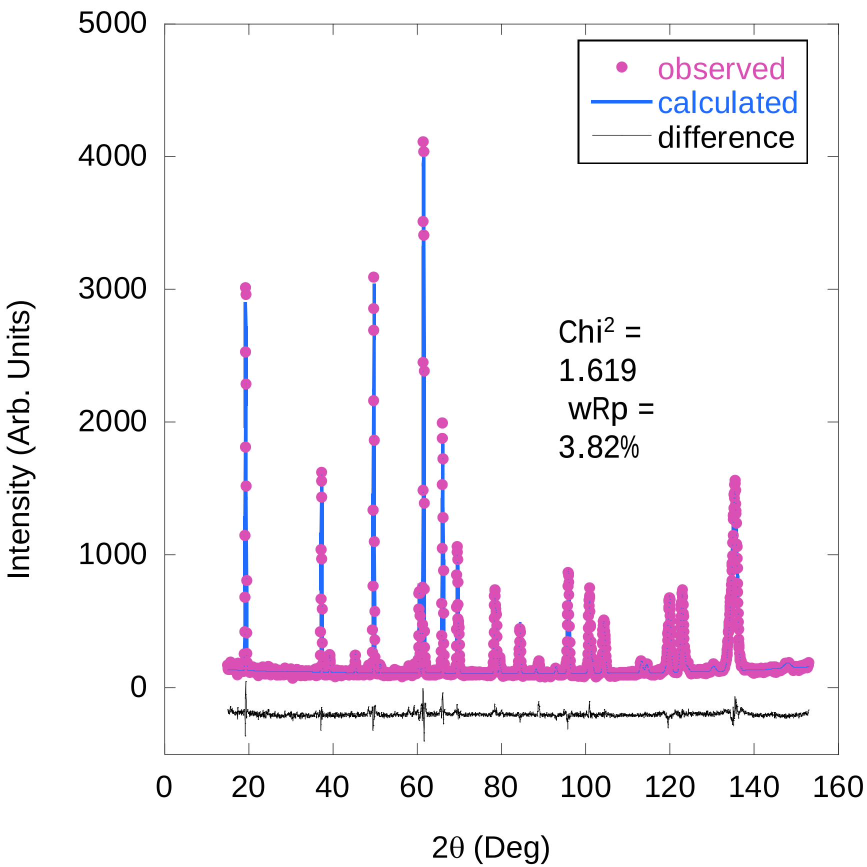


$FeTe_{0.8}Se_{0.2}$ Neutron Data RT Before and After I_2 Reaction

Before

Interstitial Iron = **3.9(3) %**
Tellurium = 80(2) %
Selenium = 20(2) %

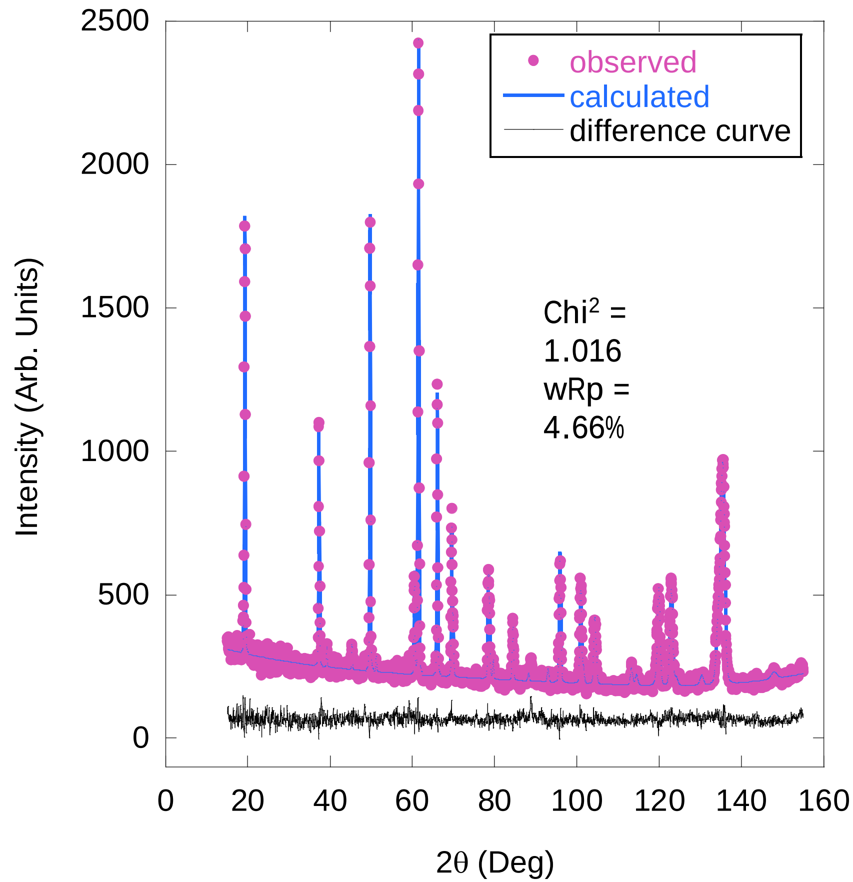
a = 3.81024(4) Å
b = 3.81024(4) Å
c = 6.1821(1) Å



After

