Fabrication of Boron Nitride Fibers by Force Spinning Method

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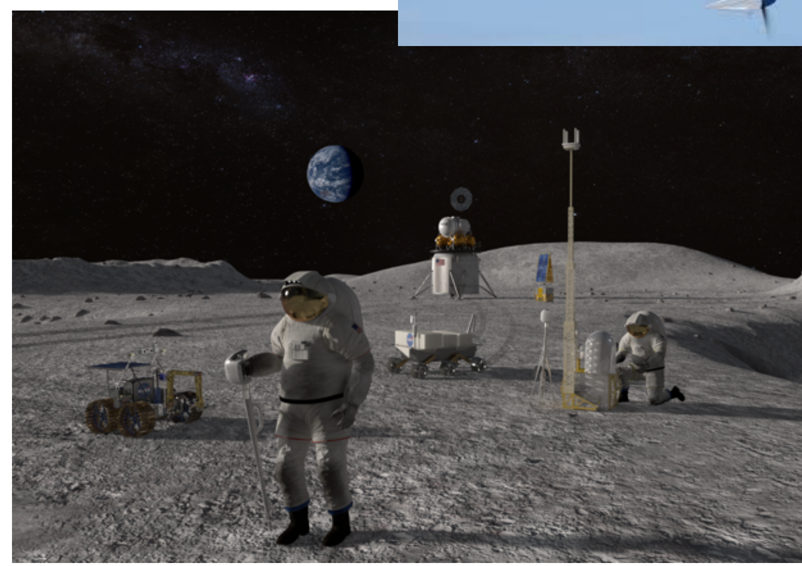
Aeronautics and Space Technology Need

<u>Vision</u>:

- Development of a material that combines chemical inertness, lightweight and high strength with high electrical resistivity and high thermal conductivity
- BN nanofibers will enhance material development for electric propulsion and other space applications:
  - Electrical insulation for high voltage components
  - Re-entry shielding
  - Radiation shielding
  - Space structures



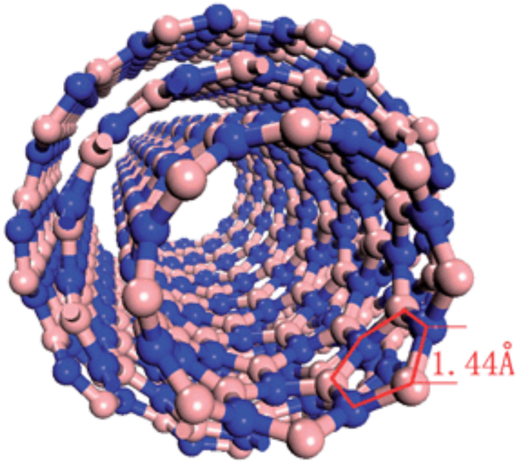
<u>Goal</u>: Develop next generation fiber technology for multifunctional opportunities

Why Boron Nitride Fibers?

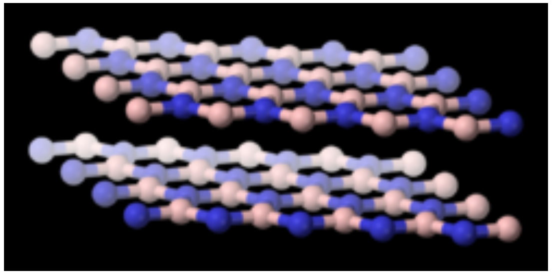
- **BN Good Electrical Insulation Properties**
  - Constant wide band gap above 5.2 eV
  - On BN nanotube, it's independent of diameter, chirality or number of tubular walls
- **High Thermal Conductivity**
  - Thermal Conductivity of 600 $W(m°K)^{-1}$
  - Larger thermal conductivity than CNTs
  - Ability to dissipate heat in nanoelectrics
- **Chemically and Thermally Stable**
  - Hydrophobic
  - Chemical stability
  - Oxidation in air above 1100 °C
- **Fiber form brings continuity of properties on composites**

