# Flux Growth Development, NbSe3 in BNNT

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#### Flux Growth

-Oversaturation of source material in a solvent that slowly precipitates

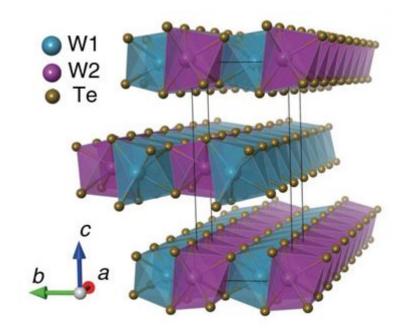
-Earth's core is composed of huge crystals by flux growth<sup>†</sup>!



<sup>†</sup>Sanders, Robert. "11.13.96 - Earth's Inner Core Not a Monolithic Iron Crystal, Say UC Berkeley Seismologists." 11.13.96 - Earth's Inner Core Not a Monolithic Iron Crystal, Say UC Berkeley Seismologists. UC Berkeley, 13 Nov. 1996. Web. 07 Dec. 2016.

#### WTe2

- -Suspect holder of Weyl fermions
- -Literature Recipe: 10g of Te and 200mg of WTe<sub>2</sub>
- -Cooled from 825°C-525°C at 2°C/hr
- -M. Ali: "The cooling rate was found to be significant influence on the crystal quality"

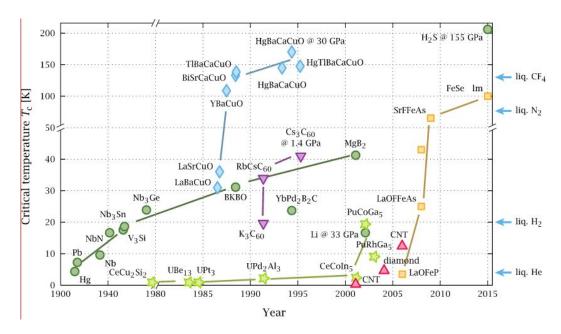


<sup>&</sup>lt;sup>†</sup>Mazhar, Ali N. et al "Correlation of Crystal Quality and Extreme Magnetoresistance of WTe2." *Europhysics Letters* 110.6 (2015)

#### FeSe

-Bulk FeSe  $T_c$  is 8K

-FeSe monolayer on substrate yields high  $T_{\rm c}$ 



-"Iron-based superconductors are extremely sensitive to impurity phases and defects in the crystal structure, which could significantly change the physical and chemical properties both in normal and superconducting states"<sup>†</sup>

#### FeSe

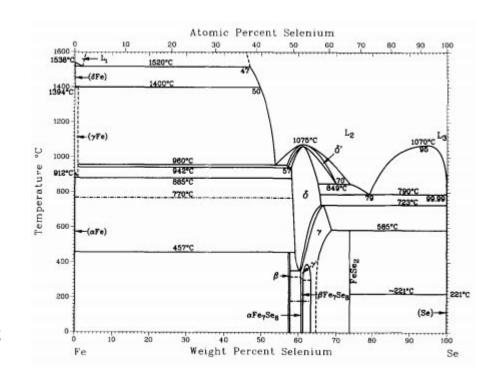
Literature Recipe:

-LiCl, CsCl eutectic

-Ampoule was heated to

715°C for homogenization

-Slow cooling from 457°C to 300°C



Hu, Rongwei. "Synthesis, Crystal Structure, and Magnetism of β-Fe1.00(2)Se1.00(3) Single Crystals." *Physical Review B.* (2011)

#### Flux Growth - Solvent

-Eutectic- A salt mixture that melts at low temperatures

-For FeSe: 50% mole KCl and 50% mole AlCl3 as solvent and

20 mole flux to 1 mole Fe, Se

-For WTe2: Tellurium as a solvent

170:1 mole ratio of Te to WTe2

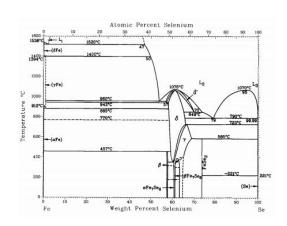
# Why flux growth?