Interstitial Iron = **2.3(3) %**
Tellurium = 80(2) %
Selenium = 20(2) %

a = 3.81013(5) Å
b = 3.81013(5) Å
c = 6.18435(1) Å



$FeTe_{0.9}Se_{0.1}$ Neutron Data Before I_2 Reactions

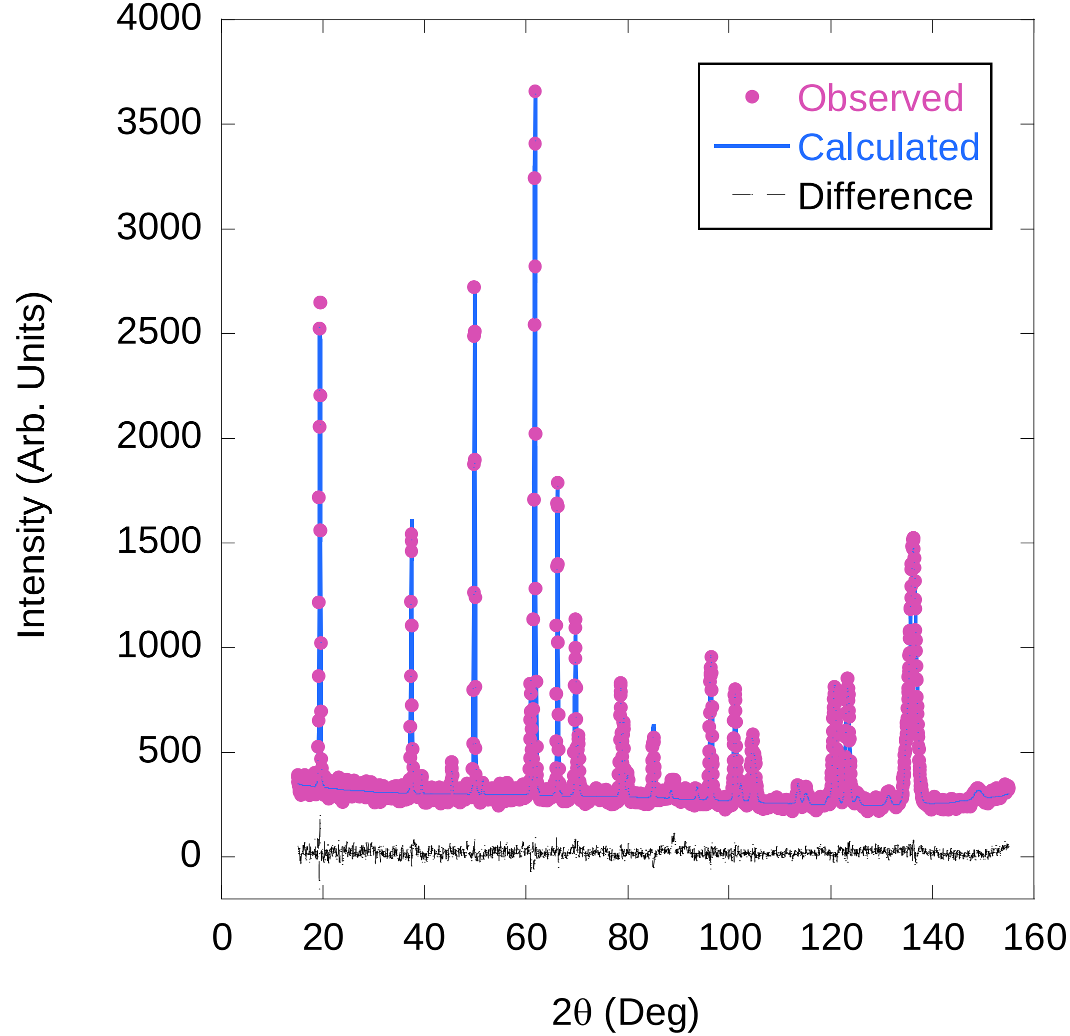
- With Selenium decrease
  - Amount of Interstitial Iron increased
  - Lattice distances increased