Multifunctional Properties for Different Applications

BNNT

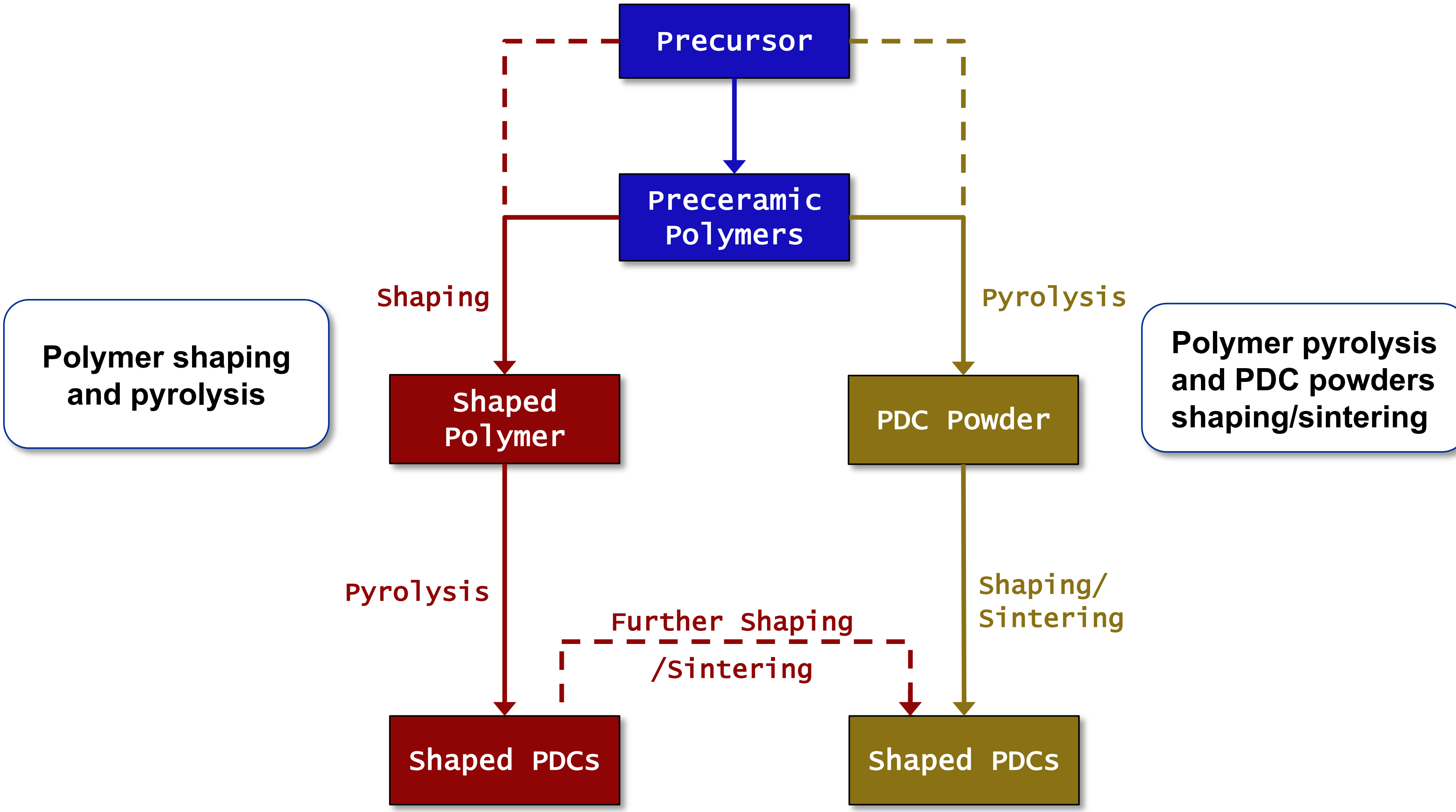


H-BN



http://en.wikipedia.org/wiki/Boron_nitride, http://pubs.rsc.org/en/content/articlelanding/2008/jm/b804575e#!divAbstract

Polymer Derived Ceramic Process



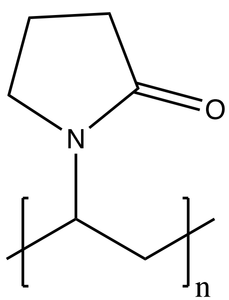
S. Bernard and P. Miele, *Materials* **2014**, *7*, 7436-7459

Forcespinning Machine

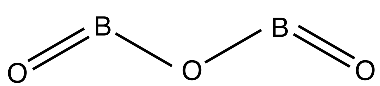
- High-yield fiber preparation
- Fibers fabricated by centrifugal forces spinning a polymeric solution into fine continuous fibers
- Fiber diameters can be tailor to ranges between ≈ 200 nm to 5 μm
- Diameters depend on precursor properties (like molecular weight), solution viscosities, and spinning speeds
- Fibers can be up to 6 ft in length

Research Plan: Polymer Shaping and Pyrolysis

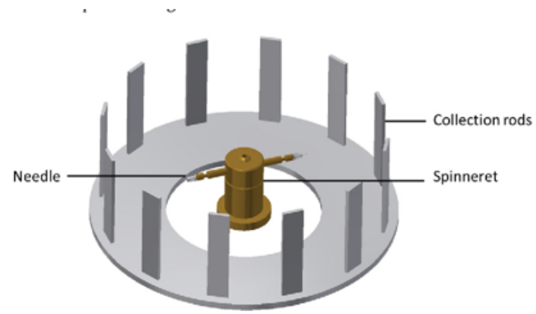
Precursors → Preparation of Solution → Shaping Fiber → UV Lamp Crosslink → Fiber Conversion → Characterization



