Boron Nitride Nanotubes and Nanotube Mats

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- 19 years old from Old Greenwich, CT
- Mechanical Engineering Major at University of Vermont
  - Computer Science Minor
- Interesting Facts:
  - Member of the Delta Delta Delta sorority
  - Play on the UVM Club Quidditch team
  - Read an average of 50 books a year

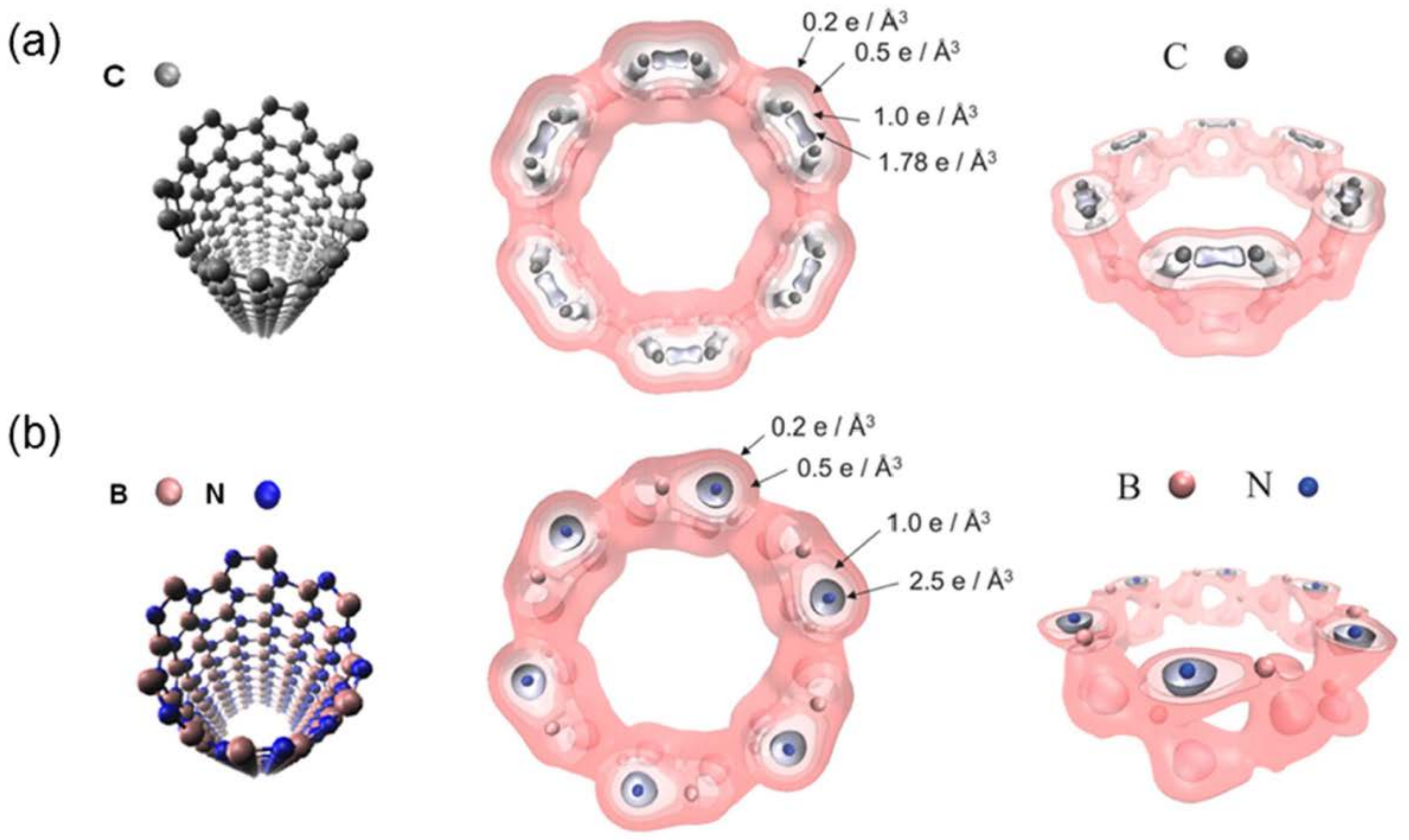




Introduction

Boron Nitride Nanotubes (BNNTs)

- Withstand high temperatures up to 900°C
- Flexible
- Lightweight
- Electrical Insulator
  - CNTs are conductive leading to galvanic corrosion
  - Beneficial for metal matrix composites used as conductors



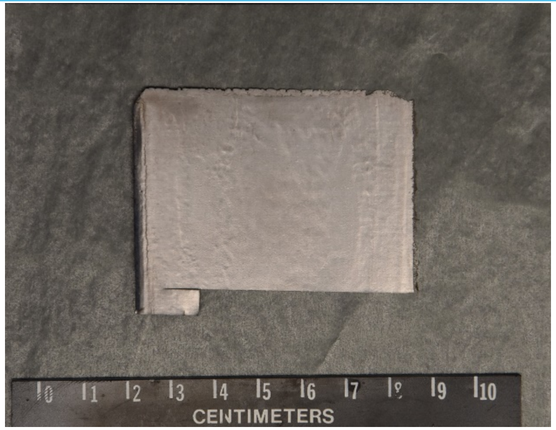
(a) Carbon Nanotube (CNT)
(b) Boron Nitride Nanotube (BNNT)

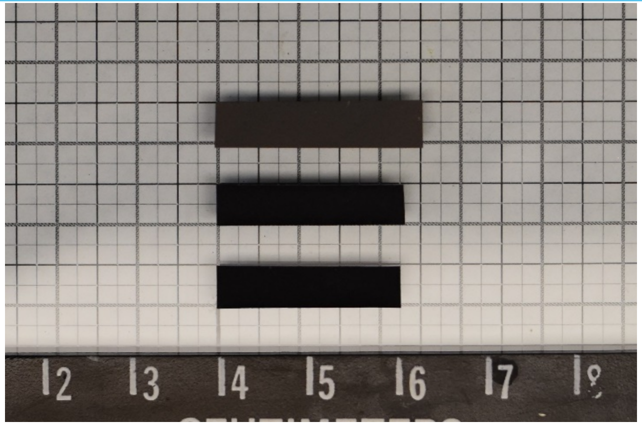
Objective

Boron Nitride Nanotube (BNNT) Nanocomposites

Thin, Dense, Flexible Mats

Metal Matrix Composites (MMC)	Ceramic Matrix Composites (CMC)	Polymer Matrix Composites (PMC)

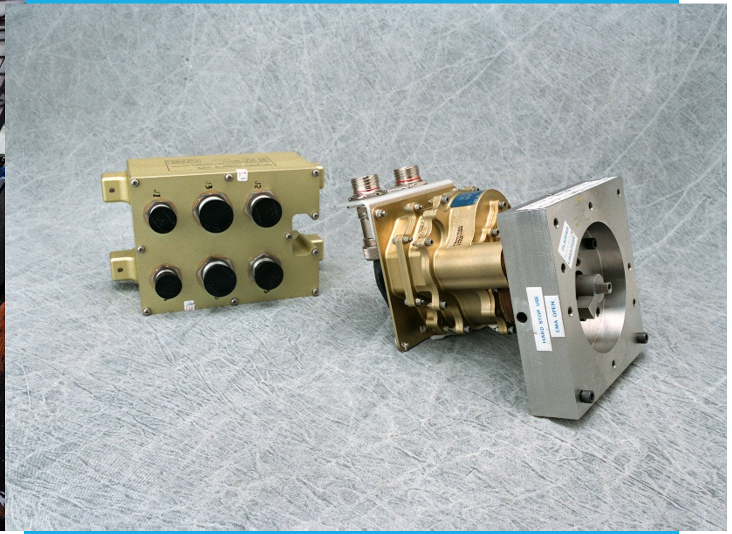




Potential Applications

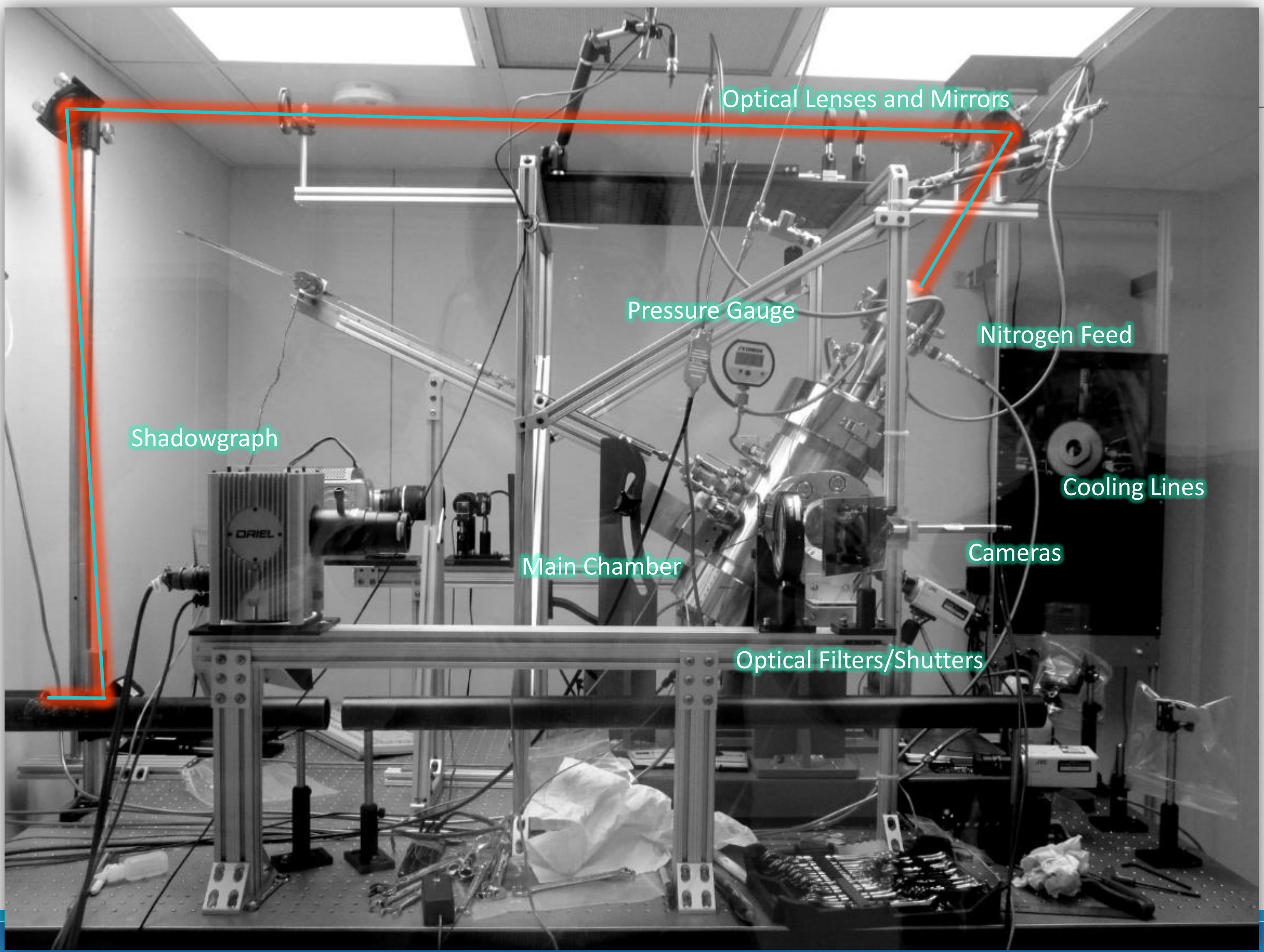
High Temperature Applications

Space Radiation Shielding



Sensors and Actuators

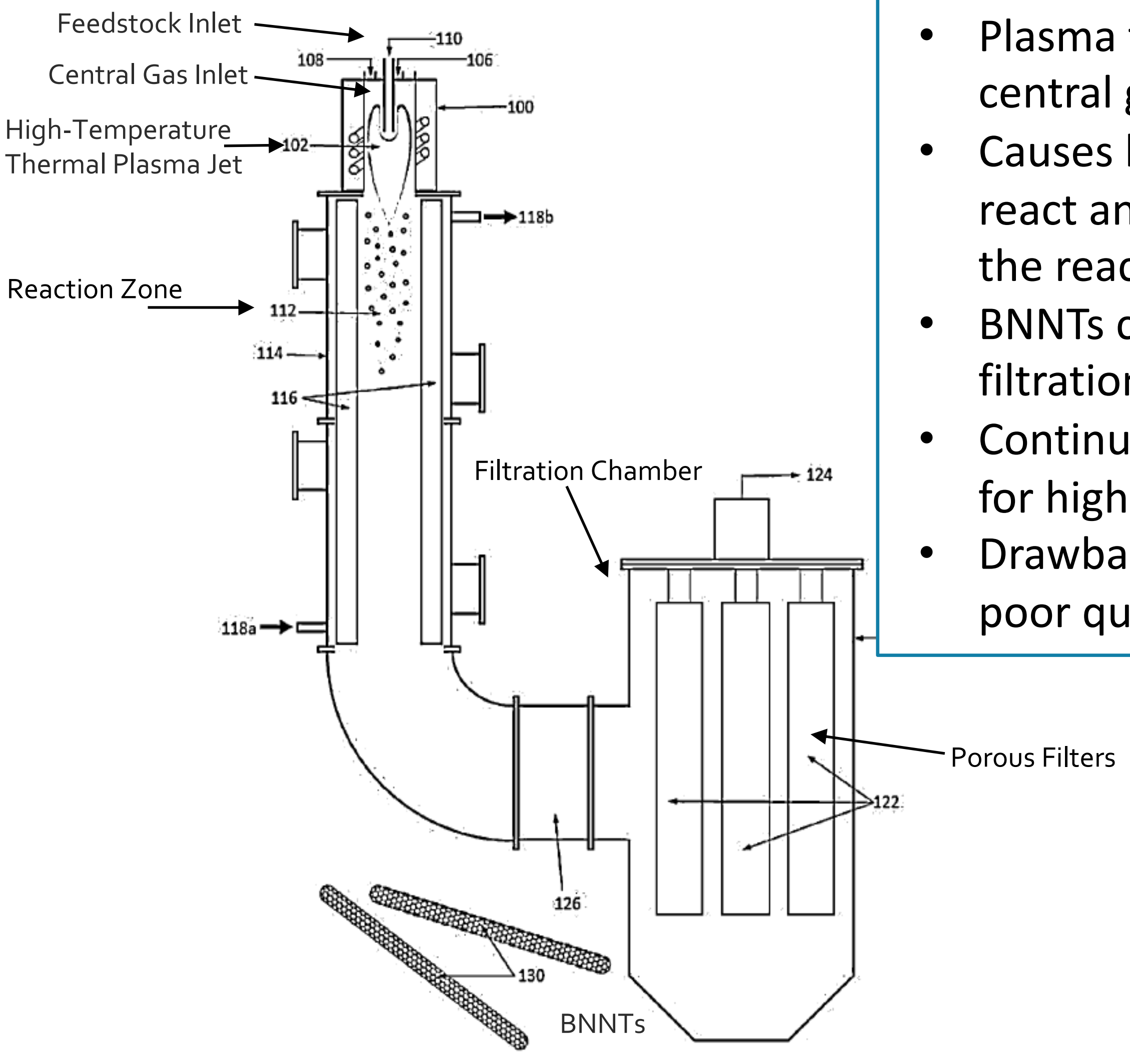
High Temperature-Pressure (HTP) Method



- CO2 laser heats a boron nitride target
- Nitrogen and boron react and condense into BNNT
- Temperature: 4000K
- Pressure: 0.7-1.4 Mpa
- Produces high-quality BNNTS