Interstitial Iron = 5.9(3) %
Tellurium = 92(2) %
Selenium = 8(2) %
wRp = 7.2%
Chi^2 = 1.284

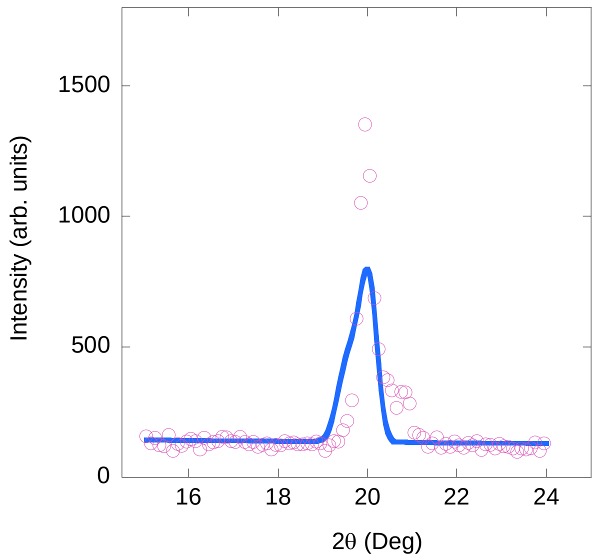
a = 3.8156(1) Å

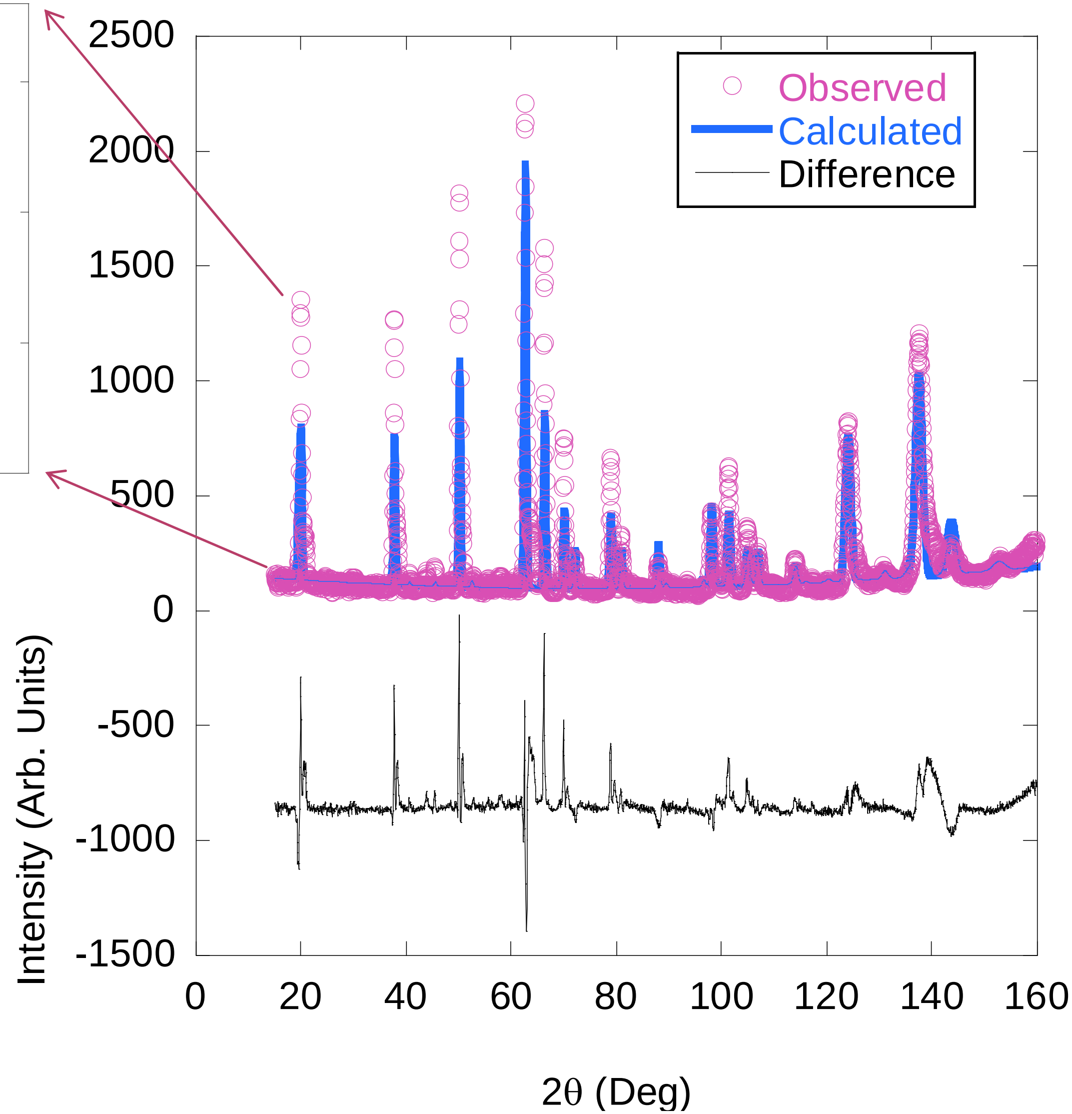
b = 3.8156(1) Å