Polyvinylpyrrolidone (PVP), C_6H_9NO



Boron Oxide, (Borea) B_2O_3

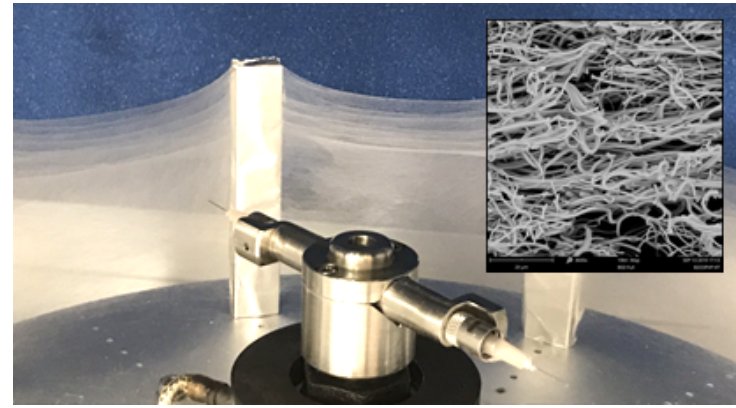


Easy, fast, and high yield process with minimum chemical wastes

Shaping Fibers

Observations:

- All solutions produced fibers
- Different concentrations gave different properties:
  - Dry or sticky
  - Strong or weak
  - Sensitivity to humidity