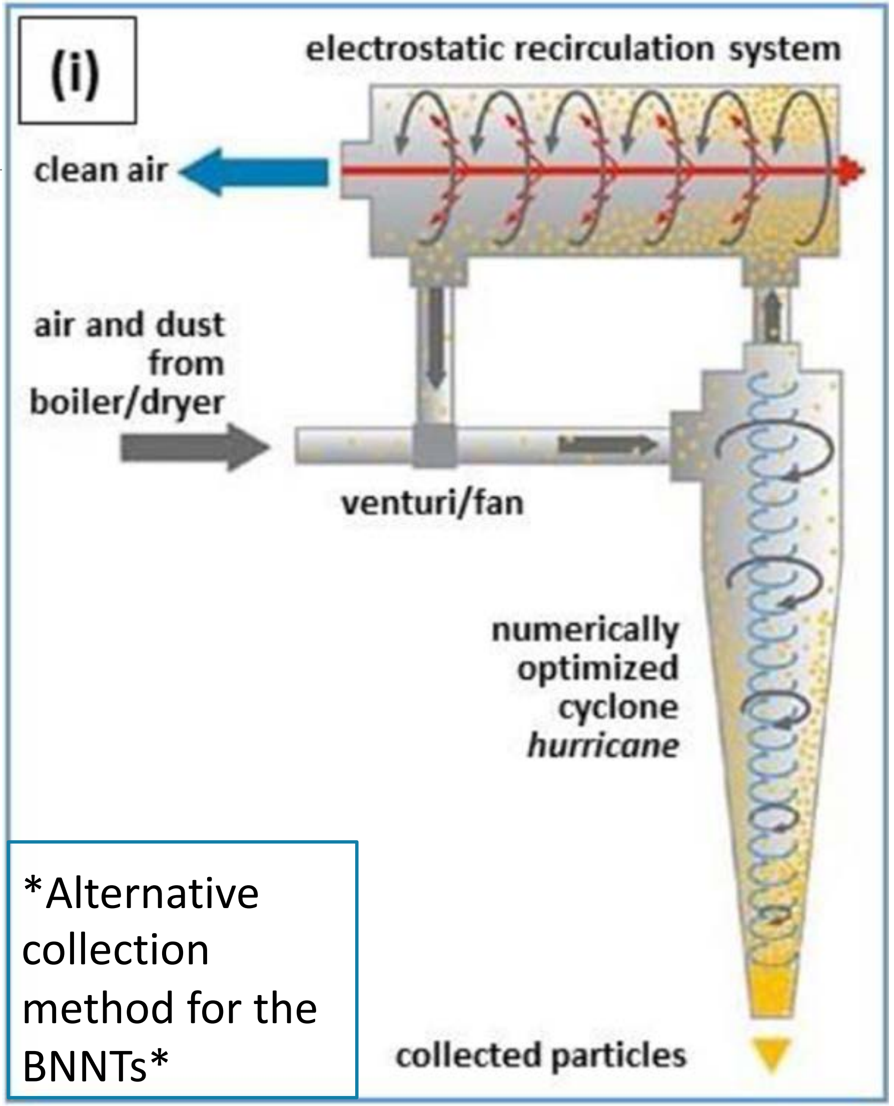
Image credit: National Institute of Aerospace

Plasma Method



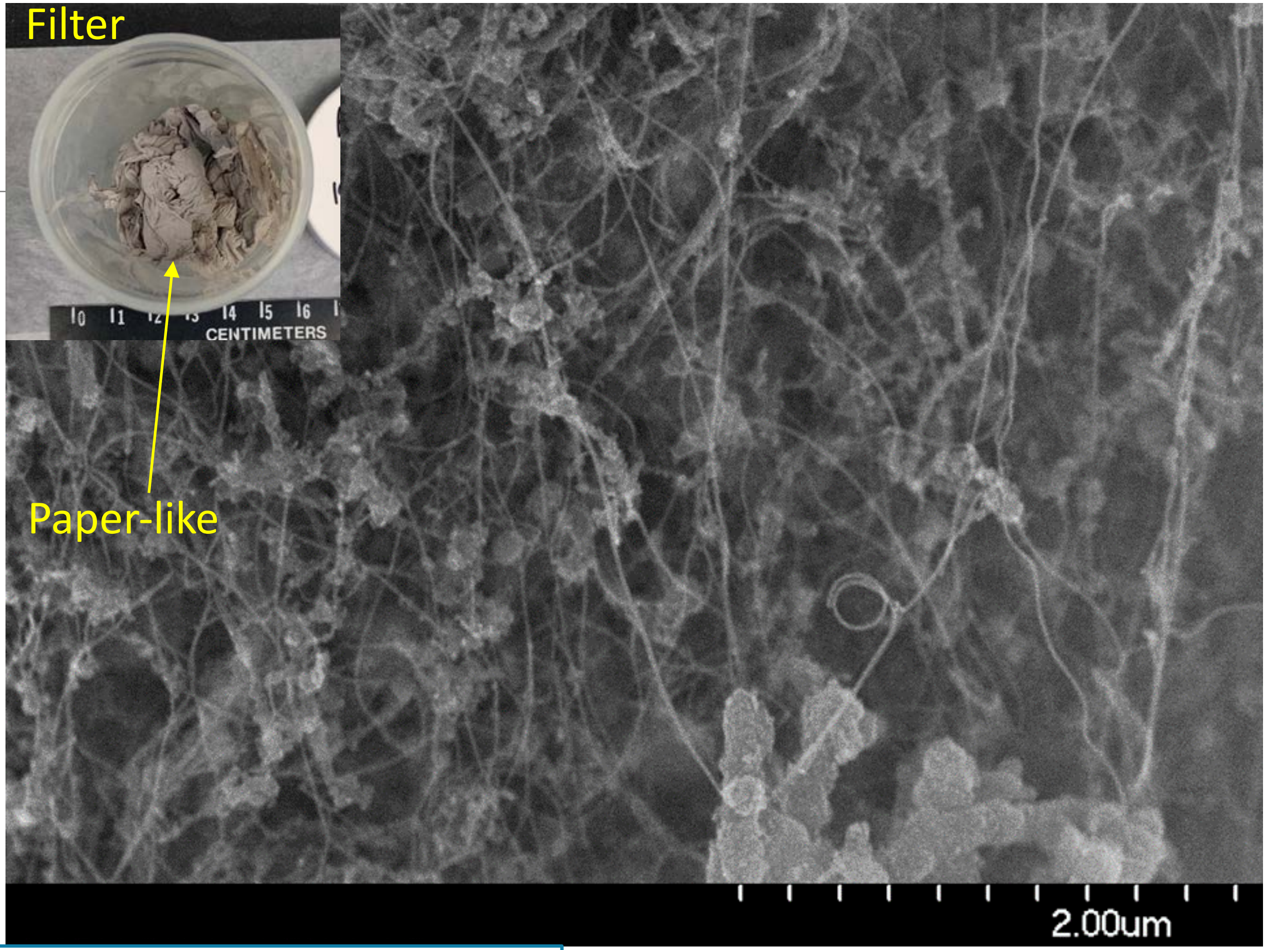
- Plasma torch heats a central gas to 8000K
- Causes h-BN feedstock to react and form BNNTs in the reaction chamber
- BNNTs collected in the filtration chamber
- Continuous process allows for high yield rate
- Drawback is the BNNTs are poor quality

Cyclone Filter:



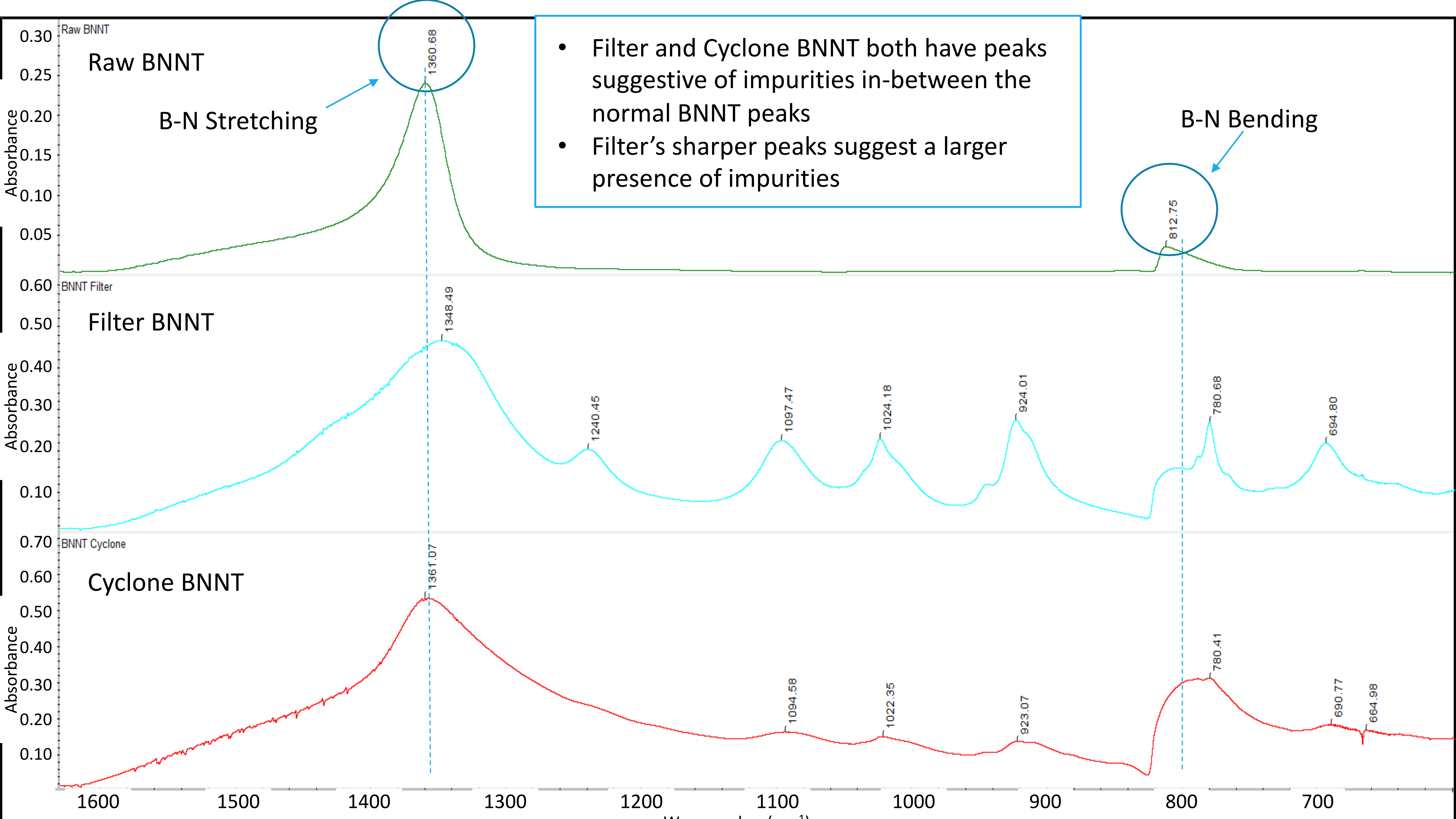
Alternative collection method for the BNNTs

Cyclone and Filter Comparison (x 25k)



Observations:

- No apparent differences in the SEM images for the Cyclone and Filter BNNT samples
- Both have large amounts of impurities visible



