Engineered Hexagonal Boron Nitride:Titanium Dioxide Composites for High Voltage Insulation

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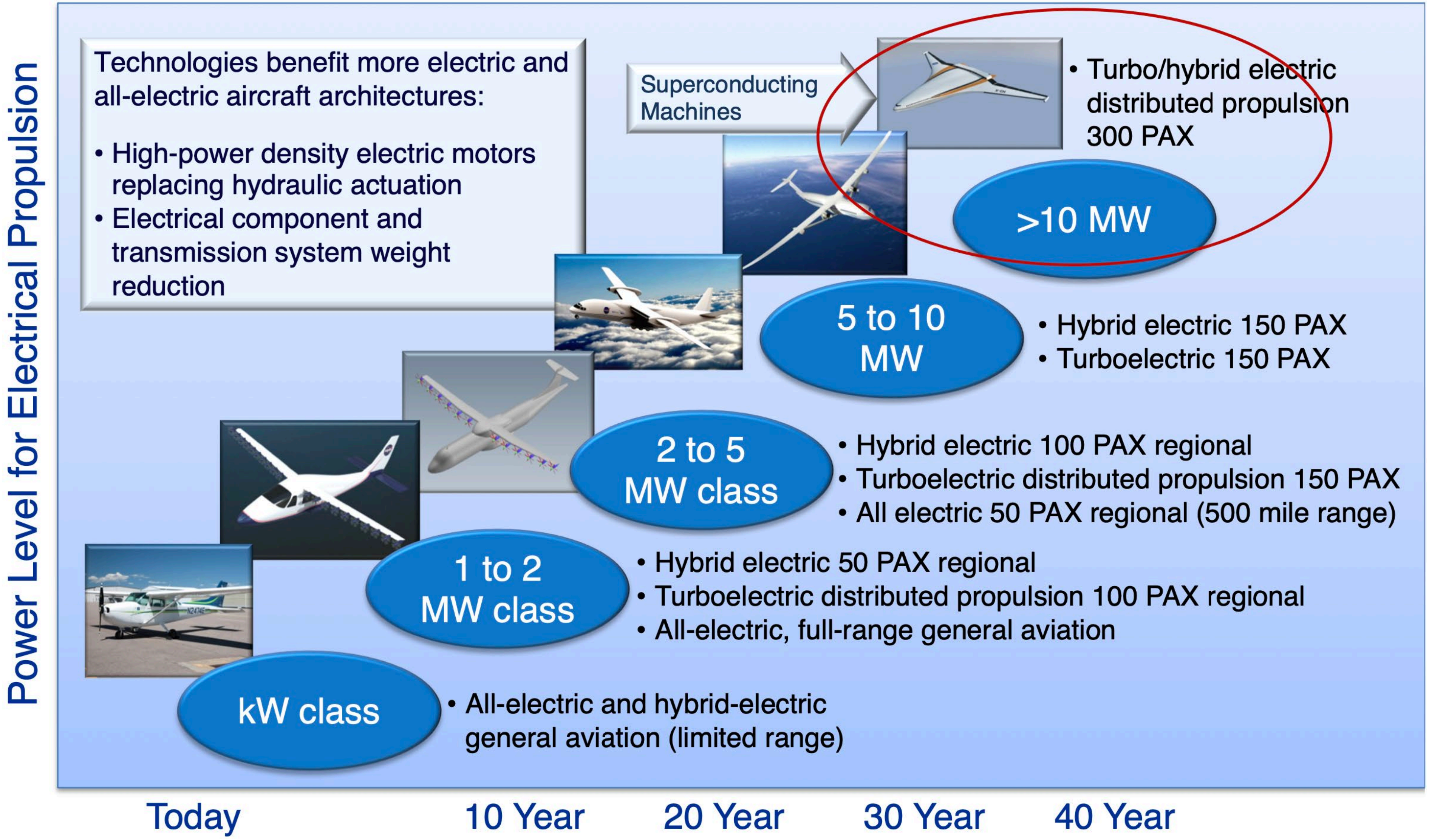
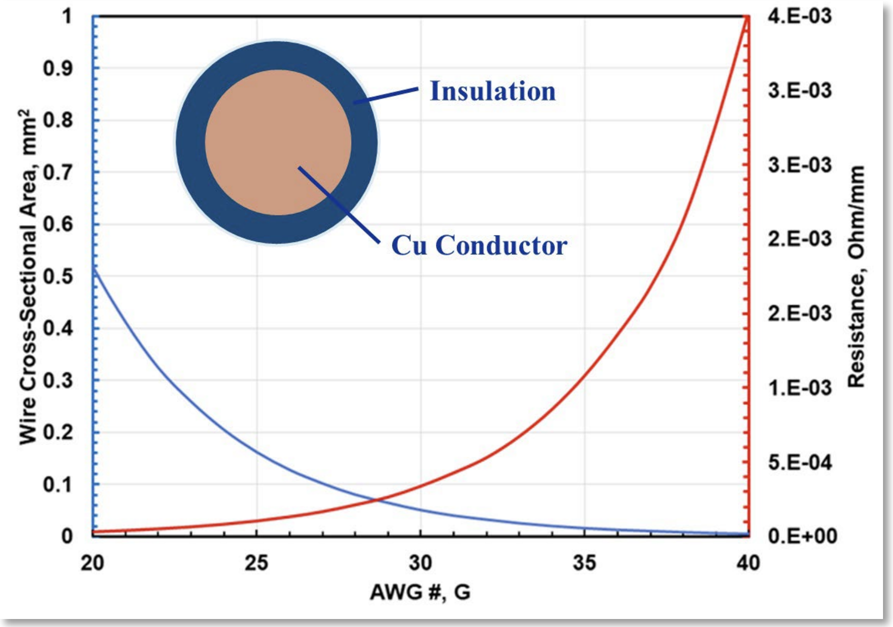
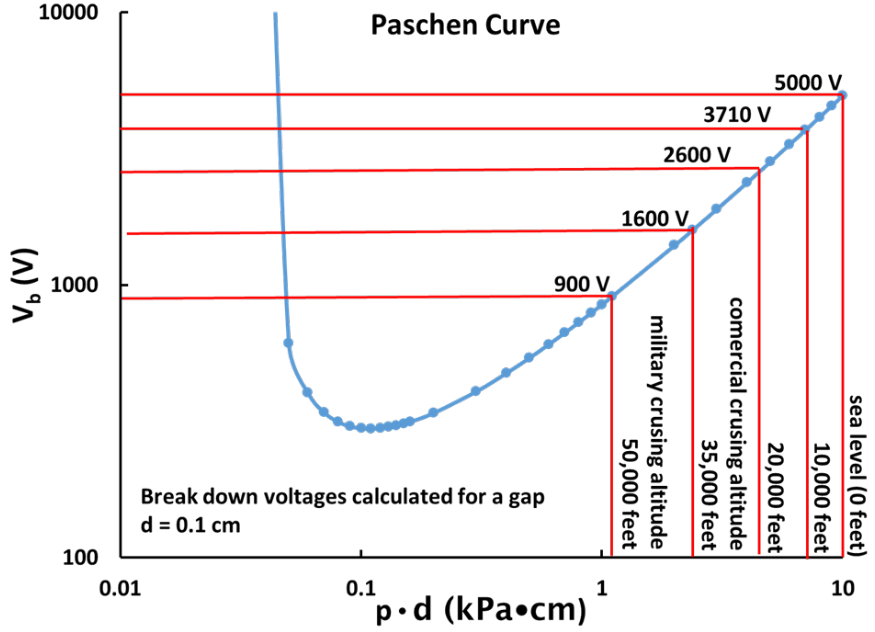
High Power Transformation of Aerospace