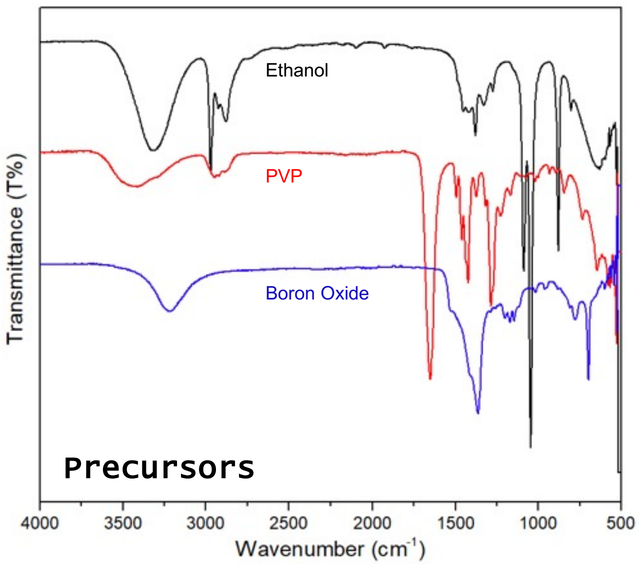


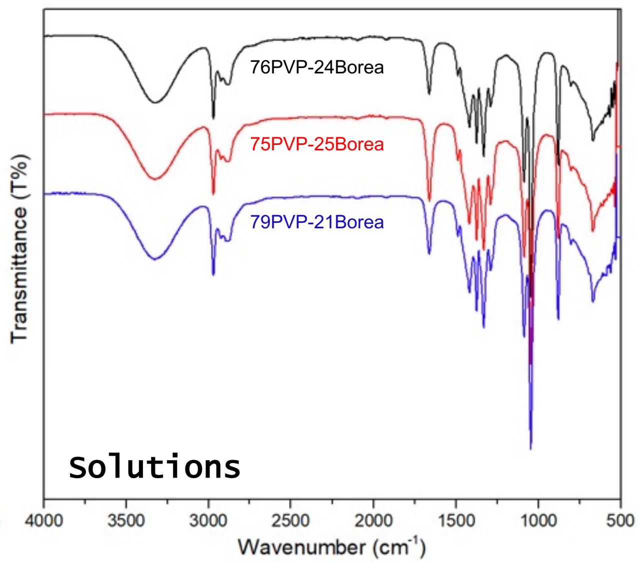
76PVP-24Borea

75PVP-25Borea

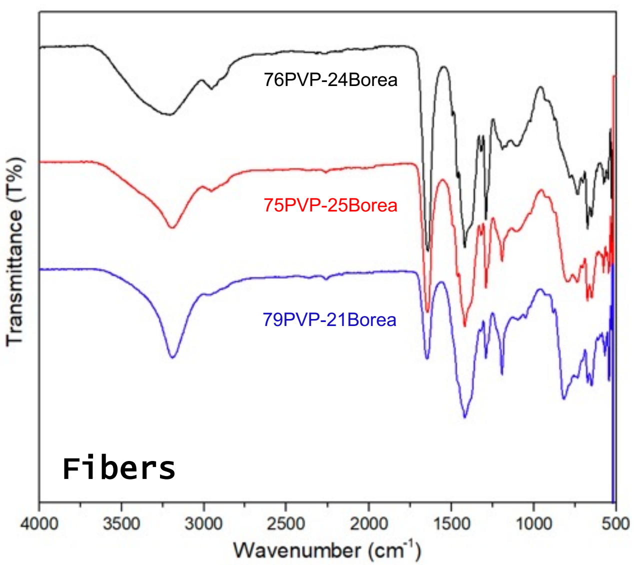
71PVP-29Borea

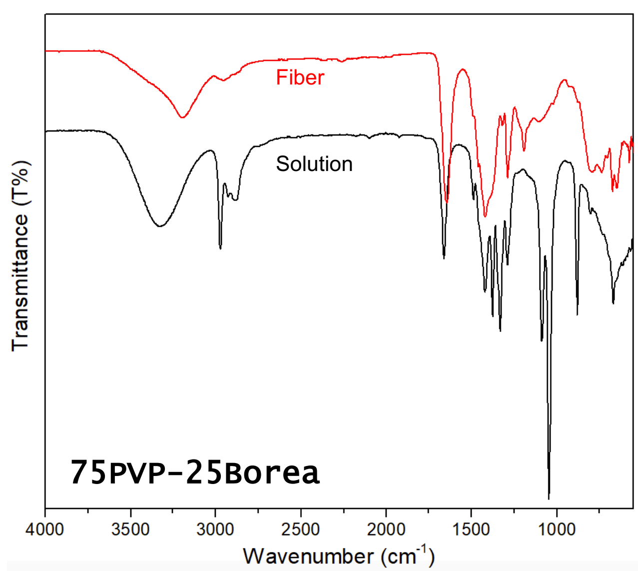
FTIR of Precursors, Solutions and Fibers





- Spectrum of solutions look the same
- Fibers produced from these solutions have small differences in their spectra among them
- Ethanol characteristic peaks are not in the fibers' spectrum

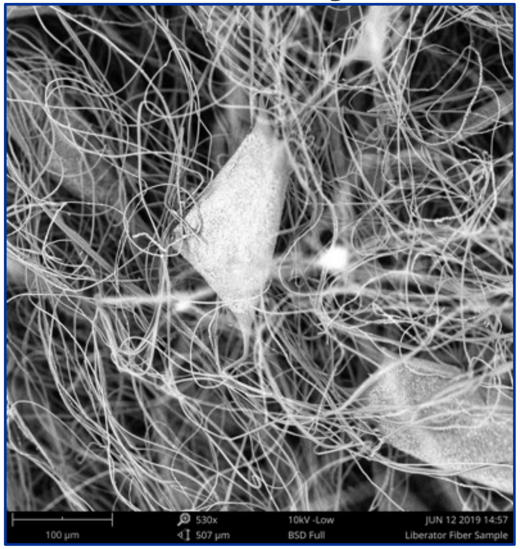


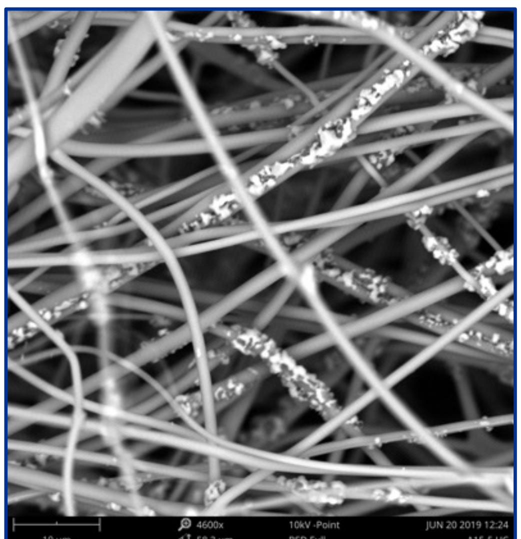


Fiber 75PVP-25Borea was used for next processes

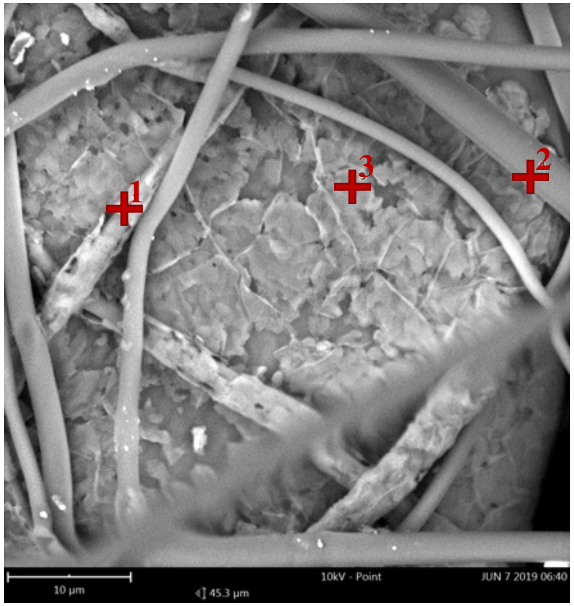
SEM & EDS Characterization of Fibers

SEM Analysis





EDS Analysis



Spot 1

Element Symbol	Atomic Conc.	Weight Conc.
O	69.70	75.06
C	13.27	10.72
N	8.47	7.98
B	8.57	6.23