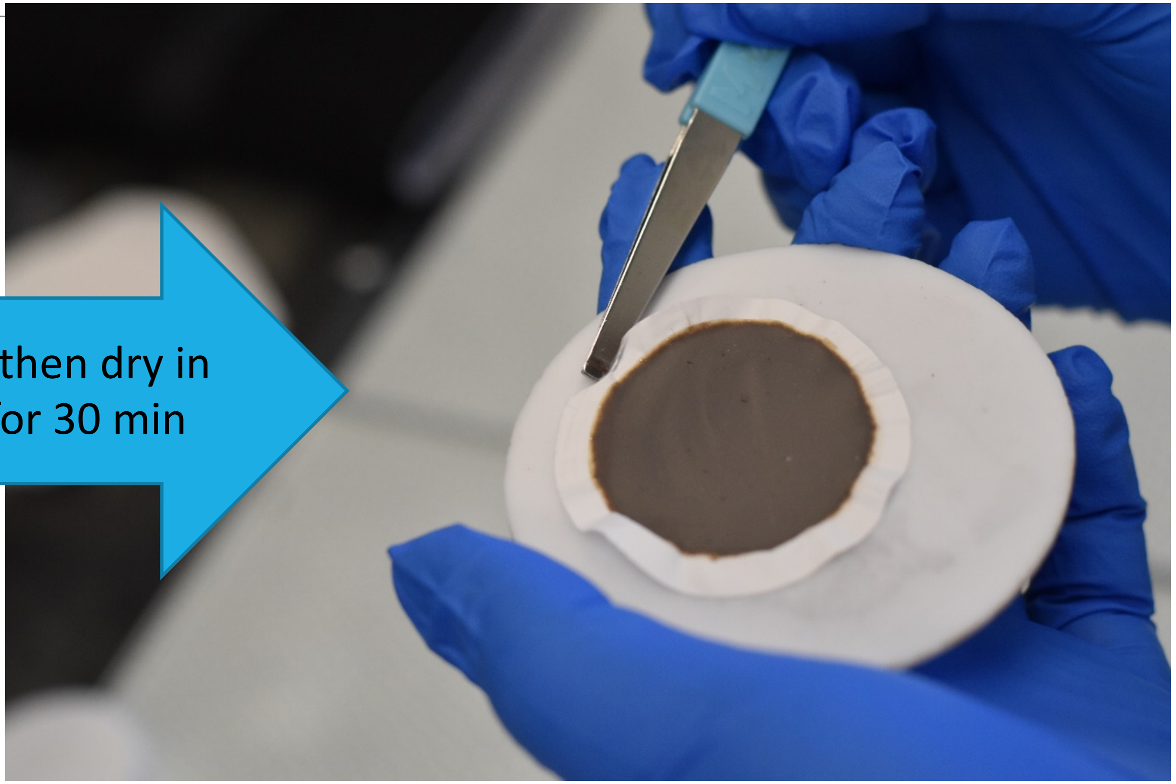
Raw BNNT
Raw BNNT
B-N Stretching
1360.68
Filter and Cyclone BNNT both have peaks suggestive of impurities in-between the normal BNNT peaks
Filter's sharper peaks suggest a larger presence of impurities
B-N Bending
812.75
BNNT Filter
Filter BNNT
1348.49
1240.45
1097.47
1024.18
924.01
780.68
694.80
BNNT Cyclone
Cyclone BNNT
1361.07
1094.58
1022.35
923.07
780.41
690.77
664.98
Absorbance
0.30
0.25
0.20
0.15
0.10
0.05
0.60
0.50
0.40
0.30
0.20
0.10
0.70
1600
1500
1400
1300
1200
1100
1000
900
800
700

Mat Making Set-Up

- A Teflon filter is placed on the glass filter with a funnel clamped on top
- Vacuum is used to filter out the excess water and surfactant in the BNNT solution

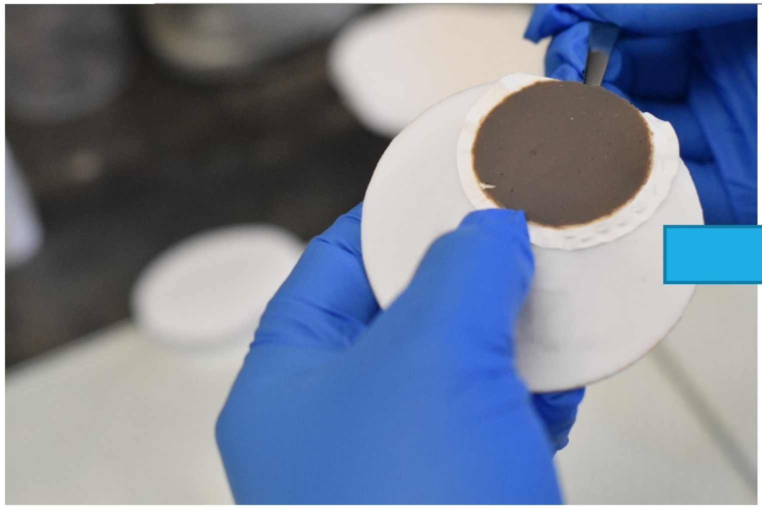
Mat Making Procedure

Filter, then dry in oven for 30 min

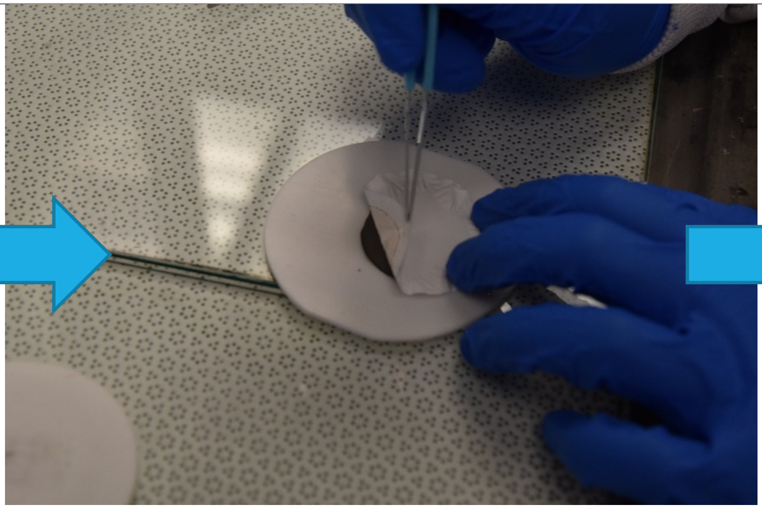


- BNNT solution is poured in the funnel and onto the filter, then the vacuum is turned on for filtering
- Once mat is barely dry, methanol is poured in to wash away excess surfactant
- When filtration is complete, mat and filter are placed in-between two Teflon plates and put in oven

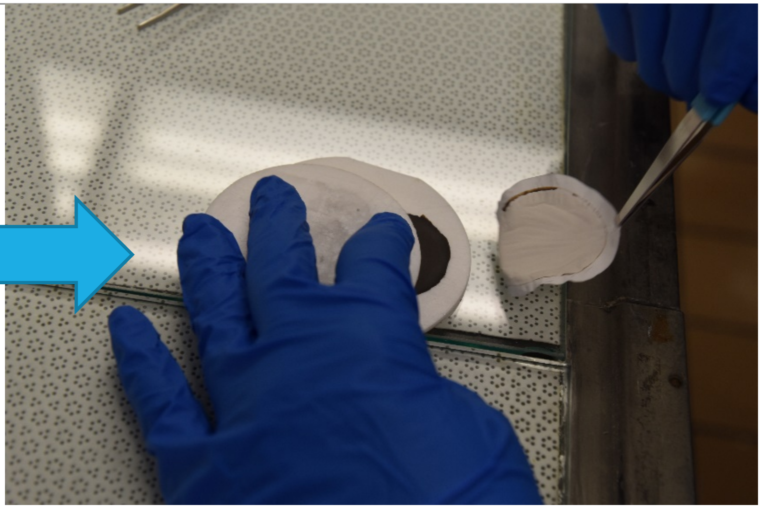
Mat Peeling Procedure



Original Procedure:
- Use the edge of the Teflon plate to free the edges by pulling on the filter with tweezers
- Repeat to gradually free the entire mat



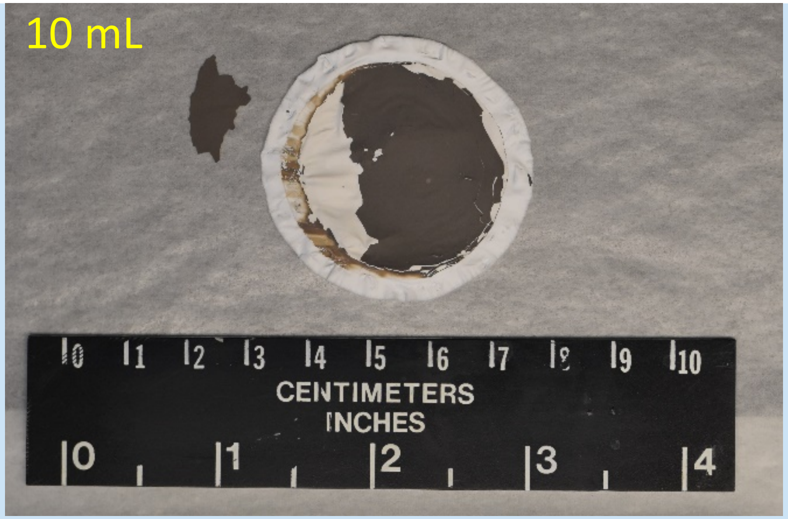
Challenges faced:
- Time consuming (usually around an hour per mat)
- Inconsistent successes
- Damaged mats



Solution:
- Free edges using original method
- Flip mat over and pull back filter to reveal freed edge
- Hold the freed mat in place with another plate and pull the filter off the mat

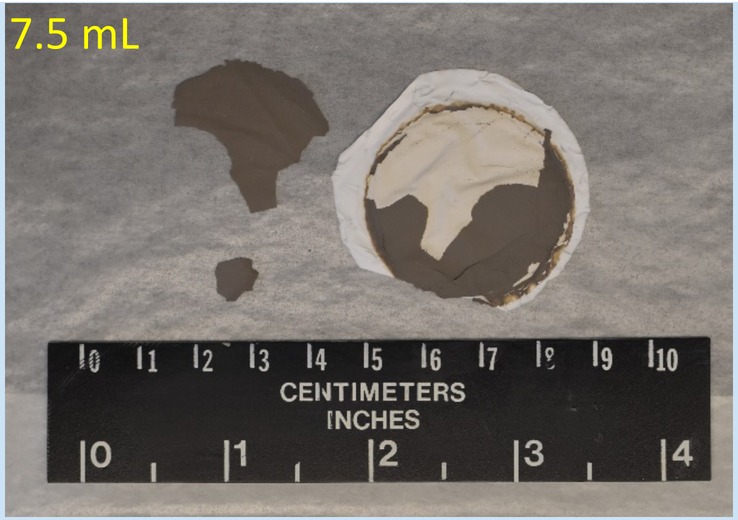
Unsuccessful mats

Filter Mats



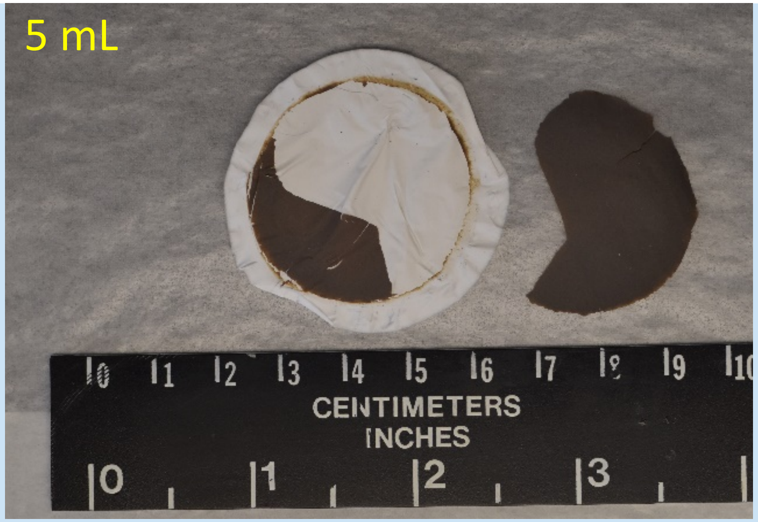
- Crumbled as soon as peeling was attempted