Limits of Power Transmission in Aerospace



A. Amasour, D. Sacksteder, A. Goretski, M. Lizcano, NASA/TM-20205008006

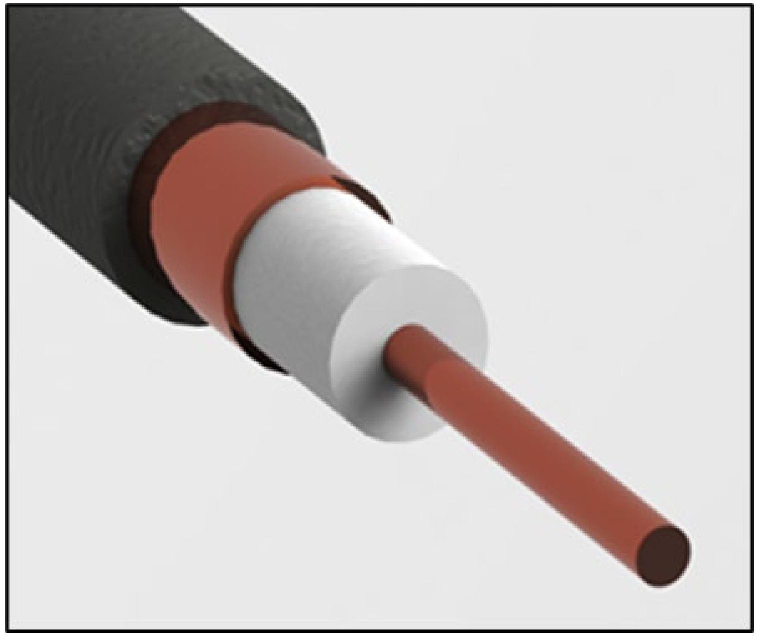


Credit: Woodworth, A.

- High current requires thick and heavy copper cables, which have high resistance
- High voltage requires thick commercial insulation
- Temperature tolerance is and issue in the state-of-the-art insulation

Insulation Material Targets

- Enhanced Thermal Conductivity
- Resistance to partial discharge
- Functional Operation Temperature > 200 ^{o}C
- Temperature Tolerant > 260 ^{o}C
- Retains Dielectric strength of SoA materials
- Decrease diameter and weight of insulation
- Retain high strength of cable system

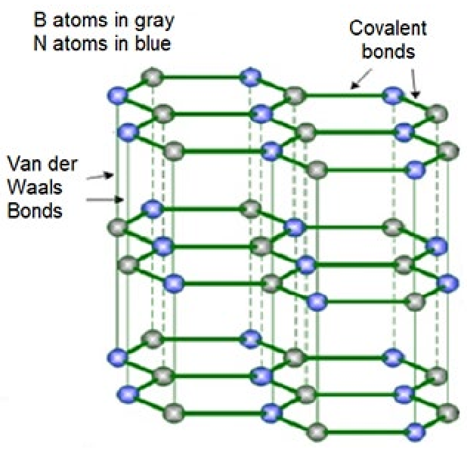


Hexagonal Boron Nitride as an Insulation Material

- NASA Glenn Research Center (GRC) has spent 20 years specializing in boron nitride-based research.
- Applied widely in many applications commercial and industrial.
  - Aerospace and space components
  - Radiation Shielding
  - Energy Storage
  - Lubricants
  - Thermoelectrics
  - Cosmetics
  - Paints
  - Insulation materials
  - Microelectronics

<u>Properties</u>

- Insulation
  - Wide bandgap above 6 eV
- Mechanical Strength
  - Similar to graphite, hard and shearing.
- Thermal Conductivity
  - hBN ~ 100 W/m·K in plane, ~ 10-30 W/m·K through plane
  - Strong thermal shock characteristics
- Stability
  - Hydrophobic
  - Chemically stable
  - Stable above 1000 ^{o}C in air
- Lightweight
  - 2.1 g/cm^3



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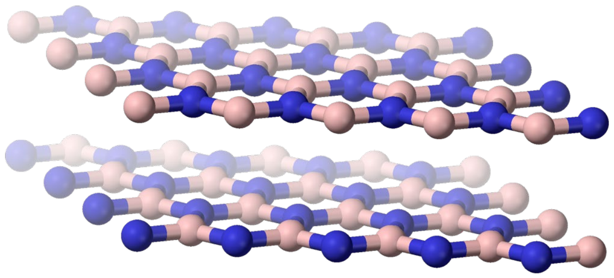