c = 6.2367(2) Å



$FeTe_{0.5}Se_{0.5}$ Neutron Data 100k Quenched 440° C





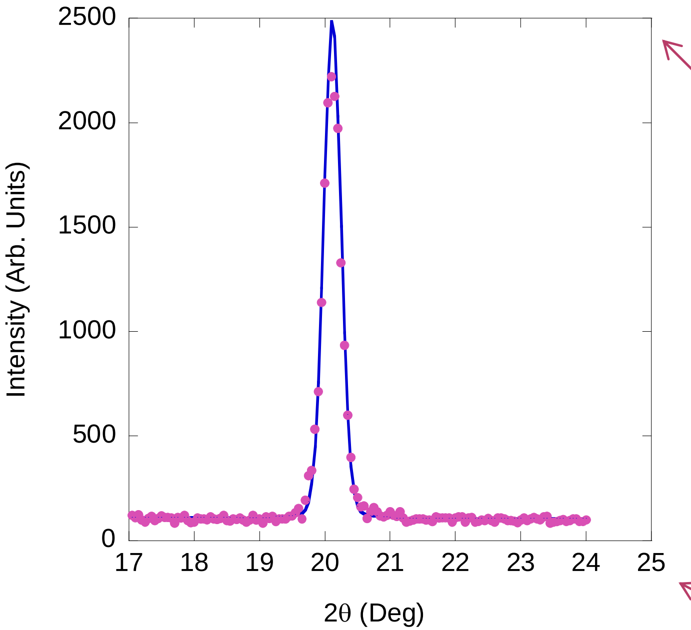
Interstitial Iron = 5(1) %
Tellurium = 34(7) %
Selenium = 66(7) %
Chi^2 = 11.5
wRp = 28.64%