Cyclone Mats



- Small tear formed when freeing edges, caused the whole mat to rip

Q2B Mats

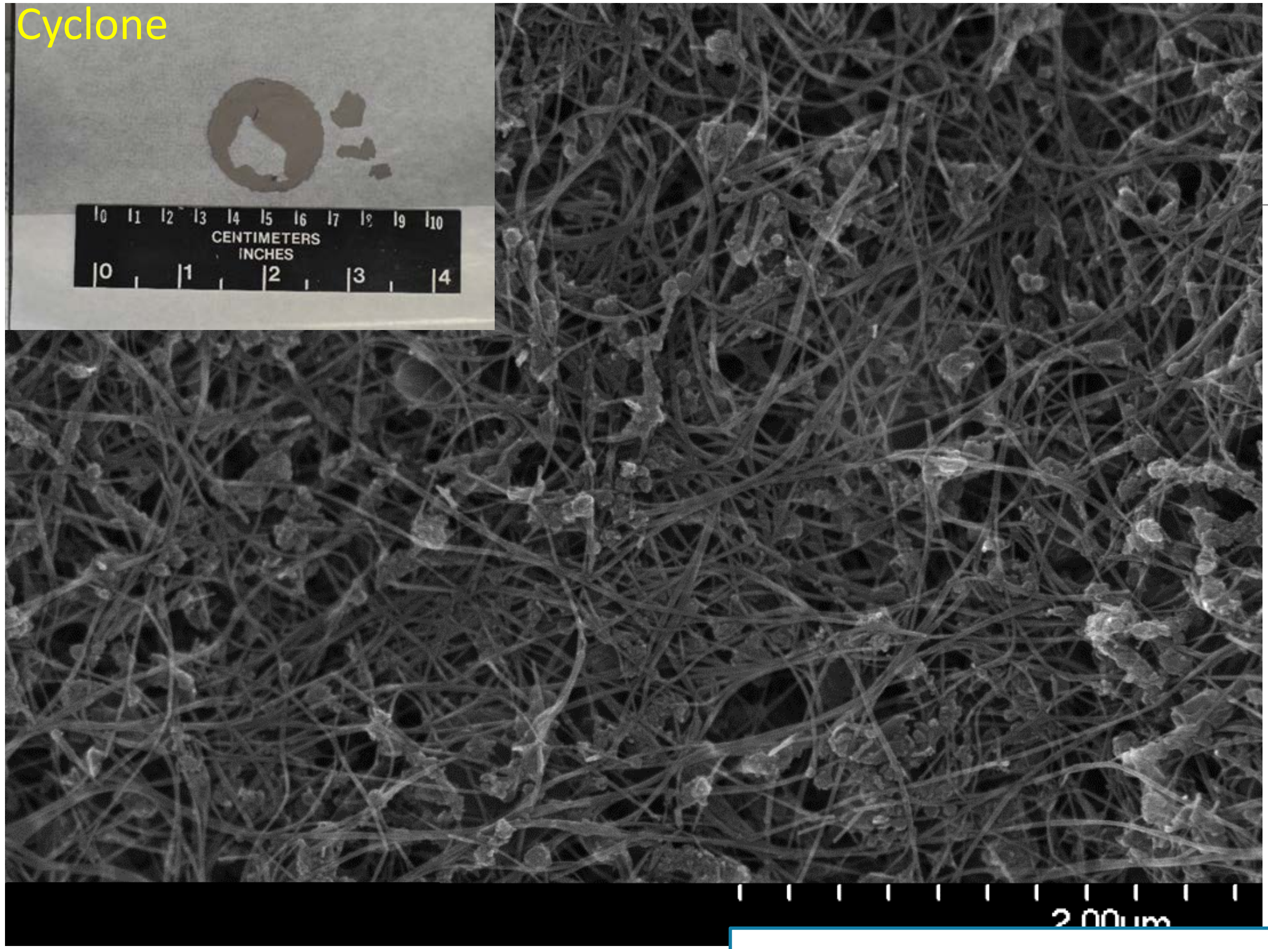


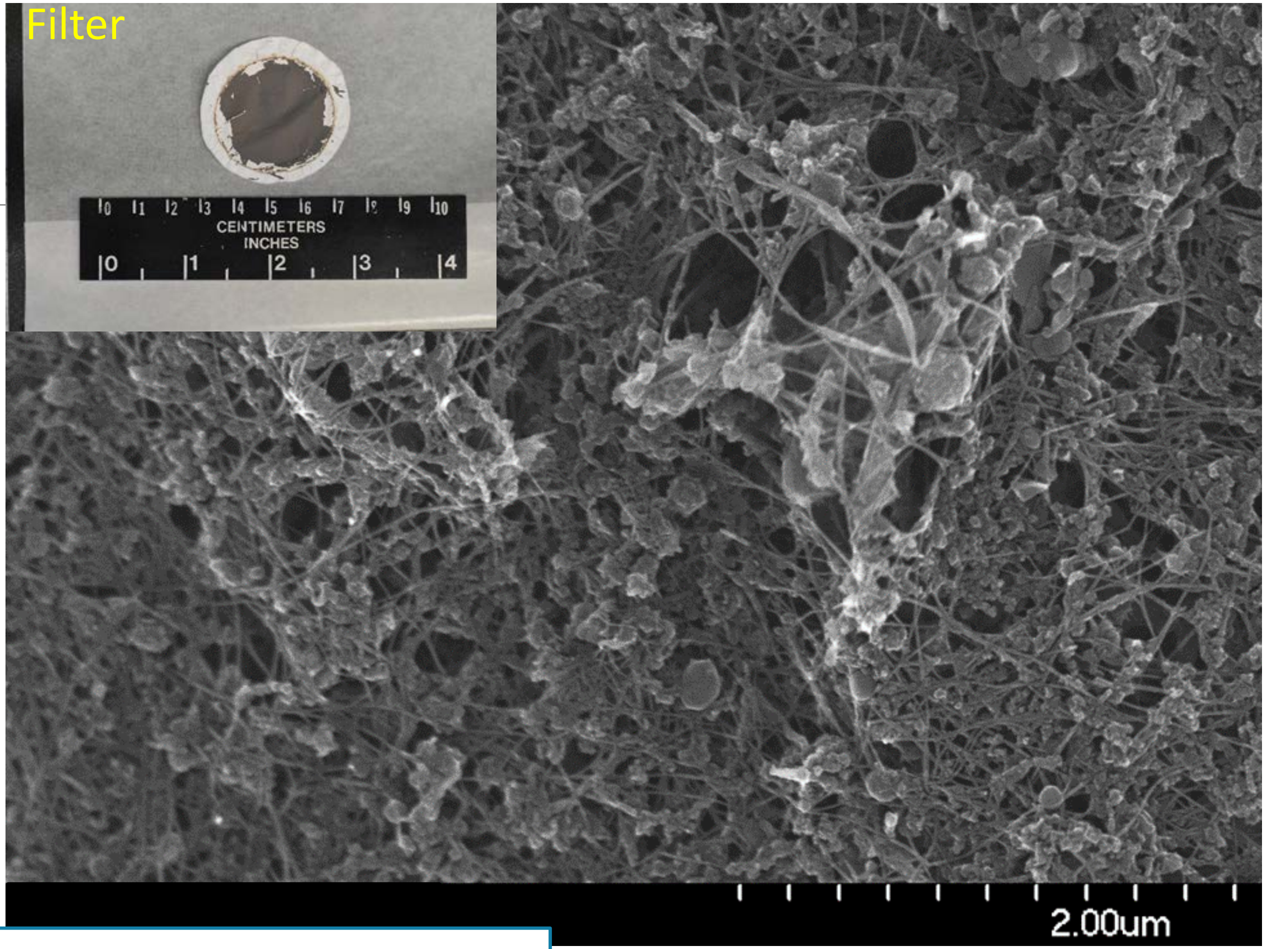
- Extremely thin and fragile during peeling

Goal: Thin Mats

- Mechanically superior
  - Flexible and tough
- More BNNTs present in thin films
  - Good for ceramic composites
- Works well for multilayered BNNT metal composites

Cyclone vs. Filter 5 mL Mat SEM (x 25k)

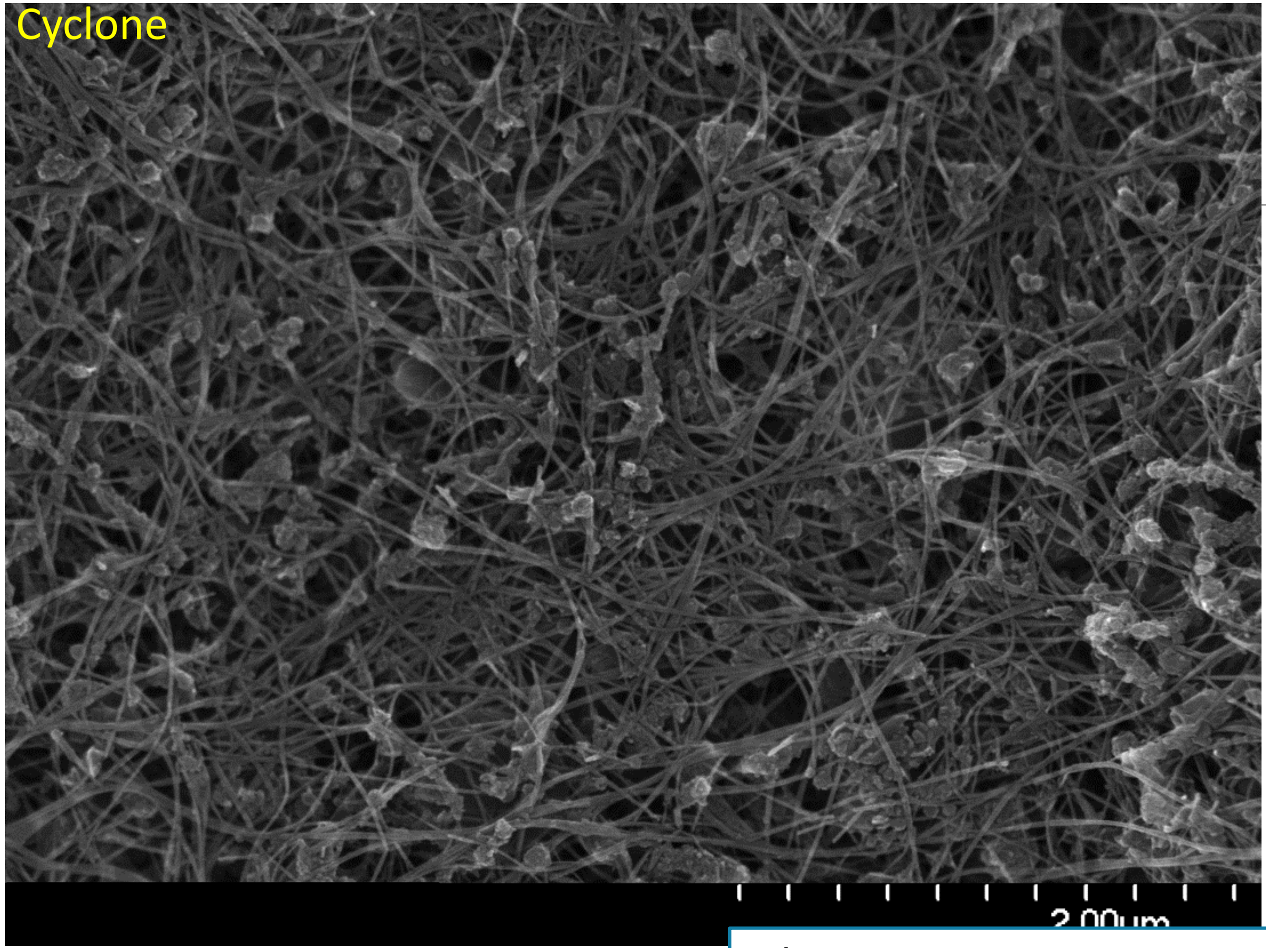


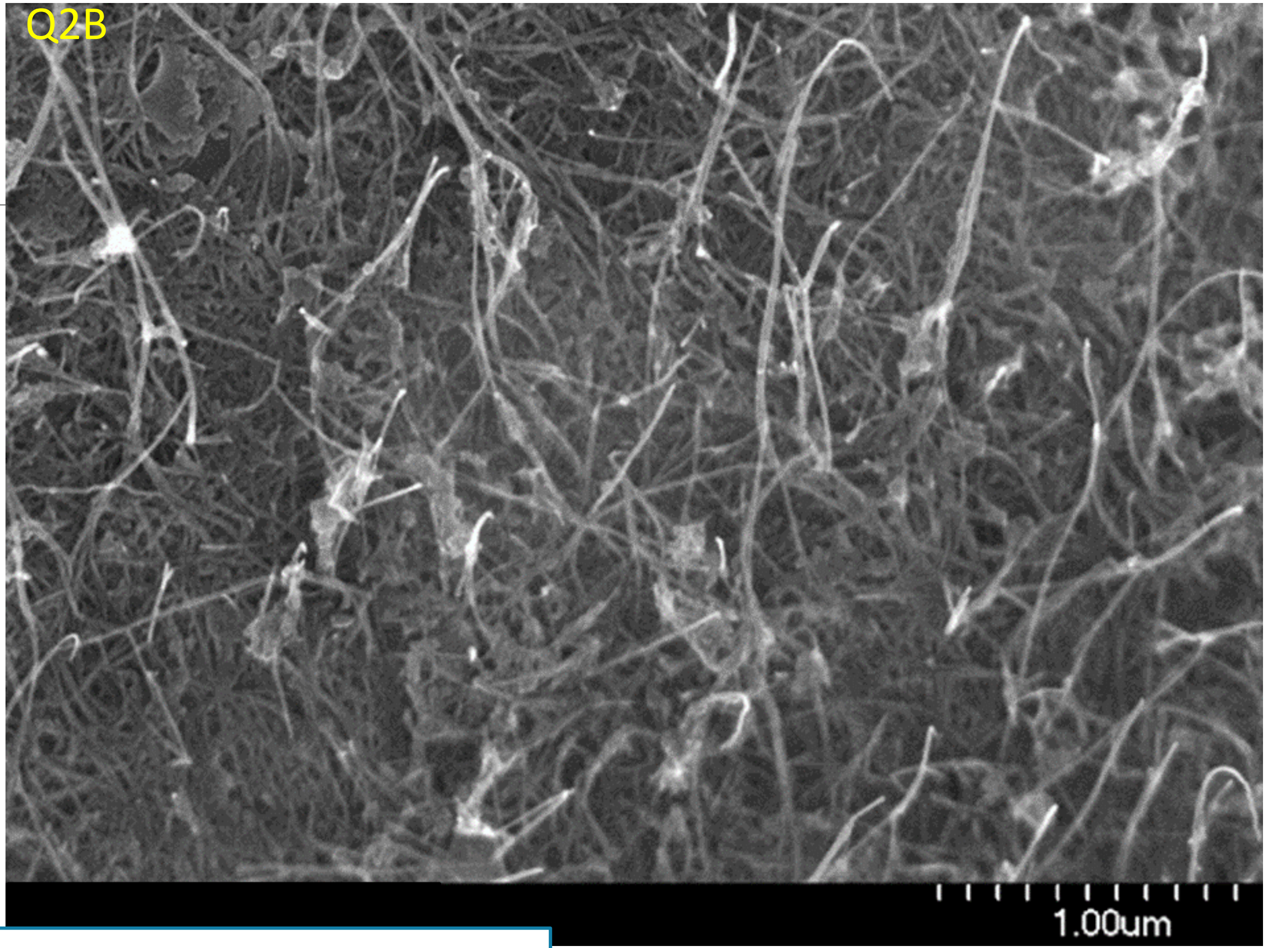


Observations:

- Filter mat has significantly more impurities present
- Can clearly see long, densely packed BNNTs in the Cyclone image