O. Shinpei, S. Fukushima, M. Shimatani, *Materials* **2023**, *16*(5), 2005

Engineered Hexagonal Boron Nitride

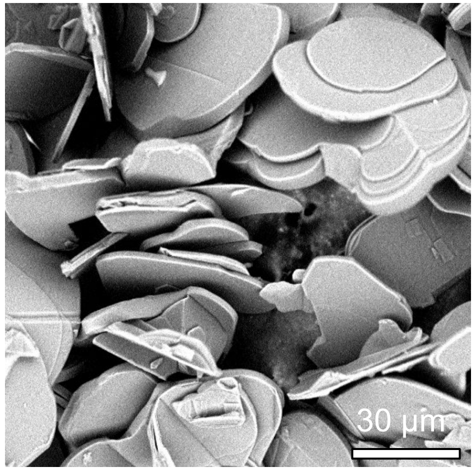
Material Control

- Shape Control
  - Platelet structure, some rods and amorphous spheres
  - Control of aspect ratio/number of layers is key
  - Can be sintered and made into larger structures. (pyrolytic)
- Size Control
  - 100s nm to 100 µm wide as platelets
  - 1 atom thick to 10s of µm thick

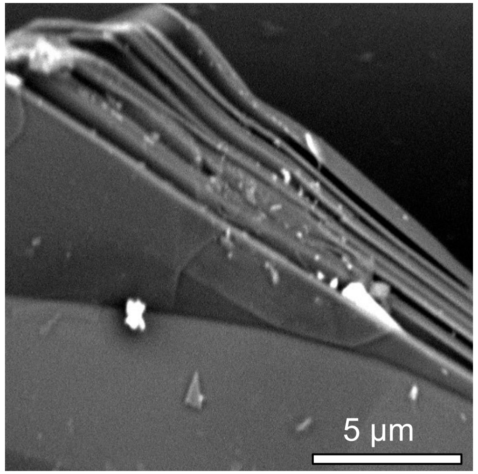
Multifunctionality

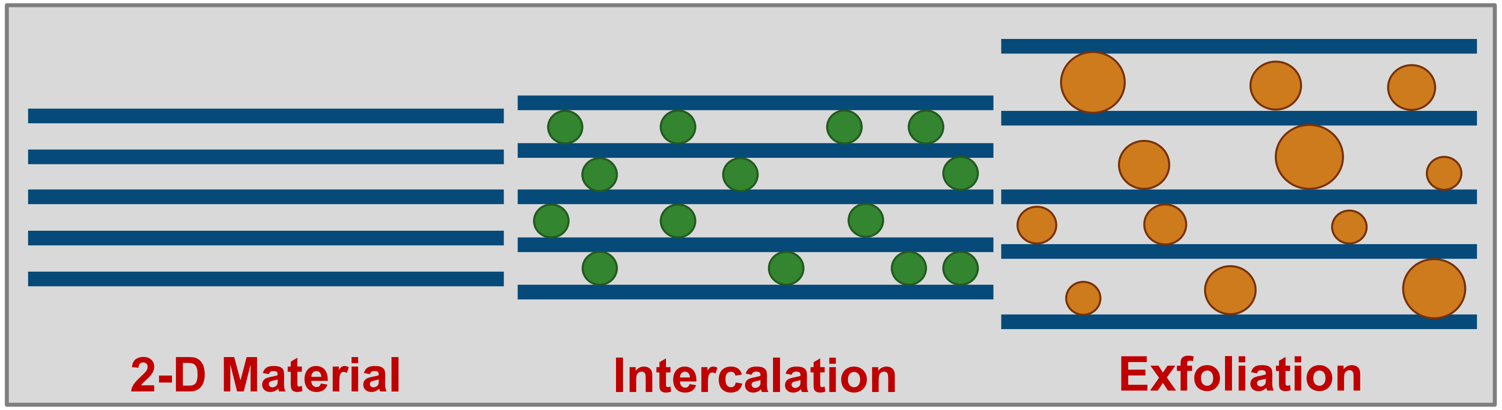
- Edge and surface functionalization
  - Oxidation, Silanation, Hydrogenation
- Intercalation
  - Metal Halides, Salts, Solvents/polymers
- Exfoliation
  - Thinner layers
  - Expanded layers

Momentive PT 110 hBN



PT 110 hBN Exfoliated with TiO_2





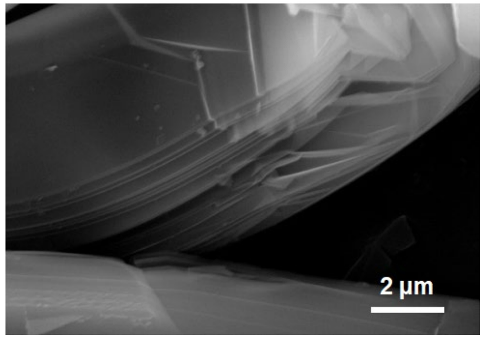
Three Types of Intercalation

A Iron Oxide
- Separating Layers
- Fully removeable

B Alumina
- Ceramic Compatibility
- Improved Thermal Shock

C Titania
- Crystal Tunability
- Dielectric Strength
- Composite Matrix

Custom Kettle Furnace for Intercalation



Single $FeCl_3$ Exfoliation hBN
Credit: Nguyen, B.

hBN + $FeCl_3$	Activation Agent → Heat, N_2	Intercalated hBN	(1)
Intercalated hBN	High Heat, air →	Crude Exfoliated hBN	(2)
Crude Exfoliated hBN	Rinse with Water →	Exfoliated hBN + Fe_2O_3	(3)
Exfoliated hBN + Fe_2O_3	Repeats of steps 1, 2, 3 →	Multi Exfoliated hBN + Fe_2O_3	(4)
Multi Exfoliated hBN + Fe_2O_3	Rinse with HCl →	Clean Exfoliated hBN	(5)

Titania Properties

- Titanium Dioxide
  - Sintering temperature ~1500 C
  - Hot pressable and formable
  - Lower Thermal Conductivity, but close (~7-10 W/m·K)
  - Capacitive

Two major crystal forms, anatase and rutile

- Anatase
  - Lower density (3.78)
  - Lower dielectric constant ~14/110[1]
  - Interesting semiconduction/ion conductivity properties, bandgap of 3.4 eV[2]
  - Longer e-hole pair lifetime can promote chemistry (better photocatalyst)
- Rutile
  - Denser form (4.23) higher than alumina
  - More stable form
  - Higher dielectric constant ~89/170[1], bandgap of 3.0 eV[2]
  - Bright white, used in paints