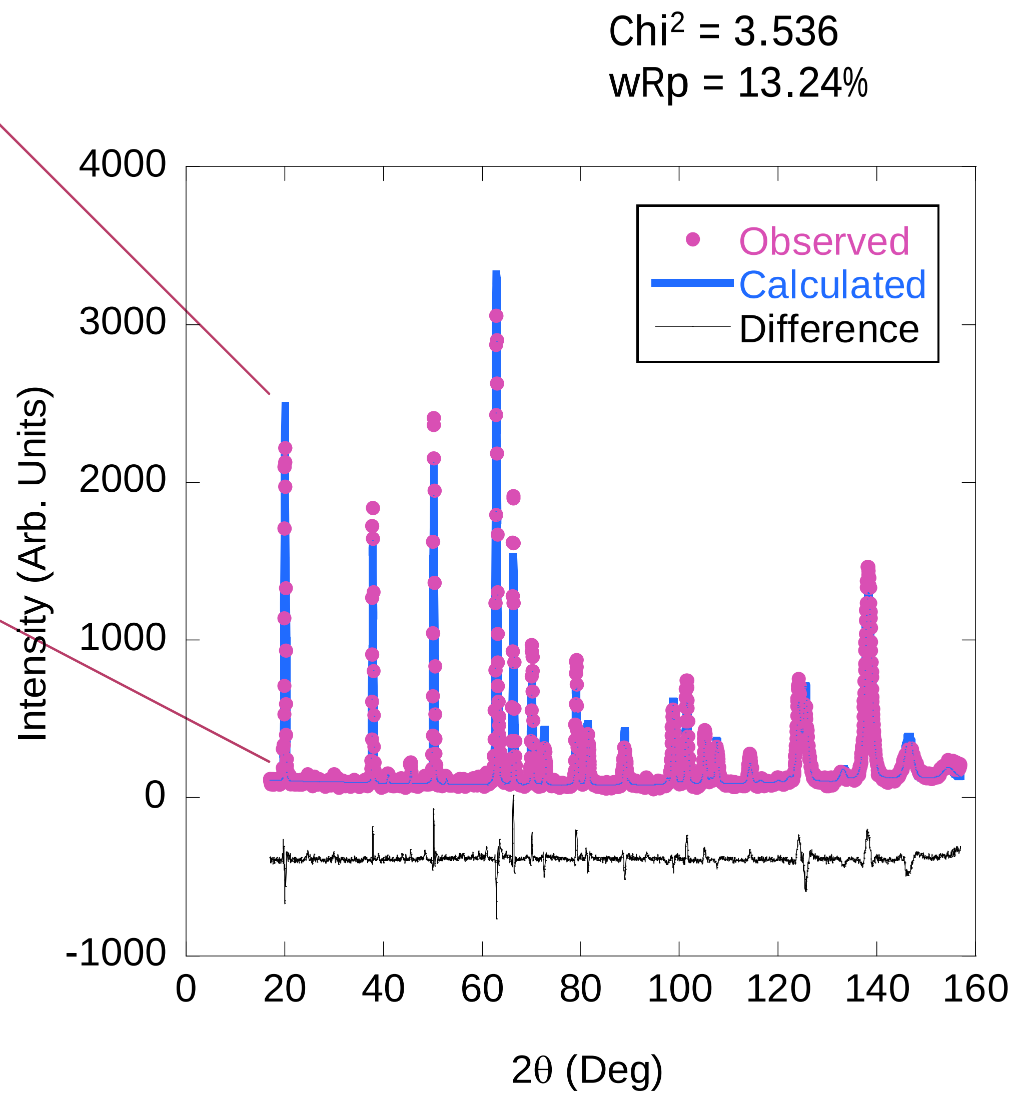
a = 3.8020(1) Å

b = 3.8020(1) Å

c = 5.9902(5) Å

$FeTe_{0.5}Se_{0.5}$ Neutron Data 100k Slow Cooled





Interstitial Iron = 2(1) %
Tellurium = 52(8) %
Selenium = 48(8) %
Chi^2 = 3.51
wRp = 13.11%

a = 3.7941(1) Å
b = 3.7941(1) Å
c = 5.9328(2) Å

Single Crystals

1.5466 g and 1.2783 g single crystals of $FeTe_{0.7}Se_{0.3}$

Deintercalation
Single Crystal diffractions and inelastic neutron scattering

Conclusions

- I_2 reactions successfully removed interstitial Fe
- Higher amounts of Se lowered amount of interstitial iron