Cyclone vs. Q2B Mat SEM





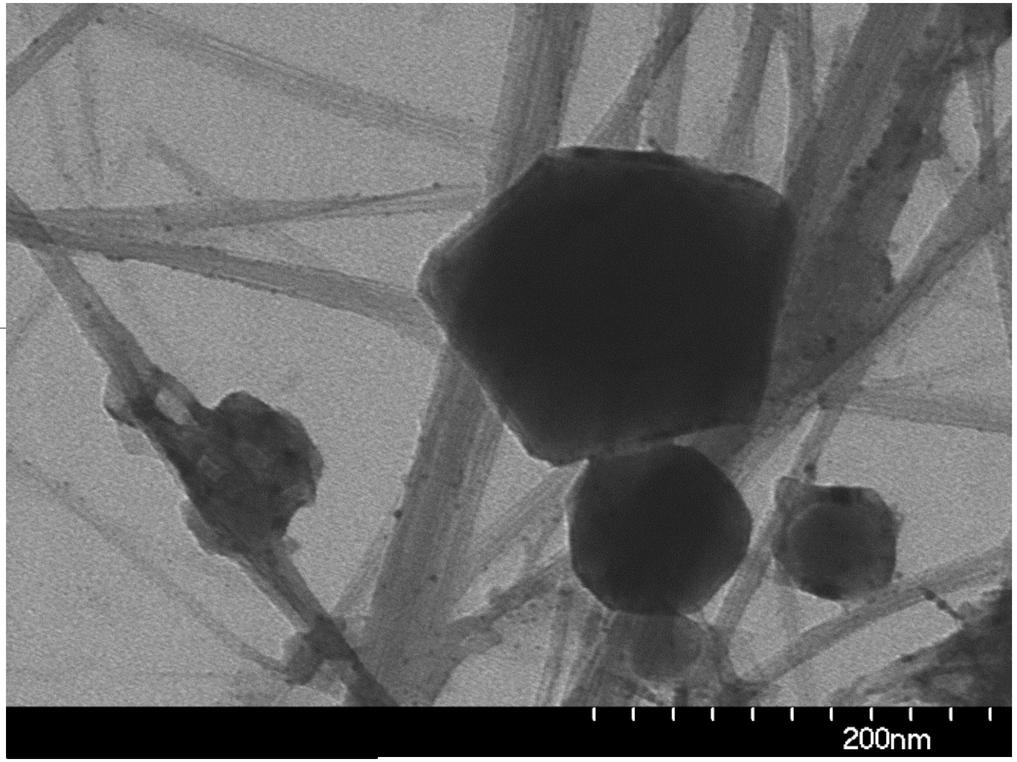
Observations:

- Q2B has less visible impurities than Cyclone
- Observed impurities could affect the creation of Cyclone and Filter mats as thin as the Q2B mats

BNNT Impurities

Types of Impurities:
- h-BN shells filled with Boron
- h-BN solids
- Pure Boron particles

Disadvantages:
- Poor mechanical properties
  - Ex. Brittle
- Poor thermal stability
  - Boron oxide melts at ~500C



Boron

Reacts with: O_2

$$4B + 3O_2 \rightarrow 2B_2O_3$$

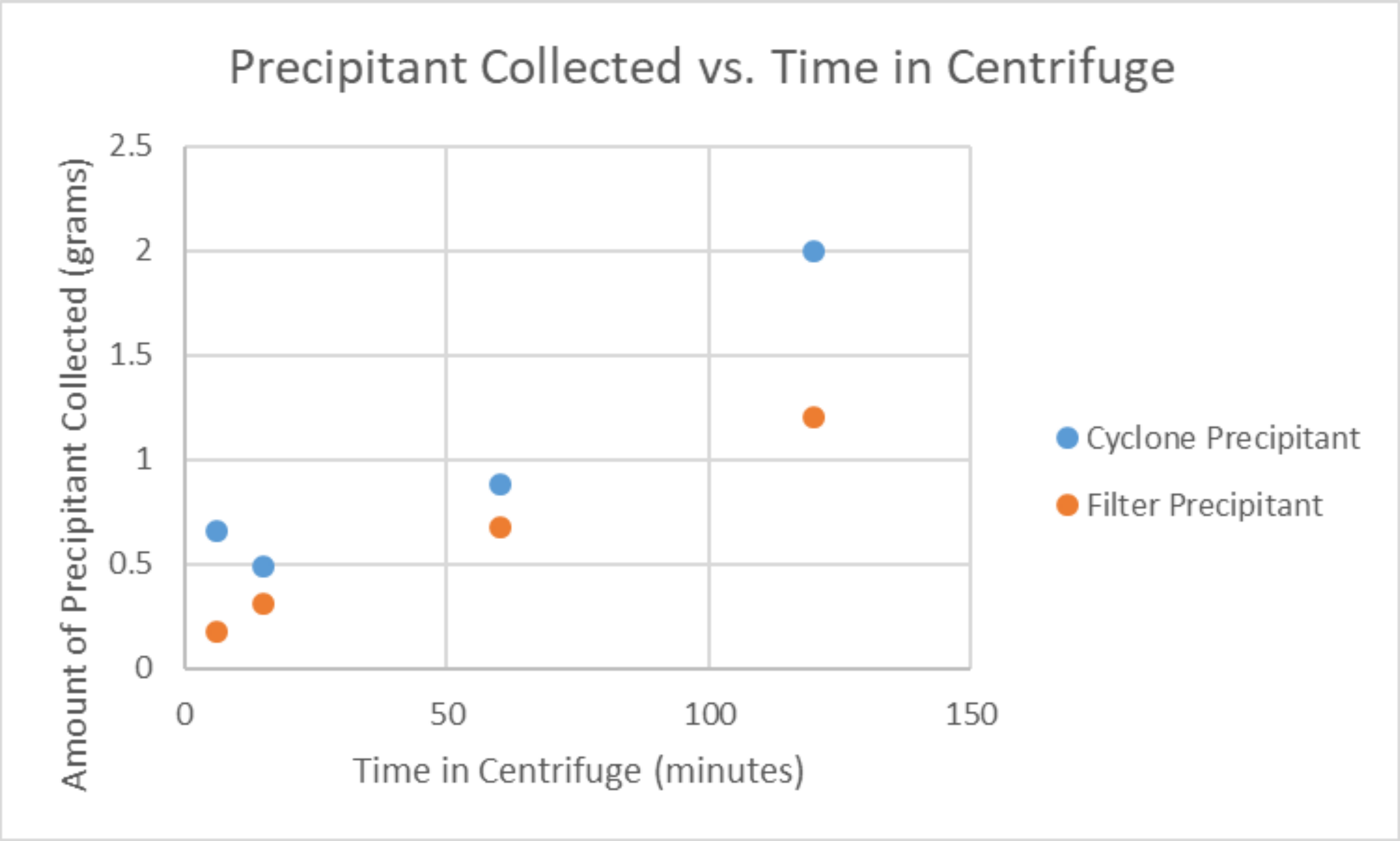
Boron Oxides

Reacts with: H_2O

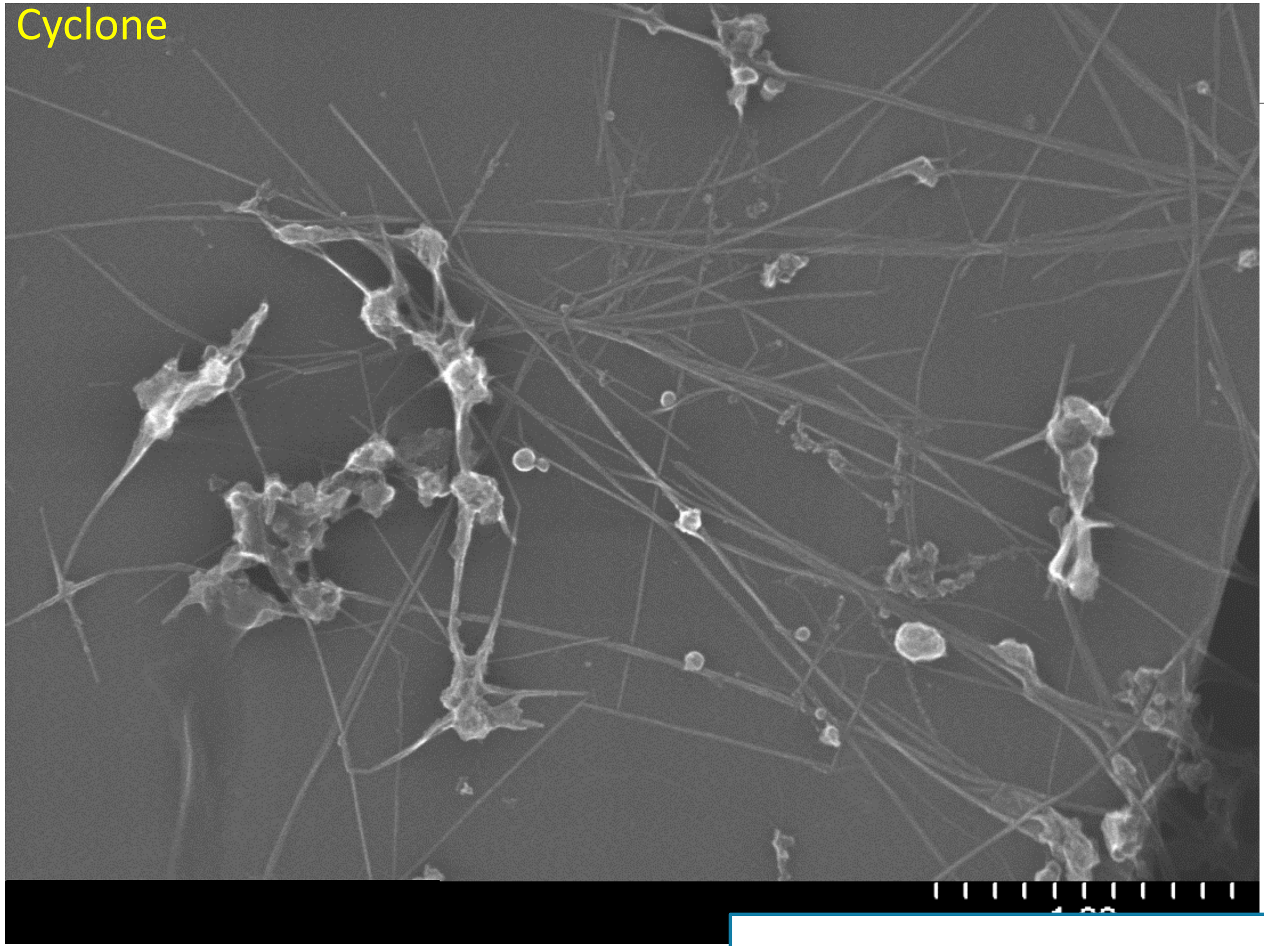
$$B_2O_3 + 3H_2O \rightarrow 2B(OH)_3$$

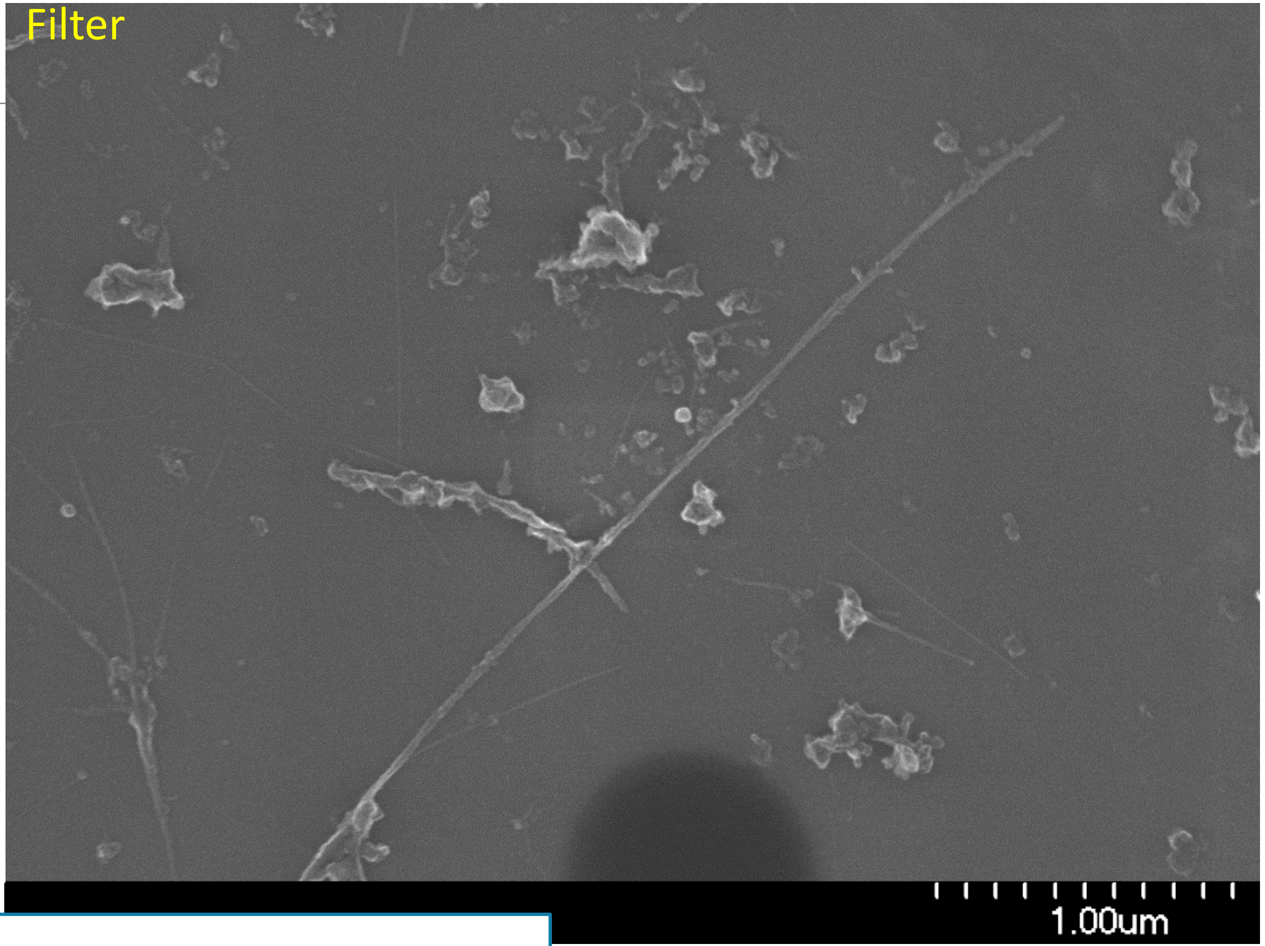
Boric Acid

Dissolves with: H_2O


Precipitant Collected vs. Time in Centrifuge
Amount of Precipitant Collected (grams)
2.5
2
1.5
1
0.5
0
0
50
100
150
Time in Centrifuge (minutes)
Cyclone Precipitant
Filter Precipitant