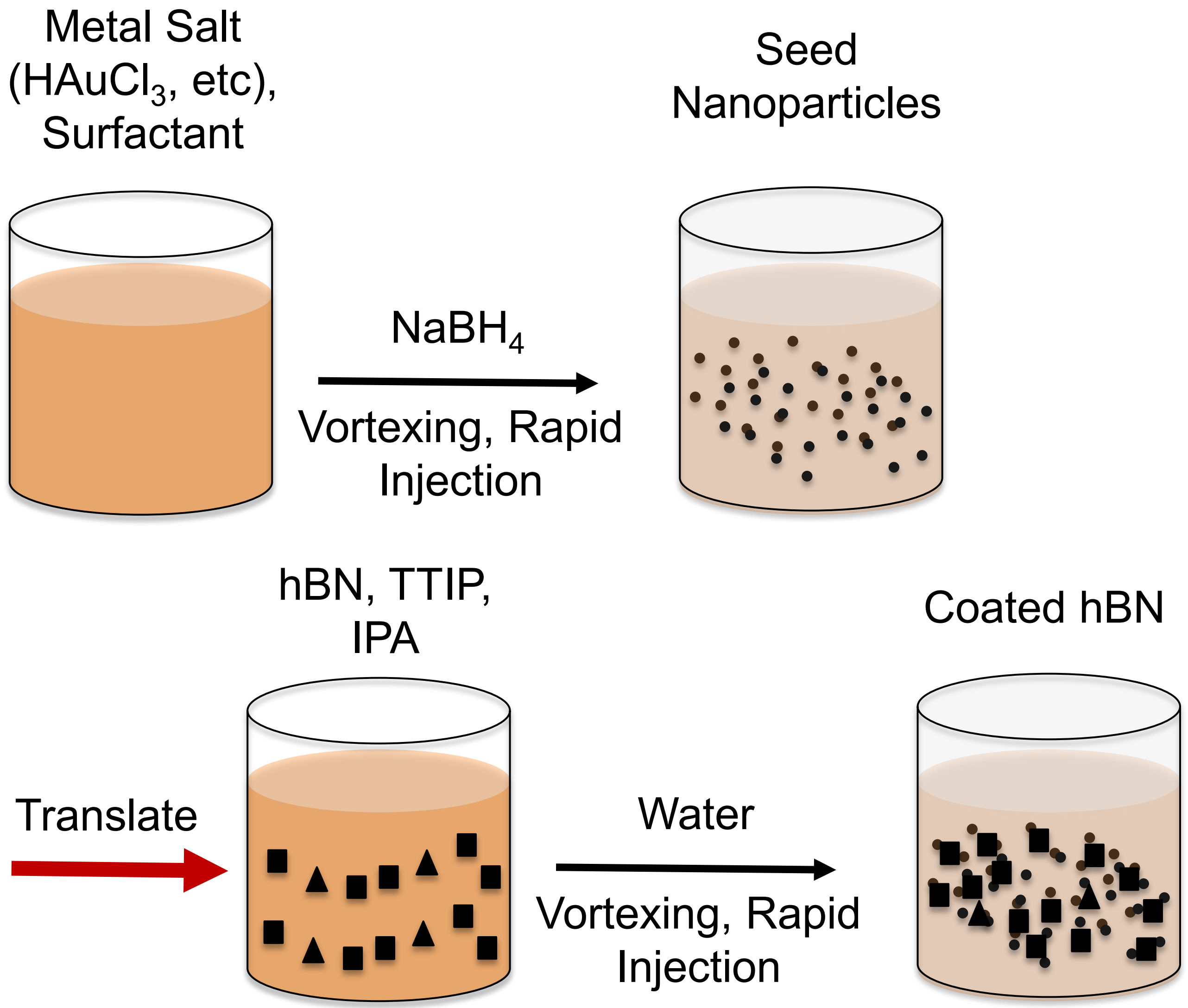
Preferentially makes small nanoparticles upon oxidation

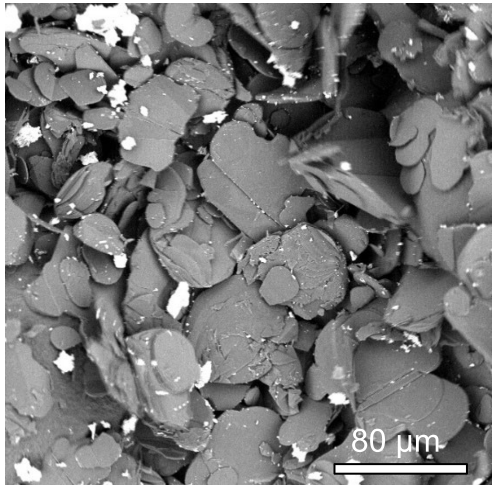
  - Allows for targeted final product control

[1]Matweb.com/Titanium Dioxide

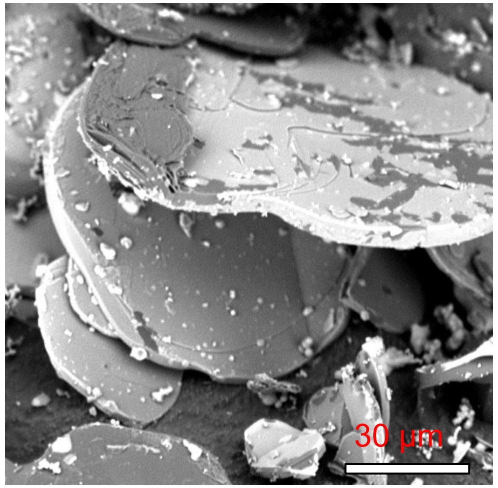
[2]Varadwaj, P. Dinh, V, Morikawa, Y, Asahi, R. ACSOmega-**2023**-22003