#### WTe,

- "The RRR of these flux-grown crystals in zero field reaches a value of 1250; much higher than the values reported for most vapor-transport-grown crystals" the values reported for most vapor-transport-grown crystals to the values reported for most vapor-transport-grown crystals."

#### FeSe

-"Therefore, the high quality of FeSe1-x single crystals was achieved primarily due to the application of the eutectic melt of salts including fusible aluminum chloride"++



<sup>&</sup>lt;sup>†</sup> Mazhar, Ali N. et al "Correlation of Crystal Quality and Extreme Magnetoresistance of WTe2." *Europhysics Letters* 110.6 (2015) <sup>††</sup> Chareev et al "Single crystal growth and characterization of tetragonal FeSe<sub>1-x</sub> superconductors." Royal Society of Chemistry (2013)

#### **Current Furnace**

-Current furnace cools at 6°C/hr

-Community furnace

-Doesn't record temperature



# Furnace (idea)

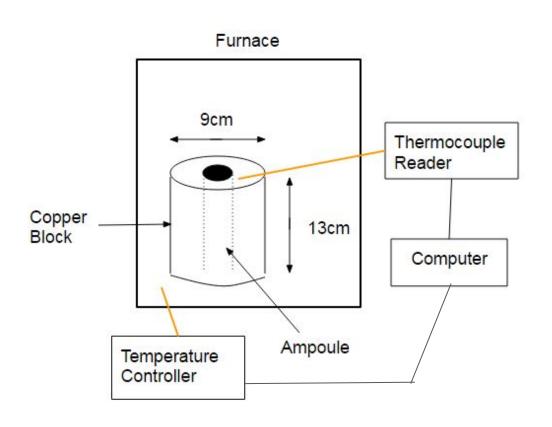
-Want to control temperatures arbitrarily and precisely

- -Record temperature history
- -Huge copper block and quartz to

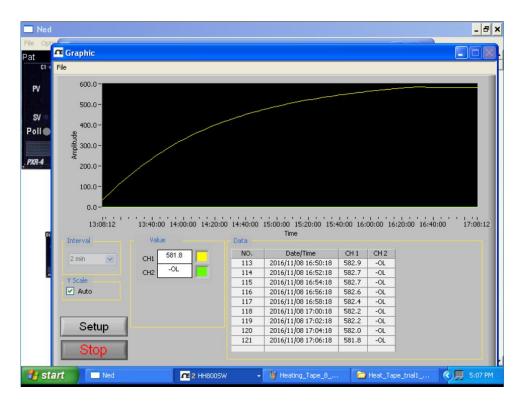
remove thermal fluctuations

-Everything is pre programmed

and happens automatically

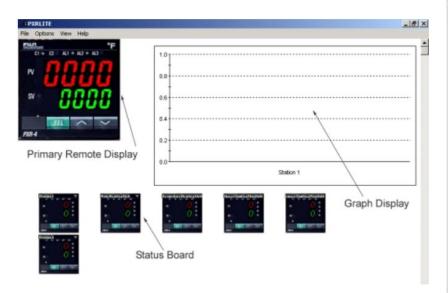


## Thermocouple Reader



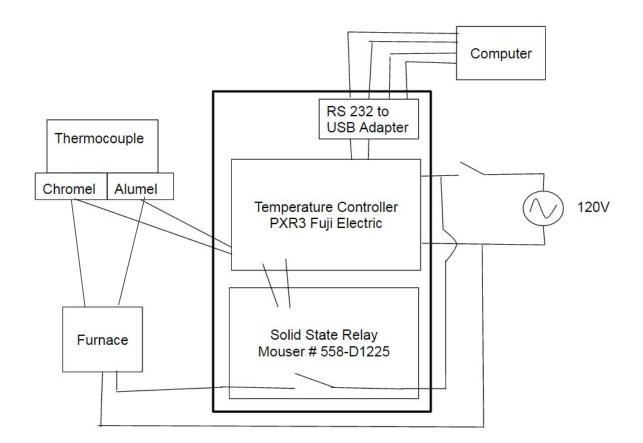


# Computer Interface



Ramp Time to Setpoint	Setpoint Value	Soak Time at Setpoint
1		
2		
3		
4		
5		
6		
, ===		
8 ====		