Cyclone and Filter Supernatant SEM (x 30k)



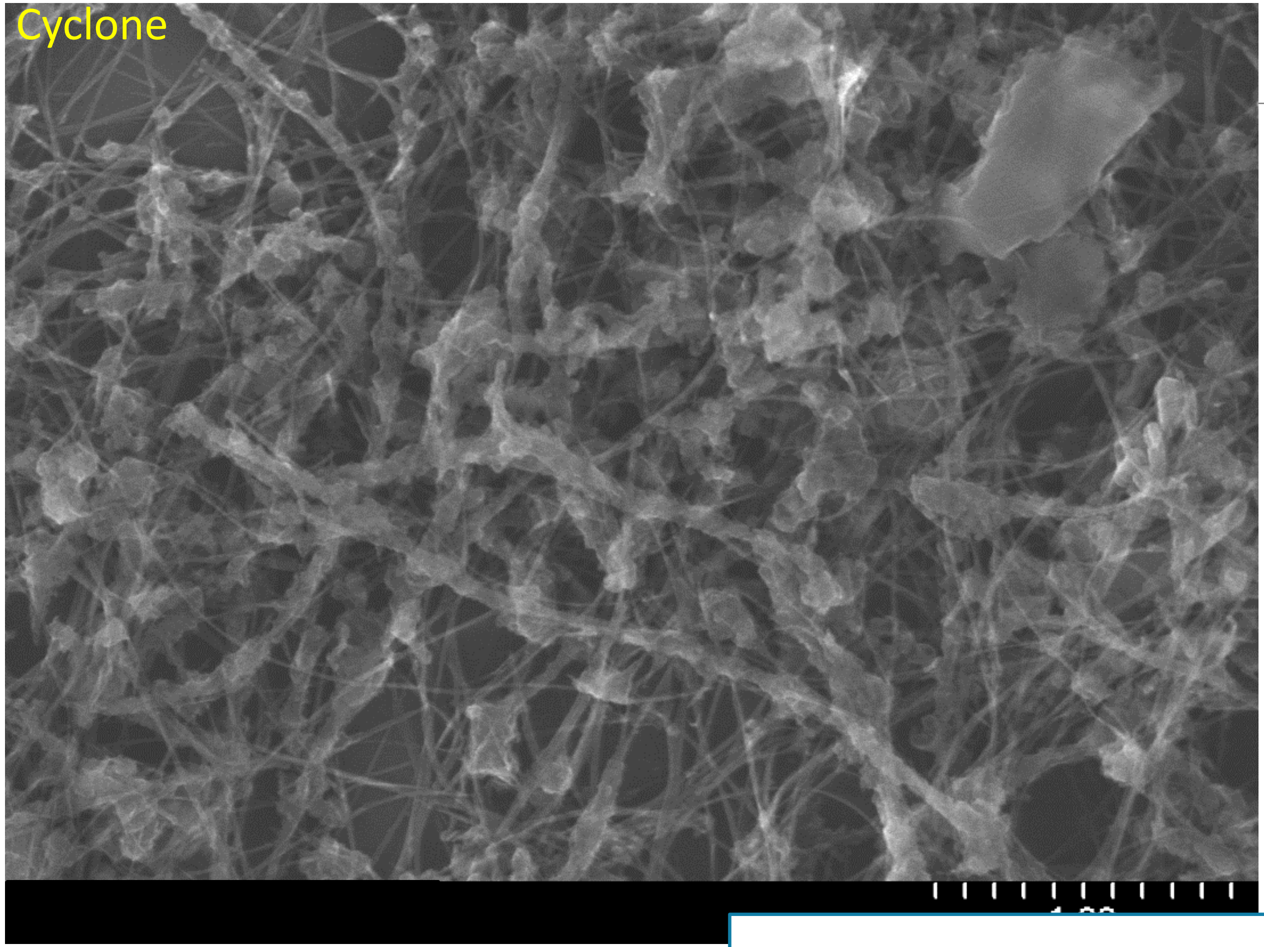


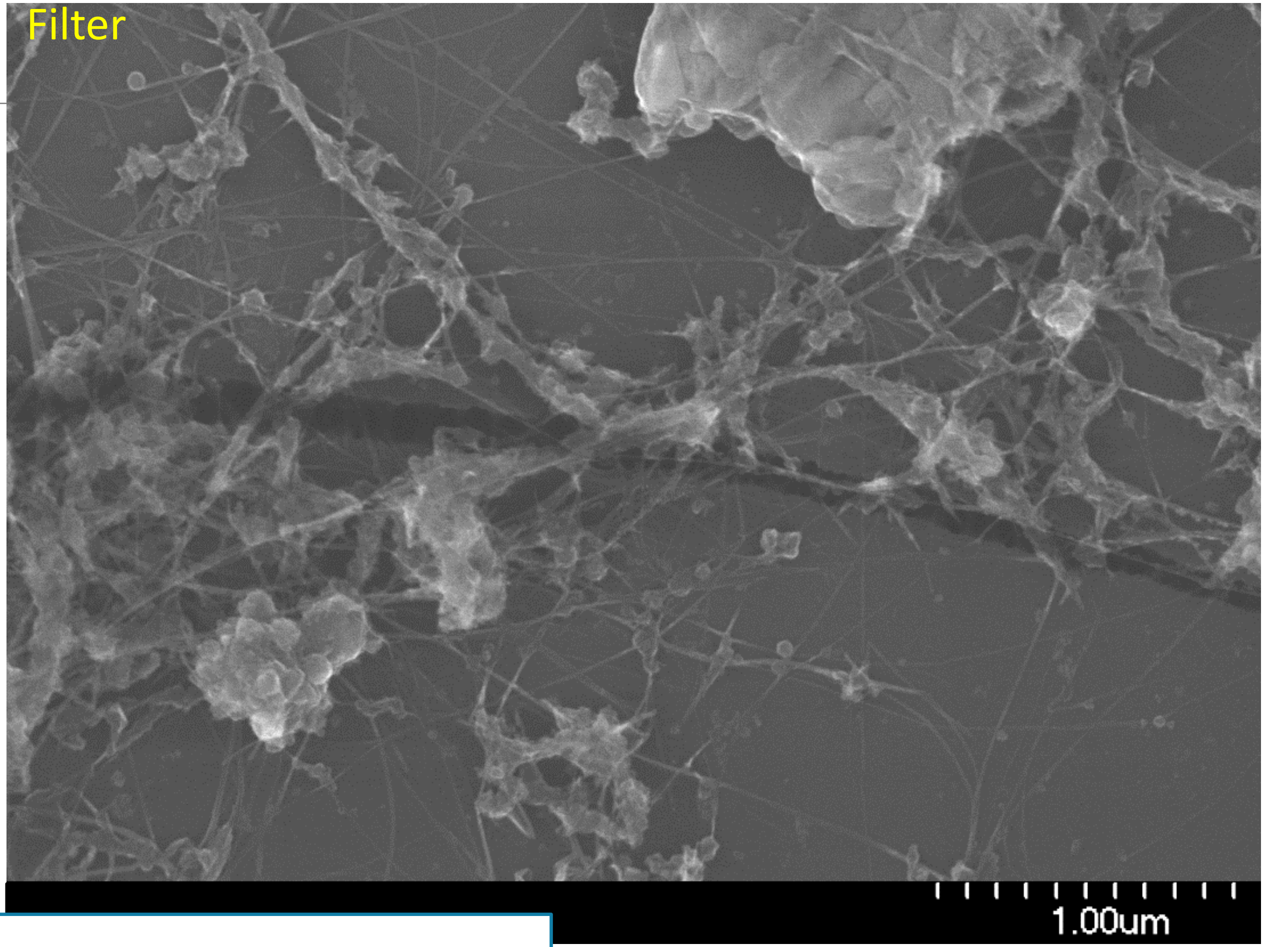
Looking for:

- Long tubes present with few impurities

Process still needs optimization

Cyclone and Filter Precipitant SEM (x 30k)



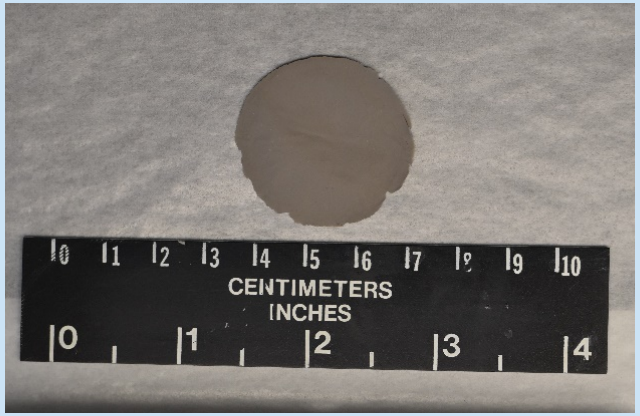
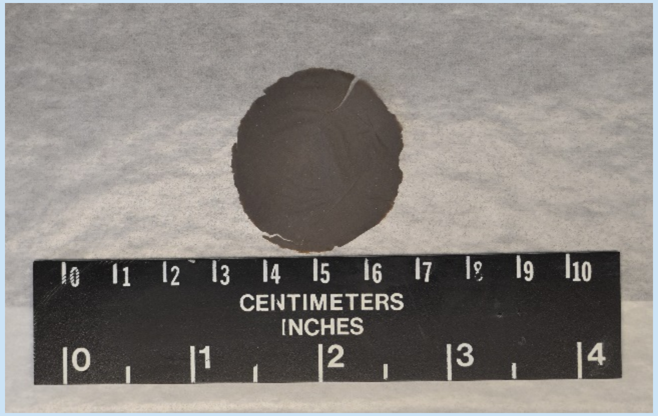
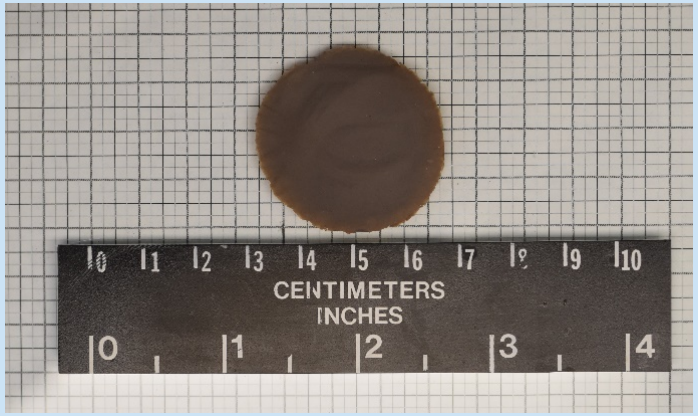


Looking for:

- Large particles that were collected with few tubes

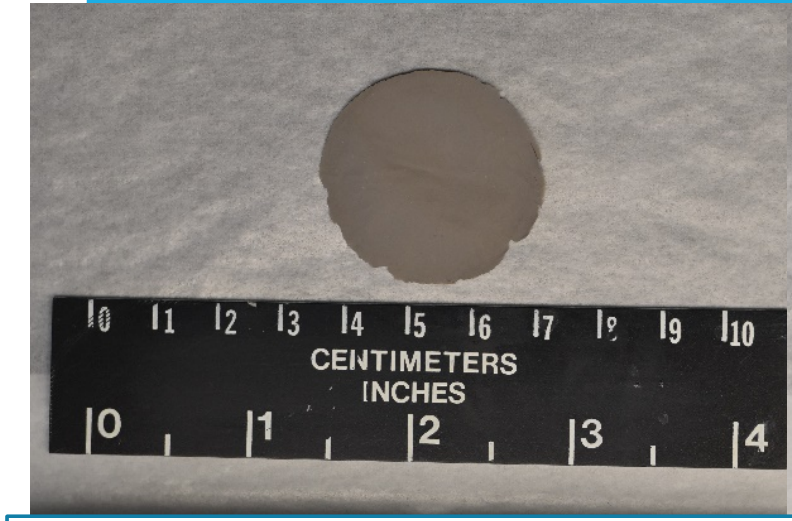
Process still needs optimization

Successful Mats

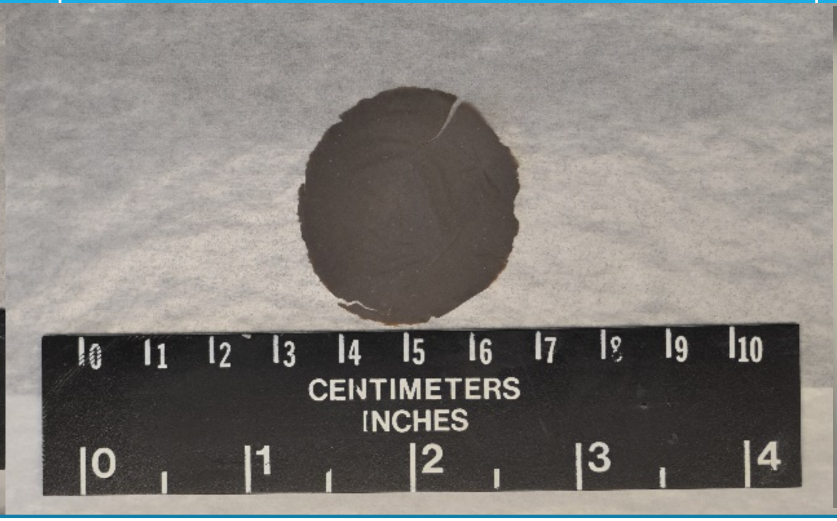
10 mL Cyclone Mat	7.5 mL Q2B Mat	Diluted Q2B Mat (5:5)
		