Titania Coating Method



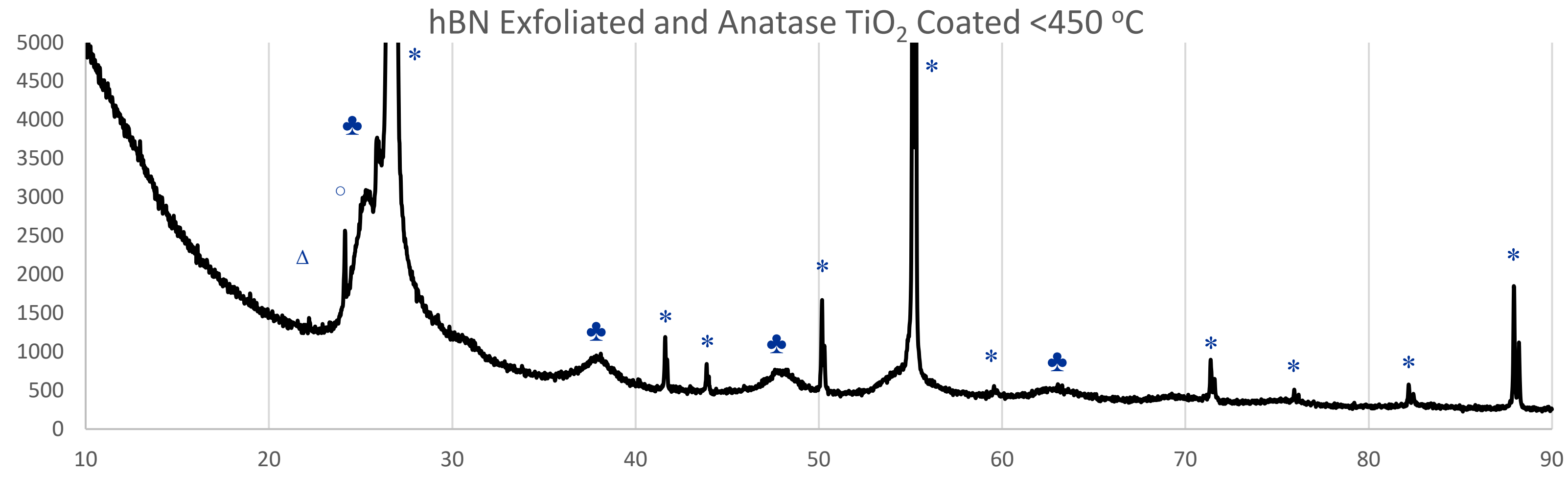


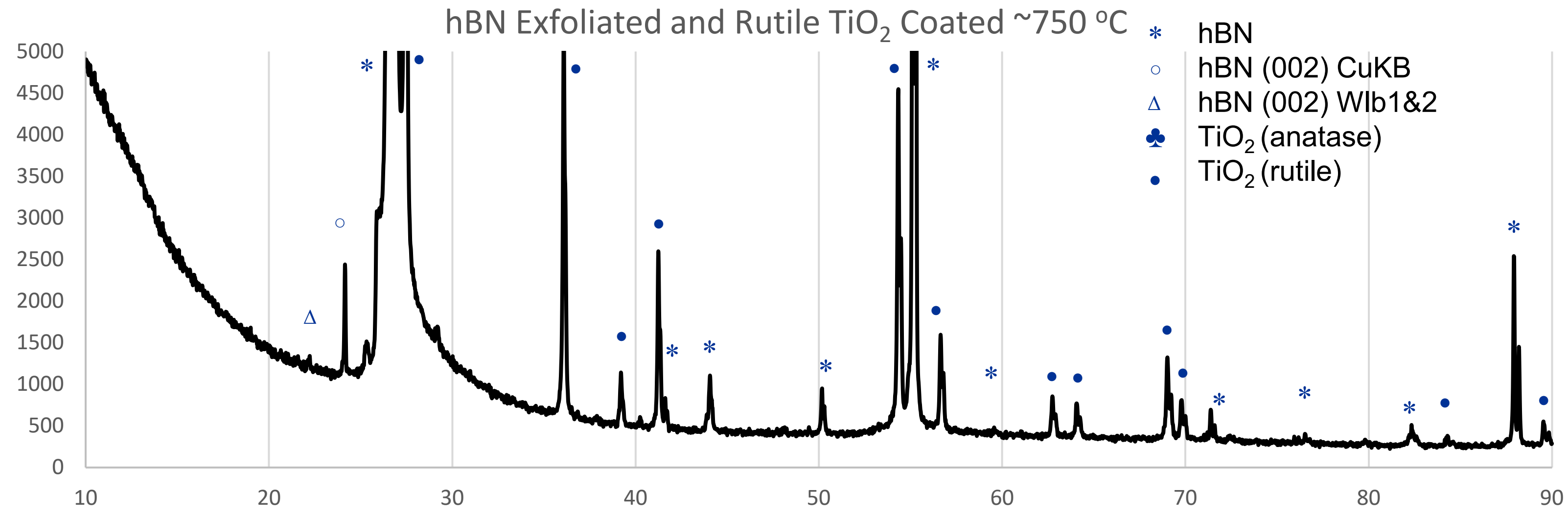
Sample Coated by
oxidation/metal addition