# Furnace (1st attempt)



# Solid State Relay (SSR) Overheats

-Performance goes down with higher heat

-Temp controller nearby functional until 50°C

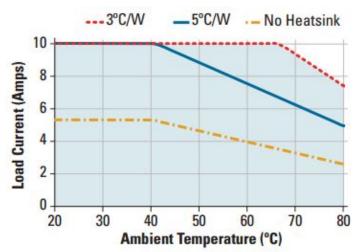
-SSR was heated to

around 62°C

-Separate SSR

from original box

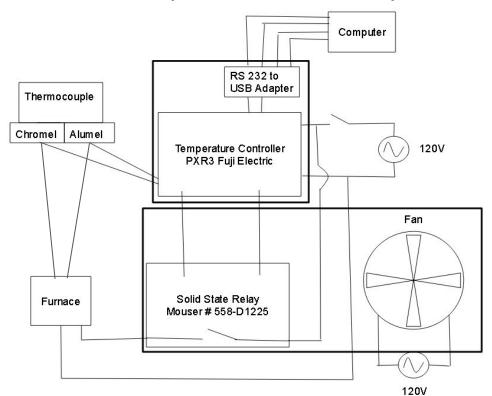
-Add fan and heatsink







# Furnace (development)





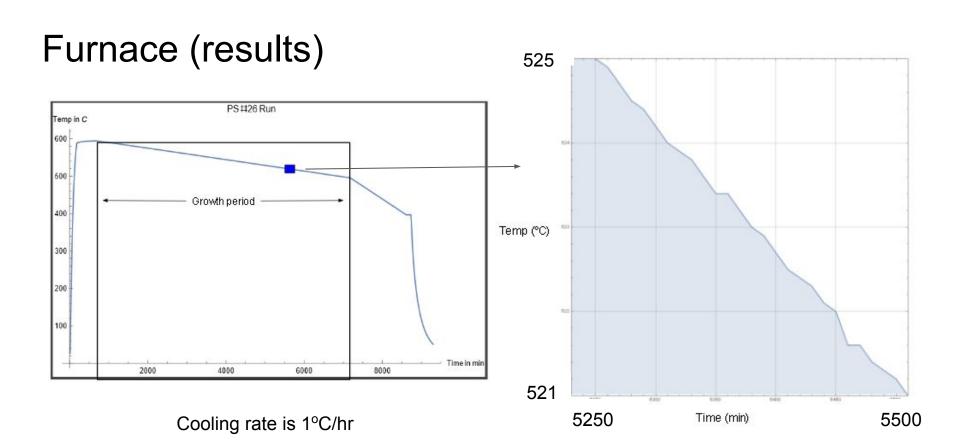
#### Heating element for furnace breaks

-Keep original setup, but wrap Cu block with heating tape

-Heating tape specified for 760°C

-Works as well as before

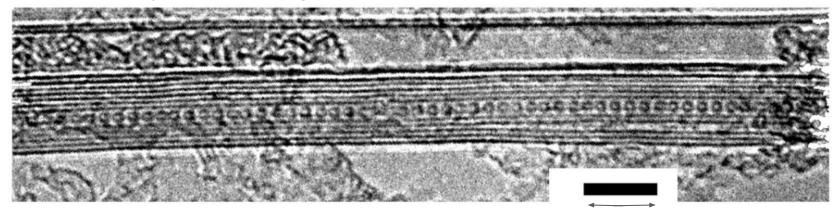




# BNNT (Intro)

Theoretically (1994 A. Rubio et al), experimentally (1995 N. Chopra et al), and packing C60 in BNNT (2003 W. Mickelson et al)