• Mass: 15.5 mg • Density: 0.833 g/cm^3 • Thicker than previous Cyclone mat attempt, aided by improved peeling method	• Mass: 12.8 mg • Density: 0.645 g/cm^3 • Cracks formed after peeling during extra drying	Measured after being cut for composite use: • Density: 1.10 g/cm^3 • Slow peeling process using new procedure • Extremely difficult to reproduce

Successful Mats

10 mL Cyclone Mat



7.5 mL Q2B Mat



Diluted Q2B Mat (5:5)

Reasons for success:

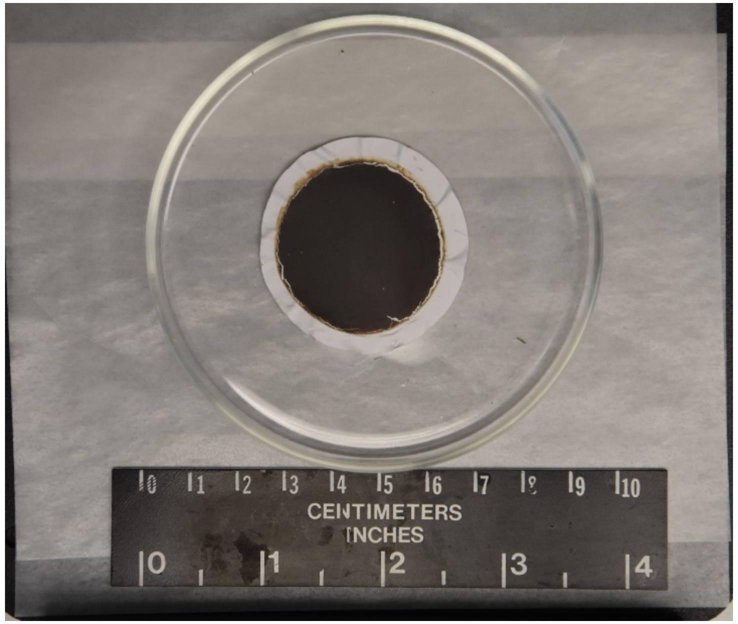
- Improved peeling method – Elle’s method
    - Freeing edges, then
    - Flipping mat over and peeling the filter back
- Worked with thicker mats
    - Gradually made thinner mats

* Some mats still were unsuccessful, even with improved techniques *

Acetone Bath Peeling Method

Method:

1. Free the edges of the mat slightly
2. Place in petri dish and fill dish with acetone
3. Leave to soak until the acetone is all evaporated
4. Wait until the filter is visibly dry (solid white again)
5. Free the edges a bit more
6. Flip over and continue with peeling method

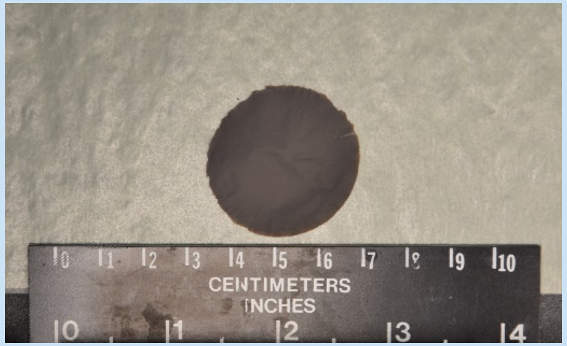
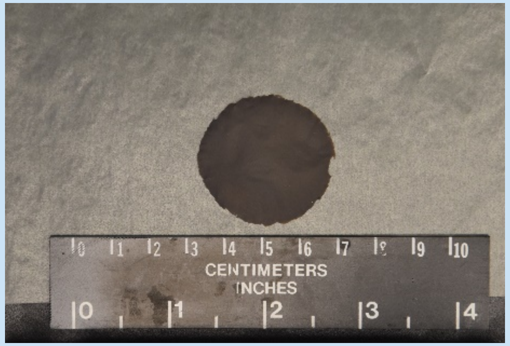
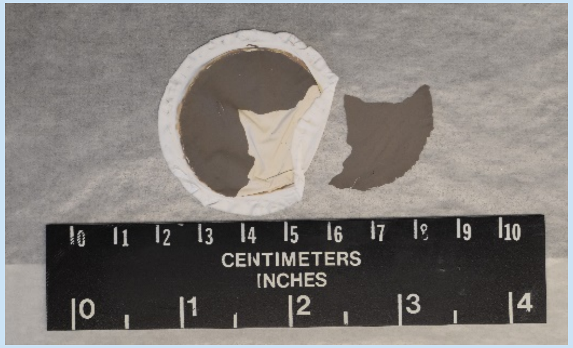
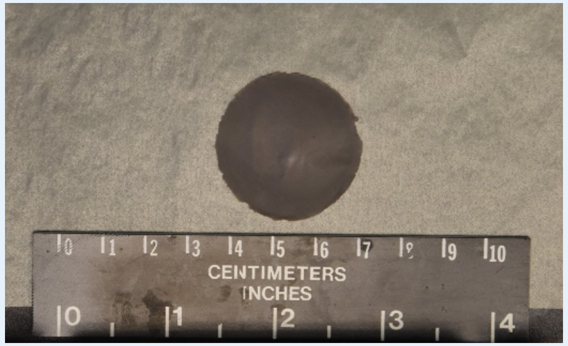
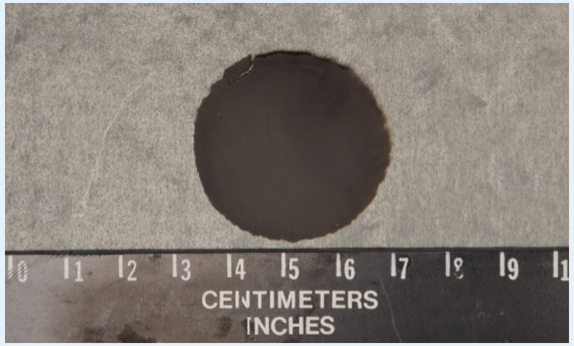
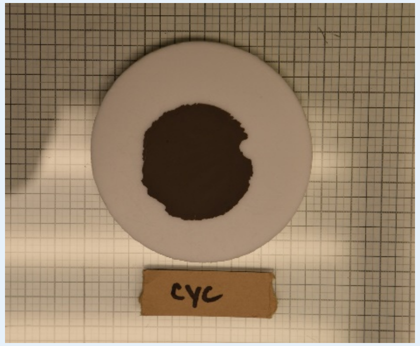


Observations:
- Easier to peel
- Less fragile
- Slightly thicker
- Denser mats

Theory:
- Acetone will cause the BNNTs to shrink away and detach from the filter

Note:
- Also began trials with ethanol and methanol soaked mats

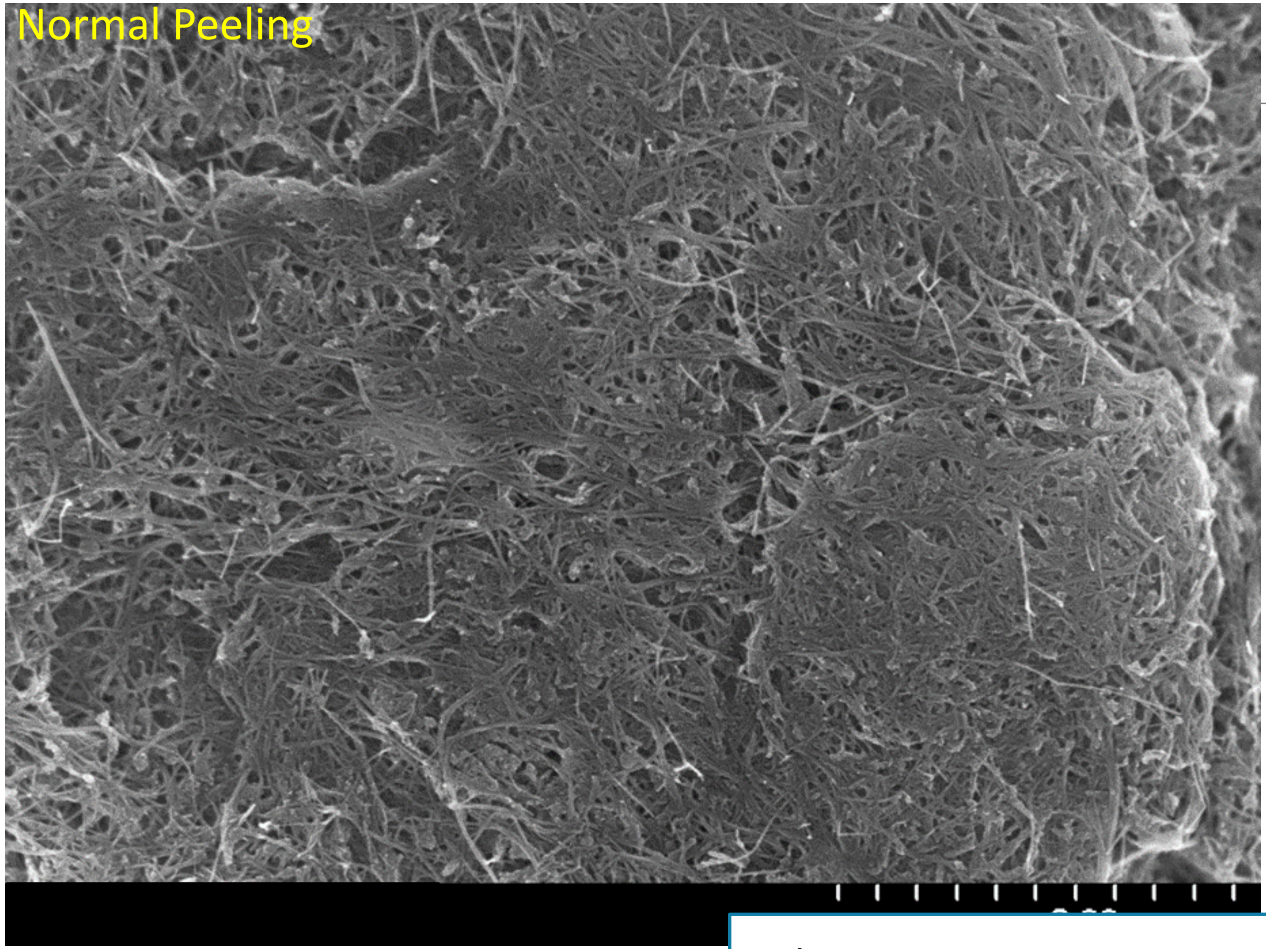
Acetone Bath Mats

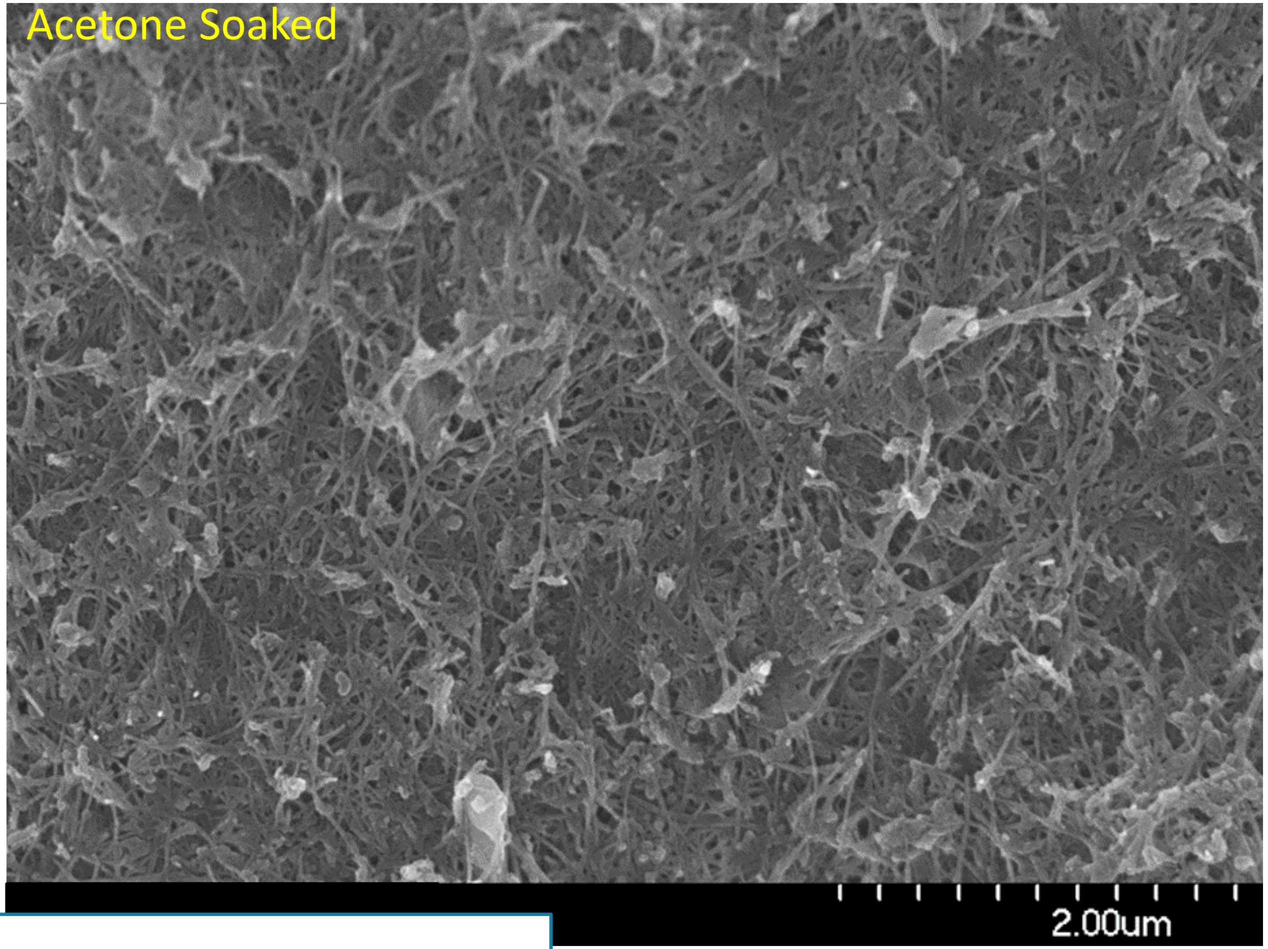
	5.5 mL Q2B Mat	5 mL Q2B Mat	7.5 mL Cyclone Mat
Normal Peeled Mats			
Acetone Bath Mats			

Observations:
- Mats had small tears that would normally have caused damage, but were able to be peeled successfully

Question:
- What effect did the acetone have on the morphology of the mat?