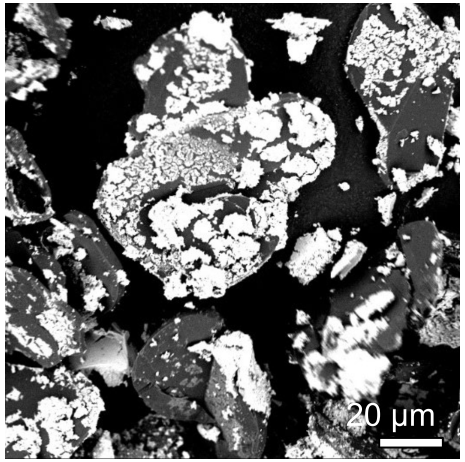


Sample Coated by diluted
TTIP/Water injection method

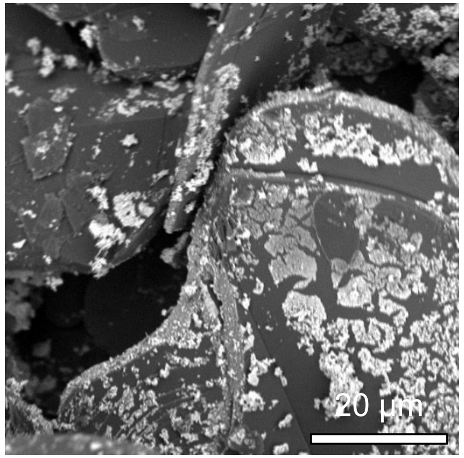
The Crystal Structure is Easily Tunable







hBN Coated with Anatase TiO_2

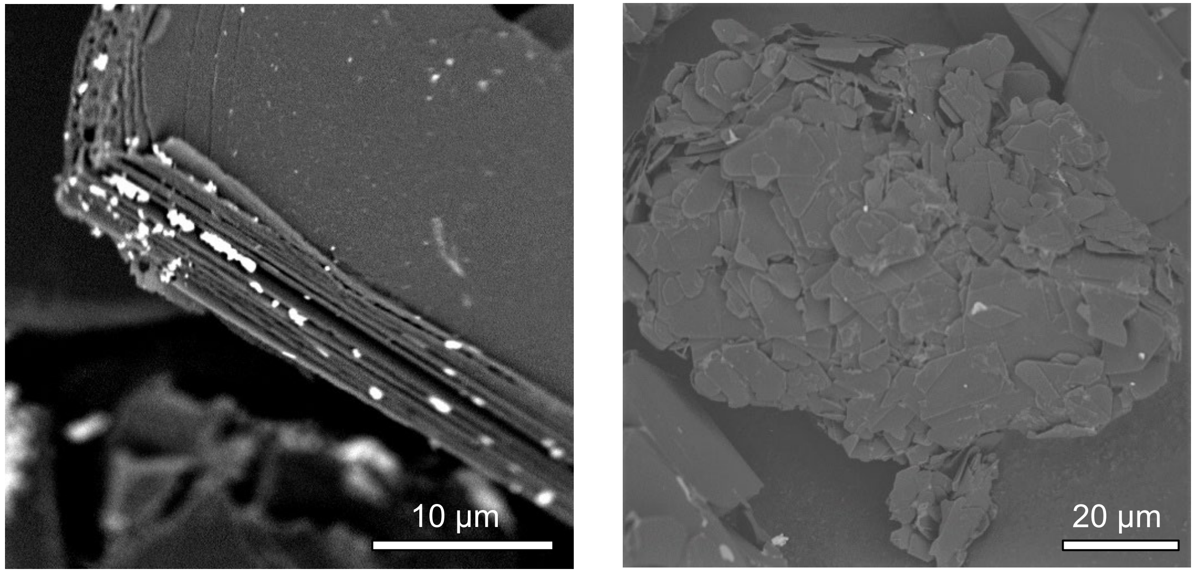


hBN Coated with Rutile TiO_2

Titania Intercalation and Exfoliation

<u>Method optimization</u>

- $TiCl_3$ is hard to work with
  - Rapidly reacts with wet air
  - Low boiling point/sublimation
  - Liquid at room temperature
  - Likely to form oxide before removal or hydration
- Titanium tetraisopropoxide (TTIP)
  - Reacts slower with air
  - High boiling point (close to $AlCl_3$ and $FeCl_3$)
  - Can be diluted with isopropanol for optimal coating
  - Larger molecule
  - Liquid at room temperature



Momentive PT110 hBN (40-100 μm platelets) Intercalated and Exfoliated with TiO_2

hBN + TTIP	**Activation Agent** → **Heat, N_2**	**Intercalated hBN**	(1)
Intercalated hBN	**High Heat, air** → **Lower Temp**	**Crude Exfoliated hBN**	(2)
Crude Exfoliated hBN	**Rinse with Water** →	**Exfoliated hBN + TiO_2**	(3)

Insulation Composite Testing

Methods of Insulation Composite Testing

- Twin screw extrusion of target high temp polymer
  - Requires large amounts of powder composite >10 grams per run depending on loading
  - Trial an error required to achieve goal loadings
  - The best method of mixing, can make tapes and wires but hard to start with
- Small scale compounding and injection molding
  - Limited to thermoplastic polymers
  - Requires expensive equipment to use high temperature polymers
  - High throughput
- Tape Casting
  - Works at very small sample sizes
  - Can use high temp stable thermosets without expensive equipment
  - Settling of composite material may be an issue
  - High throughput

High Temp Tape Extrusion

PDMS Tape Cast with hBN

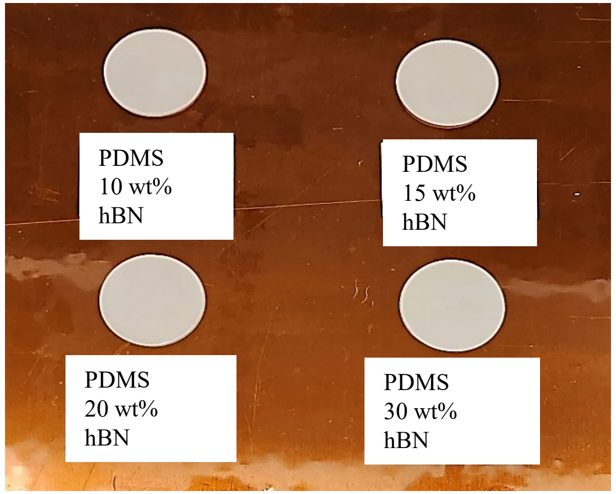
PDMS Tape Cast Composite Testing

Polydimethylsiloxane (PDMS) chosen as tape cast polymer matrix

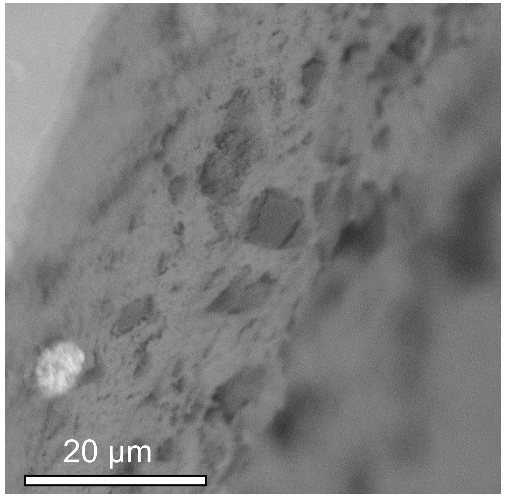
- Silgard 184 Elastomer (PDMS type)
  - Good Dielectric properties
  - Can withstand temps up to 200 C
  - Room temperature cure
  - Accelerated cure at higher temperatures
  - Low enough viscosity to mix well with composites
- Processing conditions,
  - Mix elastomer and hBN powders by shear mixing (Thinky mixer)
  - Degas
  - Pour and cast into a tape
  - Allow to cure at elevated temp over night