Spot 2

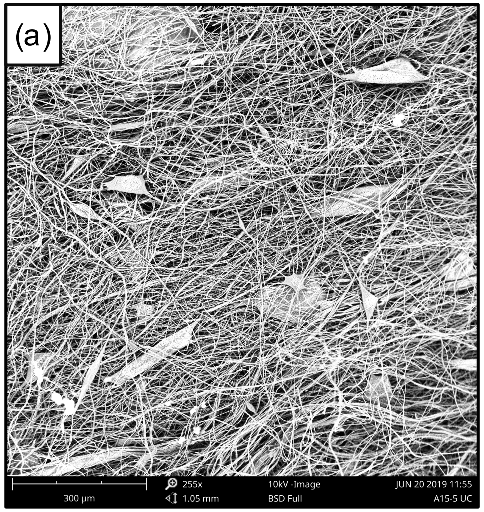
Element Symbol	Atomic Conc.	Weight Conc.
O	62.43	68.57
C	21.96	18.11
N	7.95	7.65
B	7.65	5.68

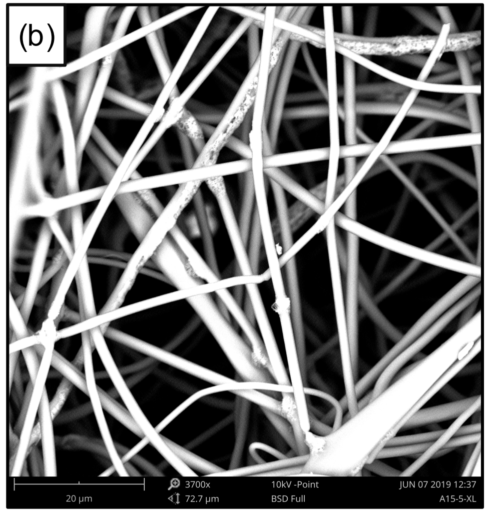
Spot 3

Element Symbol	Atomic Conc.	Weight Conc.
O	67.50	72.92
C	15.50	12.57
N	9.69	9.16
B	7.32	5.34

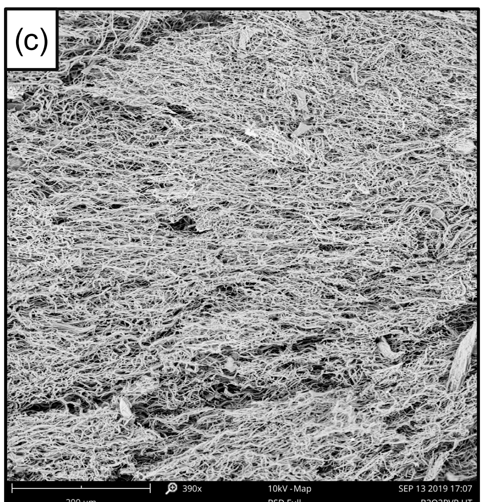
UV Crosslinking & Conversion

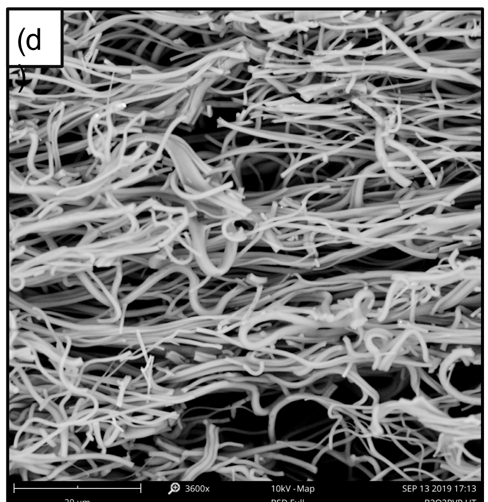
SEM of Fiber-Crosslinked: (a) and (b)



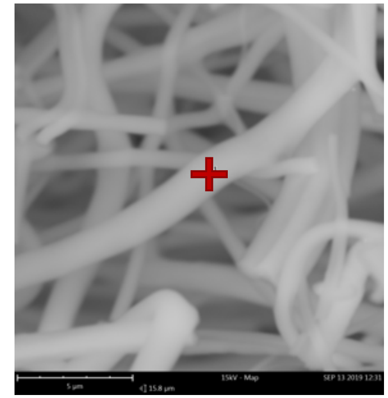


SEM of Fiber-Crosslinked-HT#1: (c) and (d)