6 mL Q2B Mat SEM:
Normal vs. Acetone Peeling (x 20k)

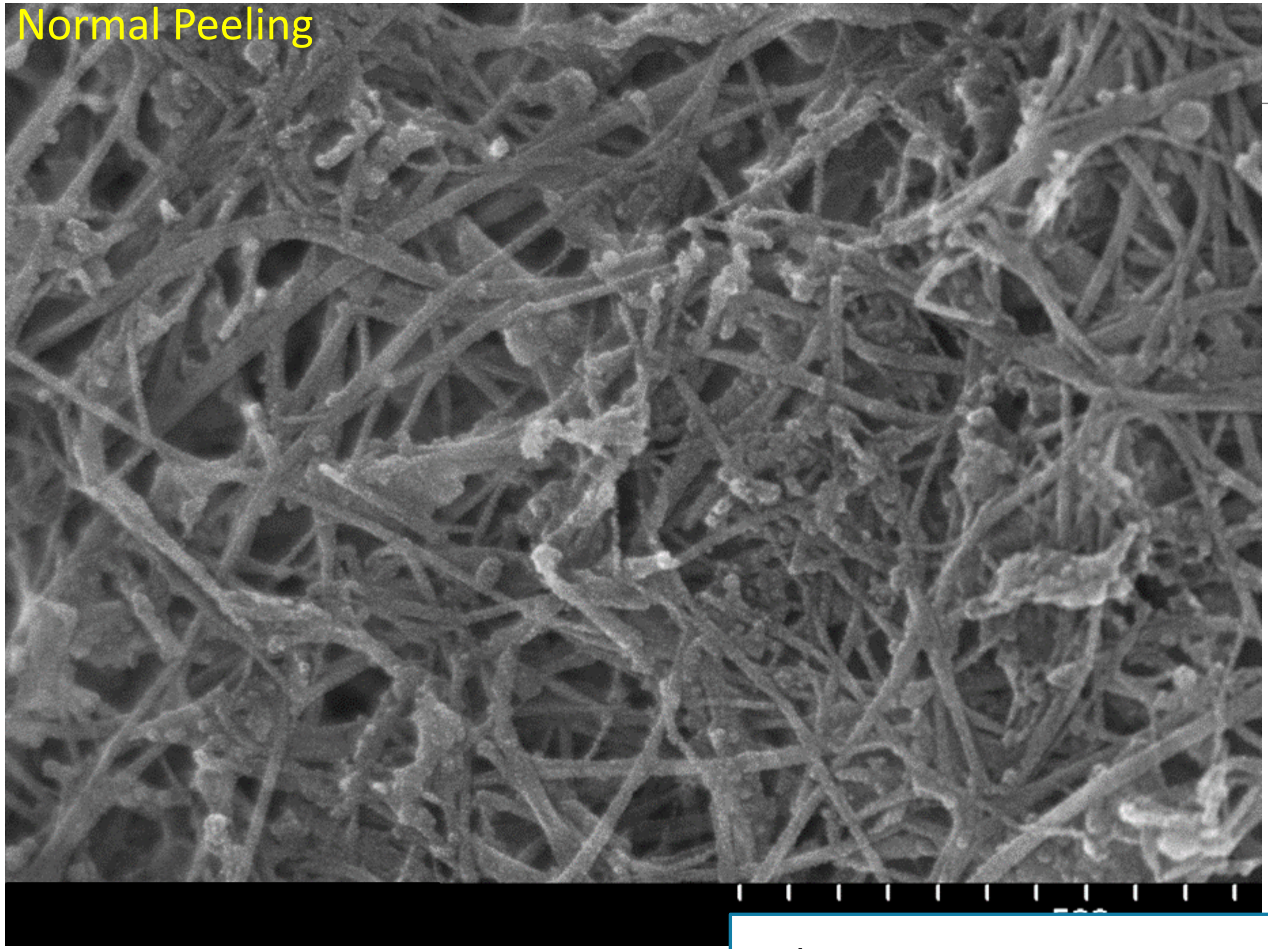


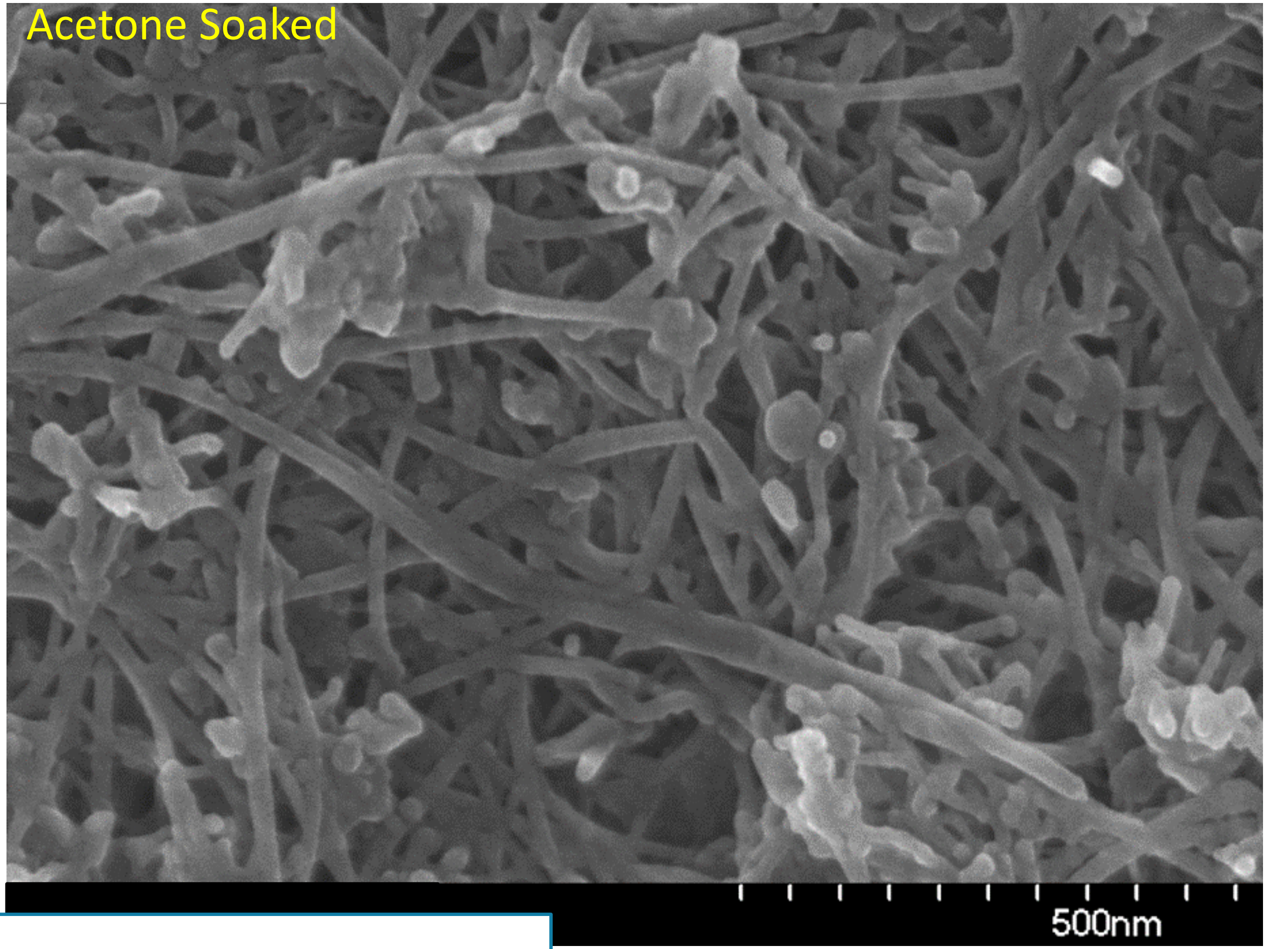


Observations:

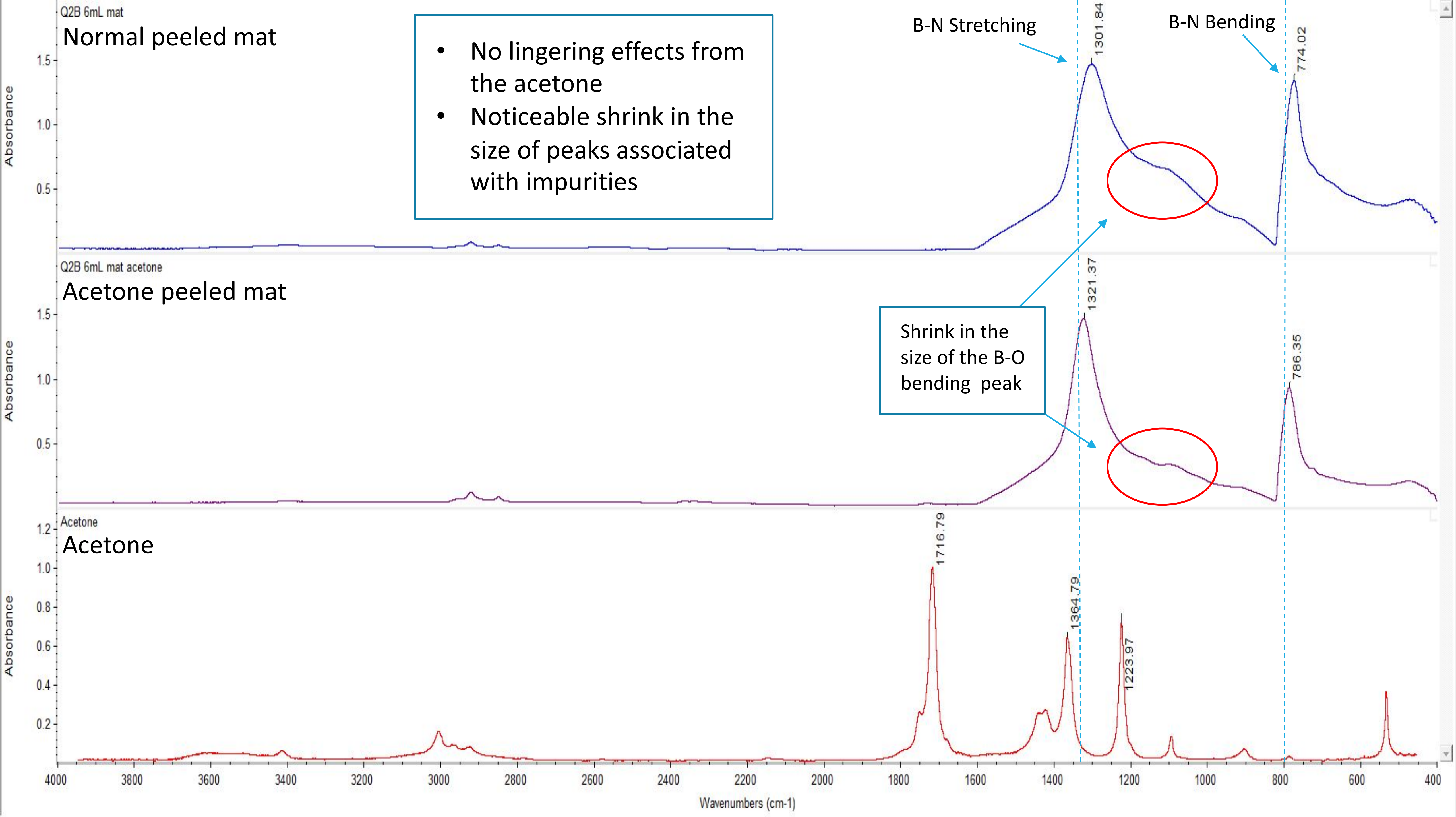
- Slight change in the observable porosity of the mats

6 mL Q2B Mat SEM:
Normal vs. Acetone Peeling (x 100k)





Observations:
- Less impurities
- BNNTs appear thicker



Q2B 6mL mat
Normal peeled mat
No lingering effects from the acetone
Noticeable shrink in the size of peaks associated with impurities
B-N Stretching
B-N Bending
1301.84
774.02
Q2B 6mL mat acetone
Acetone peeled mat
1321.37
786.35
Shrink in the size of the B-O bending peak
Acetone
1716.79
1364.79
1223.97
Absorbance
Wavenumbers (cm-1)

Conclusions

- Plasma tubes may be too impure for use in applications without purification
  - Q2B works well for making thin, high-density mats
    - Good potential for use with ceramic and metal matrix composites
- Soaking mats in acetone may be a viable option for removing impurities
- Centrifuge process also shows potential for removal of impurities

- Future Work:
  - Further investigation of the effects of the centrifuge on the BNNT solution
    - Optimization of the centrifuge process
  - Exploration of soaking mats before peeling
    - Methanol
    - Ethanol
  - Develop new mat procedure to include acetone soaking

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- Valerie Ellis
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