PDMS Tape Cast with hBN

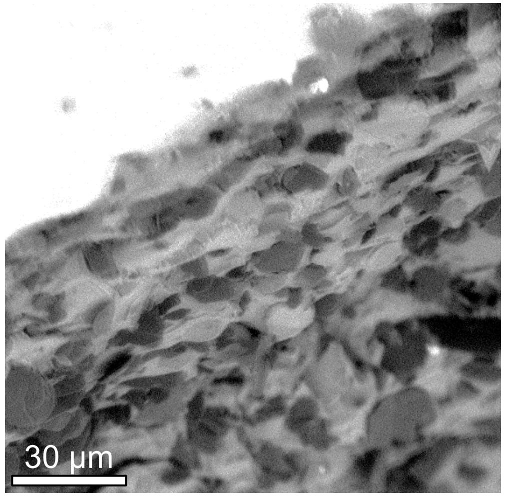
Samples Cut and Stacked for Thermal Conductivity/Breakdown



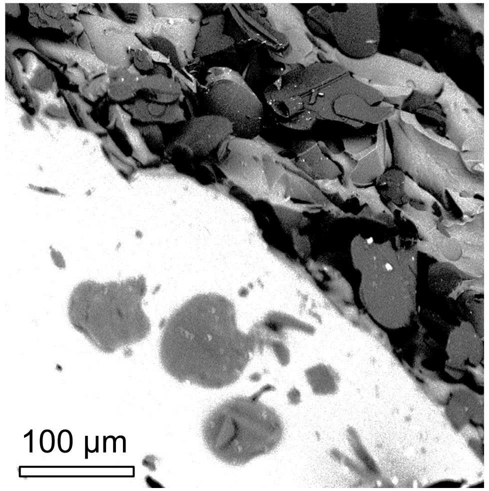
PDMS Tape Cast Composite Testing



30% AC6097 (500 nm platelets)
Small



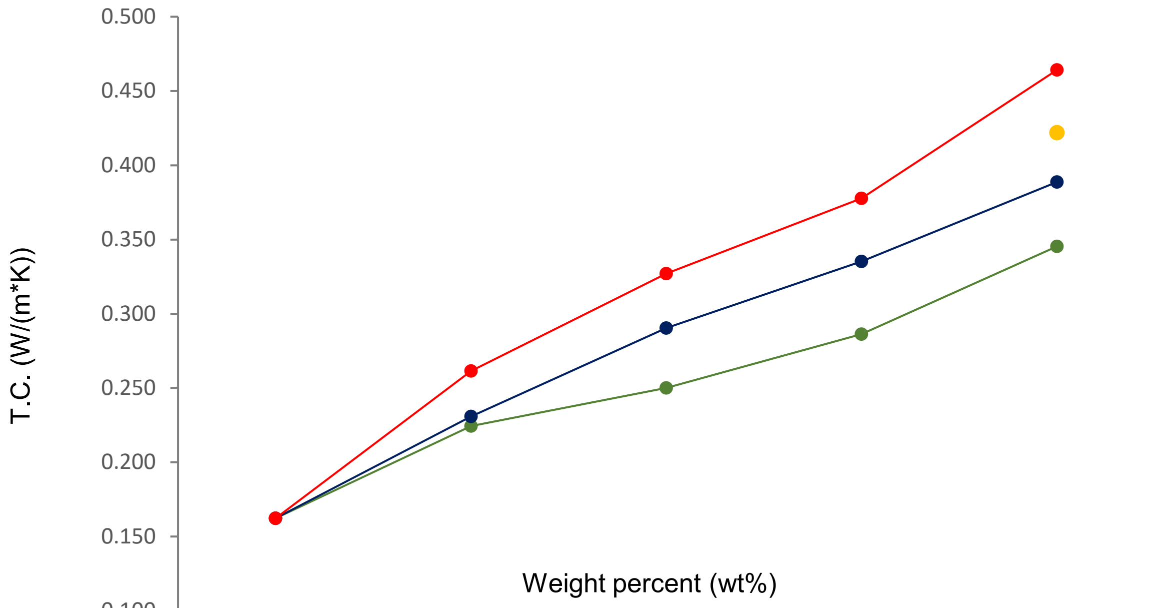
30% PTC120 (5-10 µm platelets)
Medium



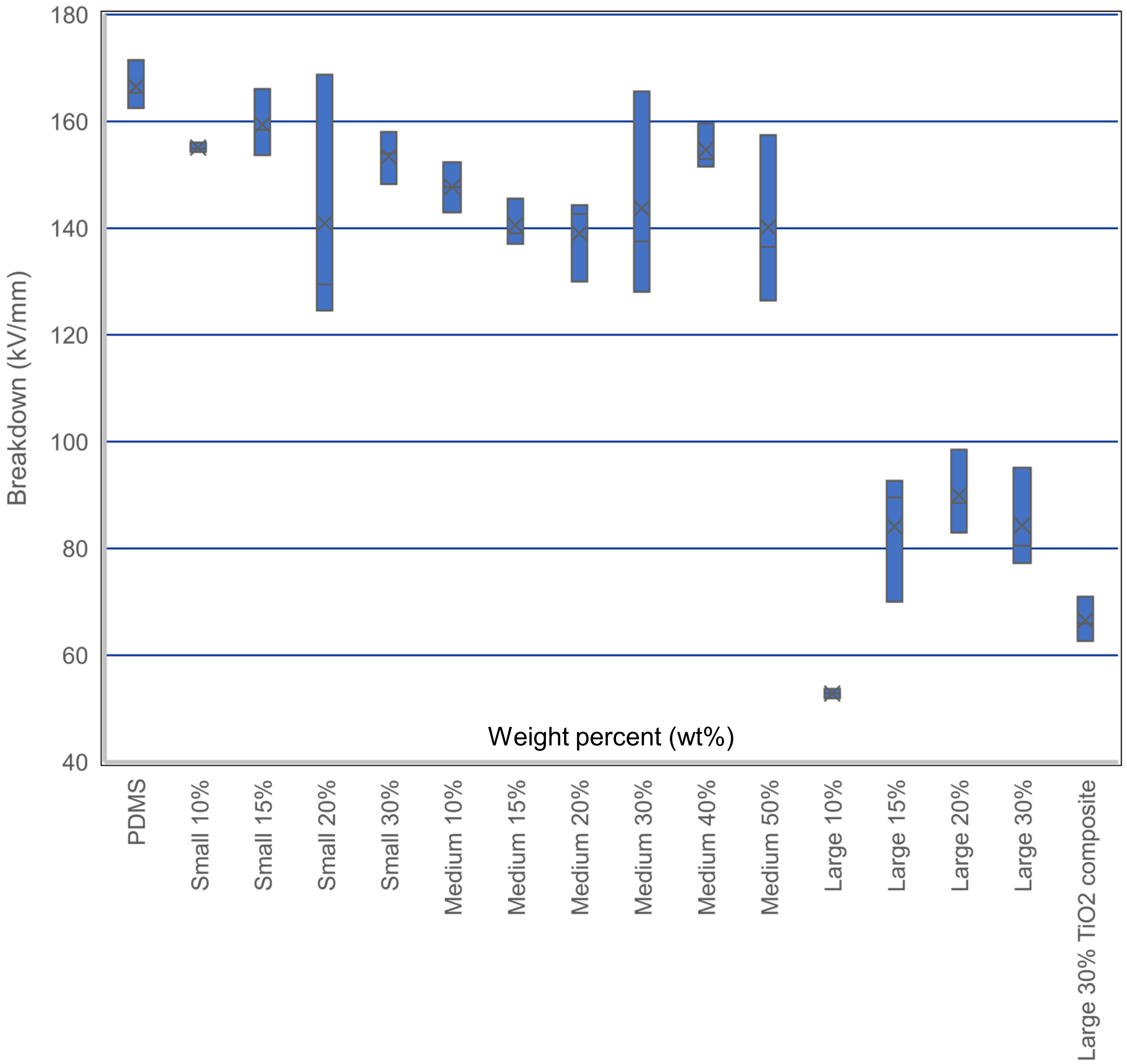
30% PT110 (40-100 µm platelets)
Large

- Cross Section Images show particle sizes inside tape
- Larger particles less well mixed and likely include air voids
- Not many particles on the surface

PDMS Tape Casting Optimization



	0%	10%	15%	20%	30%
Small	0.162	0.224	0.250	0.286	0.345
Medium	0.162	0.231	0.290	0.335	0.389
Large	0.162	0.261	0.327	0.378	0.464
Large:TiO2					0.422



- Thermal conductivity measured by a modified transient plane source (C-Therm, surface dissipation method)
- Breakdown done in DC 2 kV/s ramp
- Small (500 nm), Medium (5-10 μm), Large (40-100 μm)

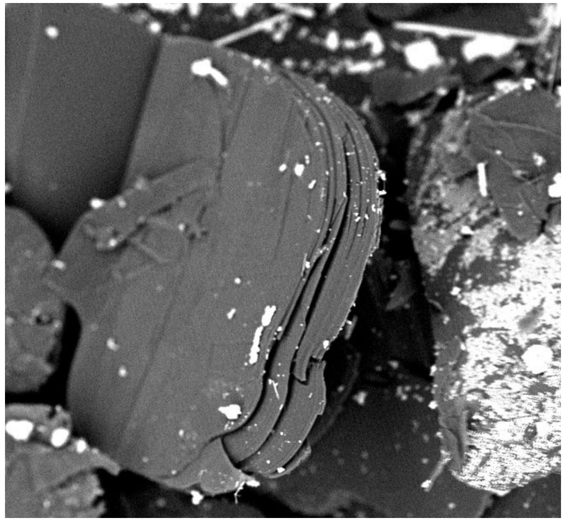
Summary of Achievements and Future Work

Accomplishments

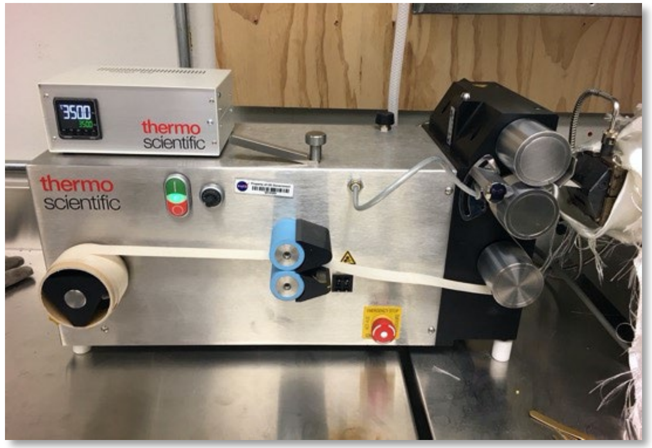