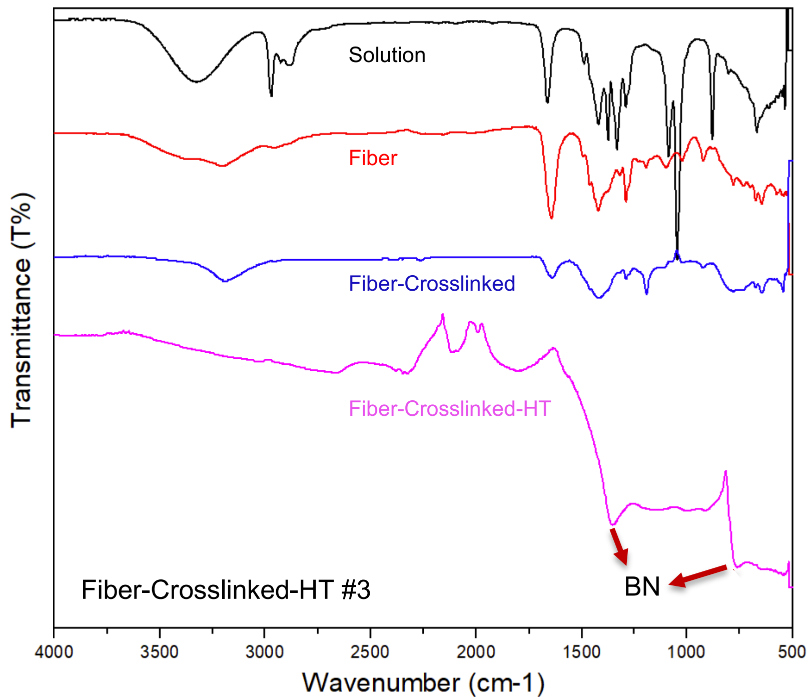
EDS of Heat-Treated Fibers



Element Symbol	Atomic Conc.	Weight Conc.
O	43.97	49.44
N	32.87	32.36
B	16.04	12.19
C	7.12	6.01

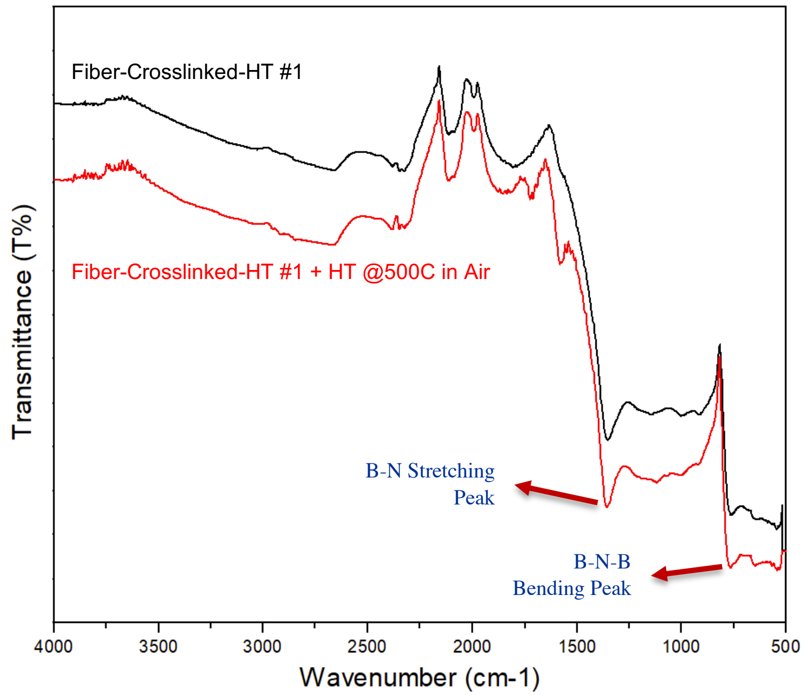
- Heat Treatment Process #1
  - Dark gray in color
  - Mechanically strong at touch
  - Look shorter or being cut
- EDS shows 2 times of N atomic concentration than B