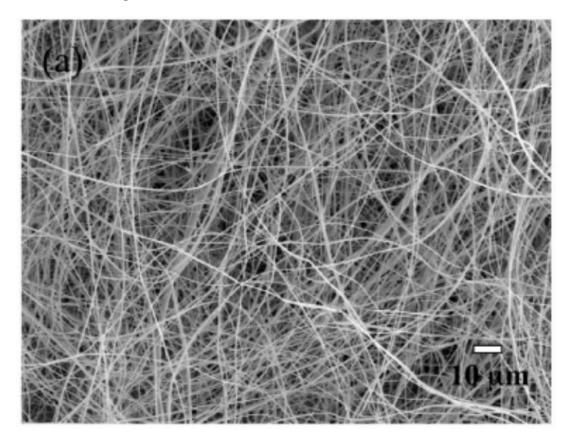
Air sensitive crystals can be grown in BNNT for protection



4nm

Mickelson, W. "Packing C60 in Boron Nitride Nanotubes." *Science* 300.5618 (2003): 467-69. *NCBI*. Web. 7 Dec. 2016.

# NbSe<sub>3</sub>



Hor, Y. S., et al. "Nanowires and Nanoribbons of Charge-Density-Wave Conductor NbSe3." *Nano Letters* 5.2 (2005): 397-401. *Nano Letters*. Web. 7 Dec. 2016.

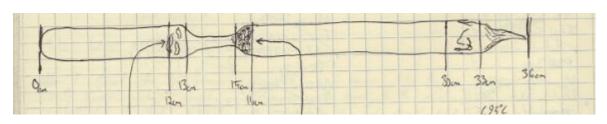
# NbSe<sub>3</sub> in BNNT (Gradient Furnace)

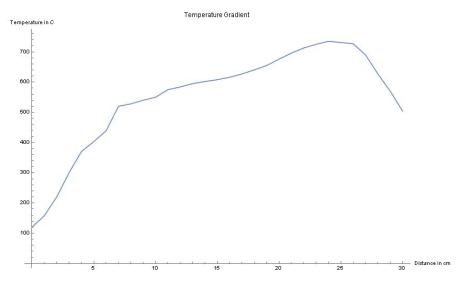
-"Borrowed" Jake's recipe

-Chemical Vapor Transport

-Nb wire (99.9%): 107.5mg

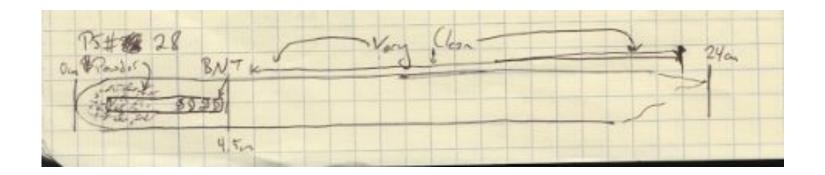
-Se shots: 271.9mg





# NbSe<sub>3</sub> in BNNT (Box Furnace)

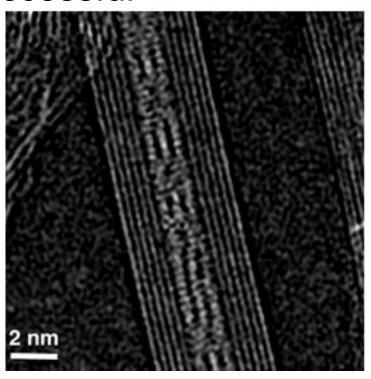
- -Nb powder (99.99%): 48.5mg
- -Se powder (99.999%): 120.6mg
- -Heated in a box furnace at 690°C for 5 days



#### Box Furnace growth is successful

- -From TEM (Thang): NbSe3 has grown inside of BNNT
- -Control runs are ongoing to supplement result





TEM image by Thang

# Thank yous!

Seita, Thang, Jake, Joey, Kevin, and Sissi

Thank you all for listening!