- hBN platelets coated with titania, and both crystal patterns formed by heat treatment
- hBN exfoliated with titanium isopropoxide and separated particles formed with titanium nanoparticles interspersed
- Test platform composite properties chosen as a PDMS matrix and tested across multiple hBN types

Future Work

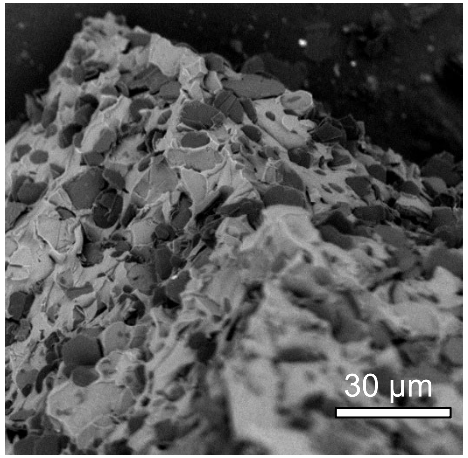
- Process improvement for large particle mixing
- Confirmation of internal structure of exfoliated hBN
- Dielectric properties testing of composites
- Multilayer stacking
- Large scale tape extrusion
- High yield exfoliation process optimization



Exfoliated PT110 with Titania



Tape extrusion from twin screw extruder



hBN/PDMS cross section

Thank You!

- Transformational Tools and Technologies (TTT) Project
- GRC Boron Nitride Team
  - Dr. Sean Patrick McDarby
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  - Dr. Tiffany Williams
  - Dr. Baochau Nguyen
  - Dr. Witold Fuchs
  - Dr. Ching-cheh Hung (retired)
  - Ms. Alanys V, Luna
  - Ms. Janet Hurst (retired)
- Materials Chemistry and Physics Branch
- Materials and Structures Division