- Slow cooled is the better method for synthesis of 50:50
- Will allow us to explore new parts of the phase diagram

FUTURE WORK

From the film "Avatar"
Avtr.com

- Analyze deintercalation through acetonitrile results
- Neutron Studies $FeTe_{0.9}Se_{0.1}$ after I_2 deintercalation
- Complete phase diagram

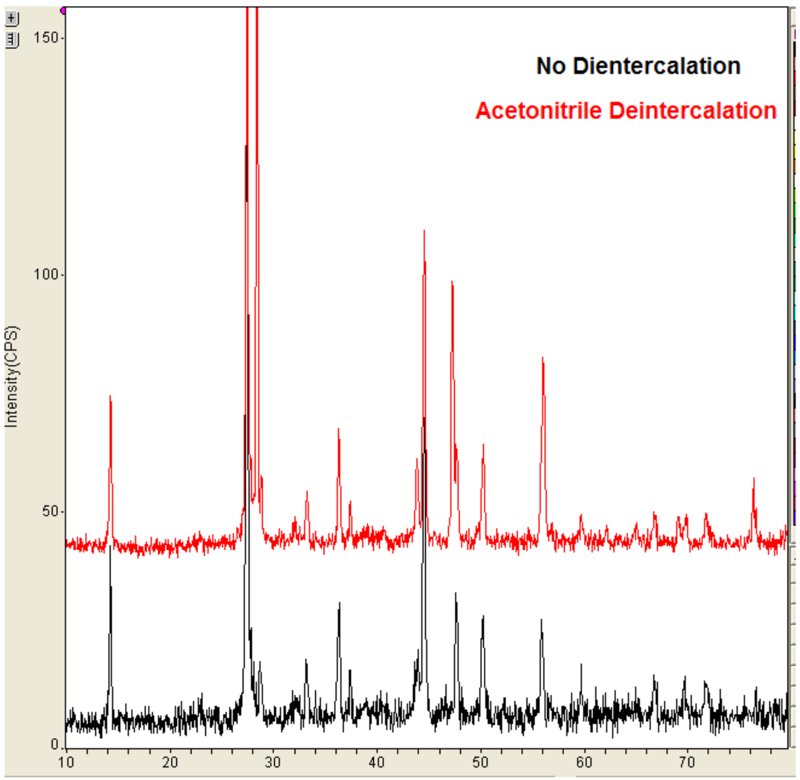
Thanks to:

- Dr. Efrain E. Rodriguez, Advisor
- Jonny Schear, SHIP student
- Dr. Julie Borchers
- NCNR Staff
- Surf Organizers

NIST Center for Neutron Research

Acetonitrile Reactions Still in progress...

FeTe0.9Se0.1 + Si Standard



FeTe0.8Se0.2 + Si Standard