FTIR Analysis of Fibers

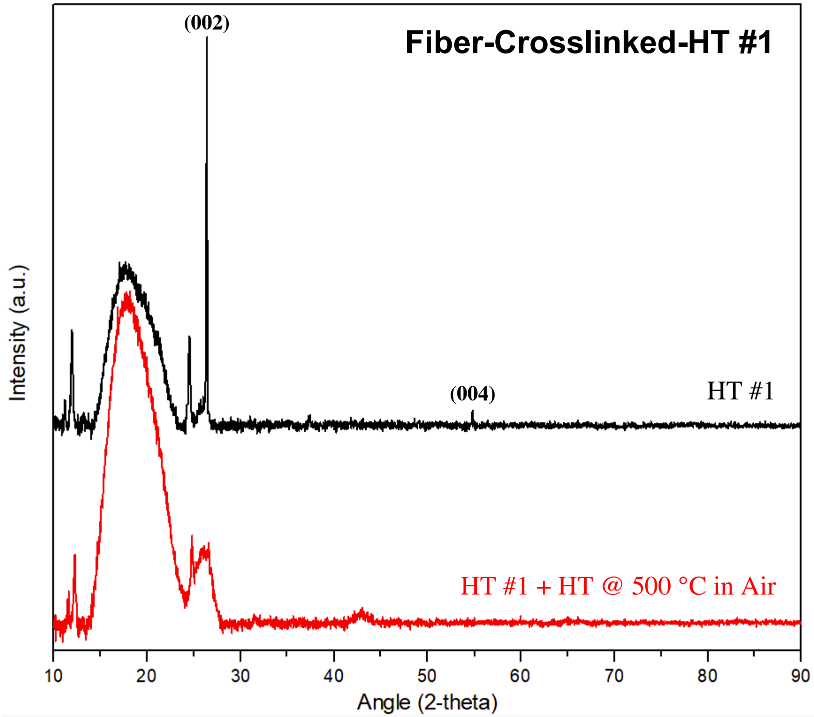


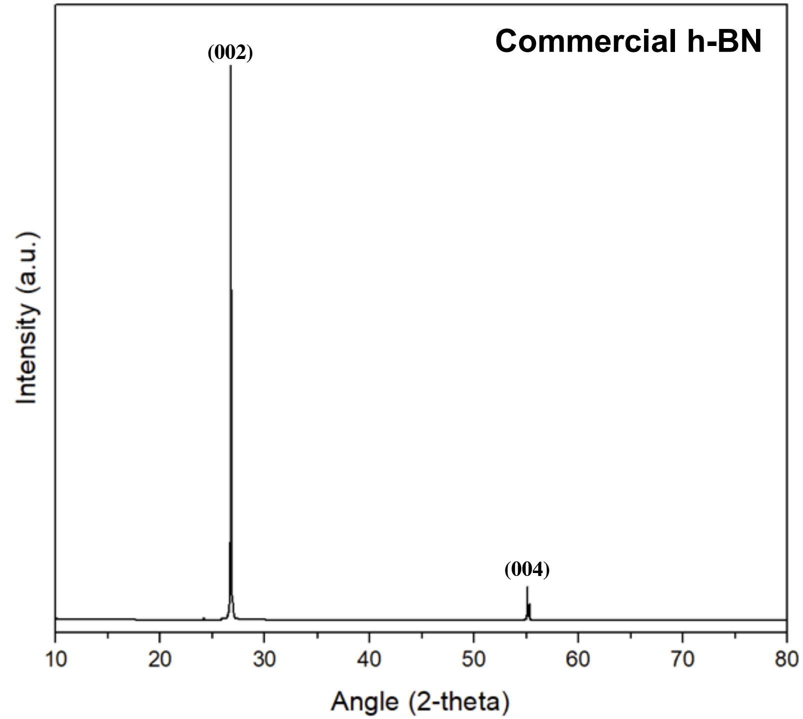
- ***Heat-treatment process #1 produced BN!***
- FTIR shows characteristic BN stretching and bending peaks at 1356 cm^{-1} and 775 cm^{-1}
- There are other peaks between 1700-2400 cm^{-1} that can be related to C

- Second heat-treatment at 500 °C in air:
  - possible amorphous carbon impurities
- The characteristic peaks of BN are still showing after heat treatment
- Possible carbon peaks are also showing in spectra



XRD Analysis



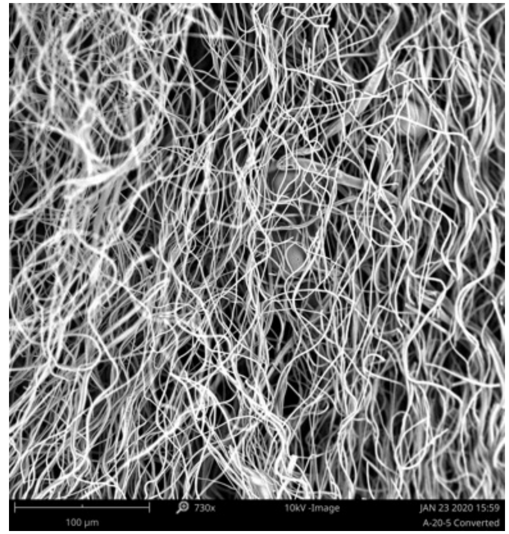


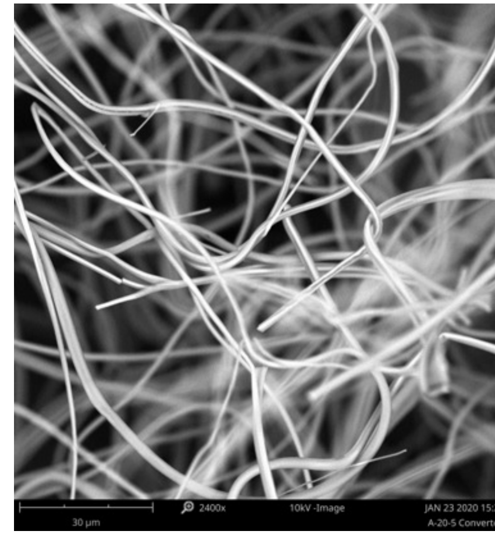
- ***XRD shows the crystalline BN!***
  - Peaks at 26° and 55°
- Subsequent heat-treatment at 500 °C in air disappeared the BN peaks of 26° and 55°
- Crystalline structure is not very stable yet, different heat-treatment is needed

Fiber – Heat Treatment Process #2

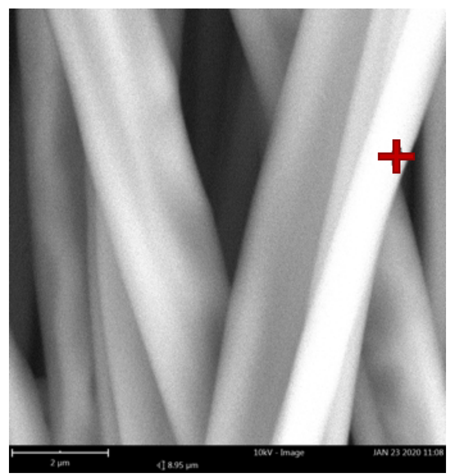
Fiber - Heat Treatment Process #3

SEM Analysis





- Black in color after HT
- Fibers are flexible
- SEM shows long fibers
- EDS shows more even N/B ratio

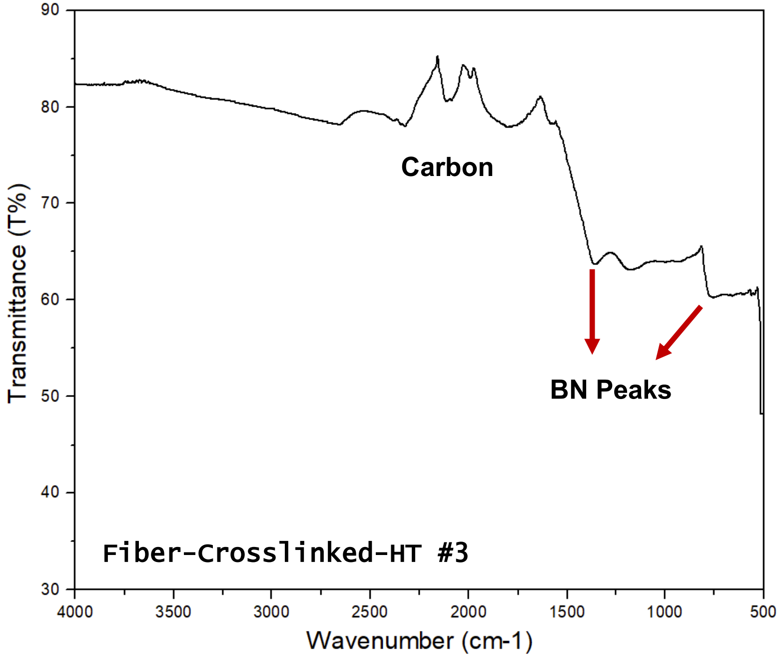


EDS Analysis

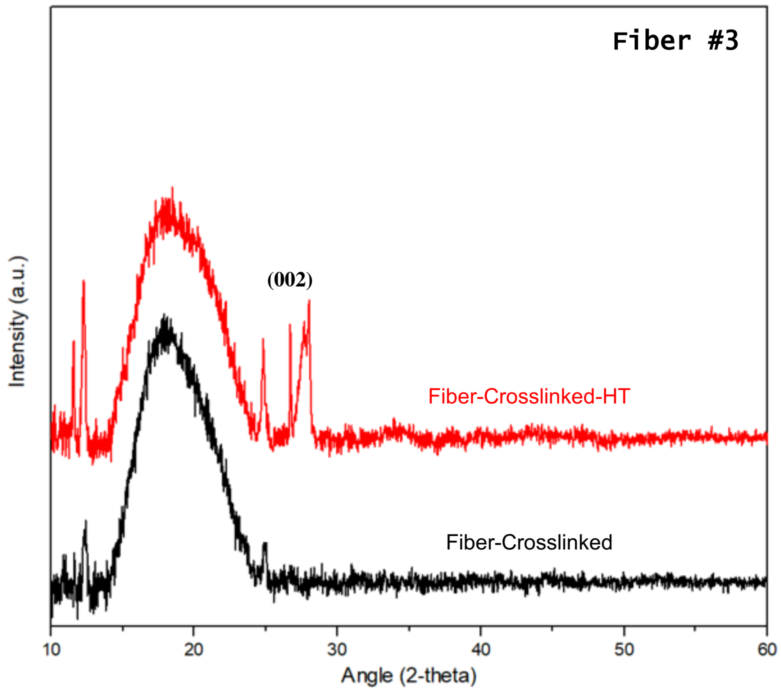
Element Symbol	Atomic Conc.	Weight Conc.
O	56.38	62.50
N	16.81	16.31
C	13.25	11.03
B	13.56	10.16

Fiber - Heat Treatment Process #3

FTIR Analysis



XRD Analysis



- FTIR shows BN stretching and bending vibration peaks, but also carbon peaks
- XRD of the heat-treated fiber shows just a small (002) BN peak at 26°
- The other peaks in XRD are related to two species of carbon
- These fibers need further conversion
- More HT are on the work

What's Next

- Finishing analyzing new fibers processed with new heat-treatments
- Using other fillers that contain boron
- Heat treatment optimization
- Engineer the fiber collection for longer fibers

Conclusions

- Successful preparation of fibers containing PVP and boron oxide with Forcespinning Machine
- Crosslinking fibers with UV lamps
- Successful conversion of pre-formed polymer fibers to crystalline boron nitride with 2 of the heat treatments
- Precursors, solutions and fibers shaped, crosslinked and heat-treated were characterized on SEM, EDS, XRD, and FTIR.
- Heat-treatments still need to be improved to increase the yield of crystalline BN fibers
- Easy, fast, and high yield process with minimum chemical wastes was developed

Acknowledgements

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Thank You!