Characterization of Gamma Irradiated Aerosol Jet Printed Polyimide/h-BN Films

Student: Lucas Clark

Student Email: clark.567@wright.edu

Faculty: Ahsan Mian

Faculty Email: ahsan.mian@wright.edu

AFRL Sponsor: Carrie Bartsch

AFRL Directorate: AFRL/RY

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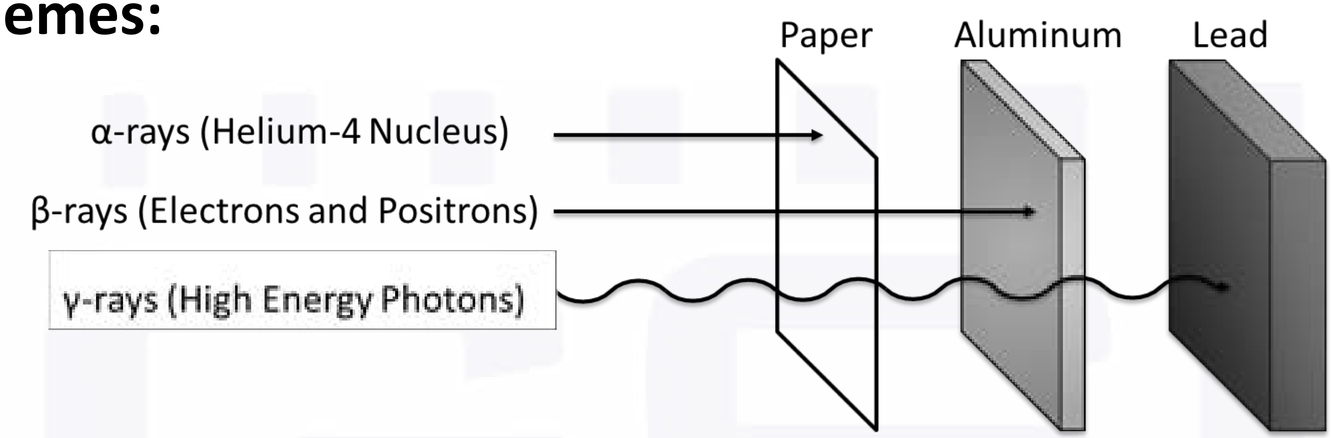
Motivation

Conventional Radiation Protection Schemes:

- Redundant Systems and Components
- Error Tolerant Coding
- External Shielding
  - ❖ Adds complexity, cost, and weight



Spot Shielding:

- Selectively shielding susceptible electronic components
  - ❖ Aerosol Jet Printing and Other Additive Manufacturing Processes
- Advanced nanocomposite materials utilizing low density host with high density guest nanoparticles
  - ❖ Higher density materials (i.e. lead) have higher gamma attenuation
  - ❖ Lower density materials could reduce overall shield weight

Aerosol Jet Printing

Direct Ink Write Technology

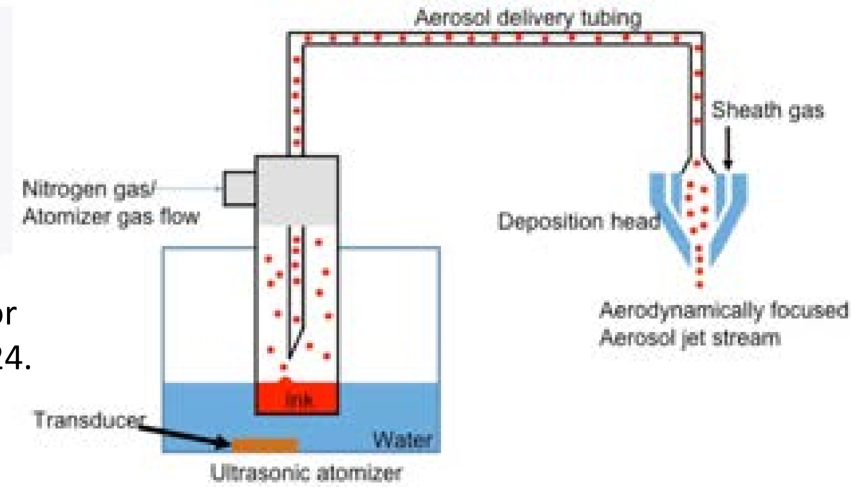
- ❖ Create Microscale 2D or 3D Functional Structures on Flat or Conformal Surfaces
- ❖ Features With Dimensions As Small As 5 µm And Up To Several mm

Materials Include

- Colloidal Inks
- Nanoparticle-filled Inks
- Diluted Thick-film Pastes
- Thermosetting and UV Curable Polymer Solutions

Ultrasonic Atomization: Ink viscosities up to 7 cP

Pneumatic Atomization: Ink viscosities up To 1000 cP



Top Image: "Ceradrop Is Exclusive Distributor of OPTOMEC in France." CERADROP Is Exclusive Distributor of OPTOMEC in France, www.ceradrop.com/en/distributeur-integrateur-optomec/. Accessed 6 Oct. 2024.
Bottom Image: Agarwala, S, et al. "Optimizing aerosol jet printing process of silver ink for printed electronics." IOP Conference Series: Materials Science and Engineering, vol. 191, Apr. 2017, p. 012027, https://doi.org/10.1088/1757-899x/191/1/012027.

Overview

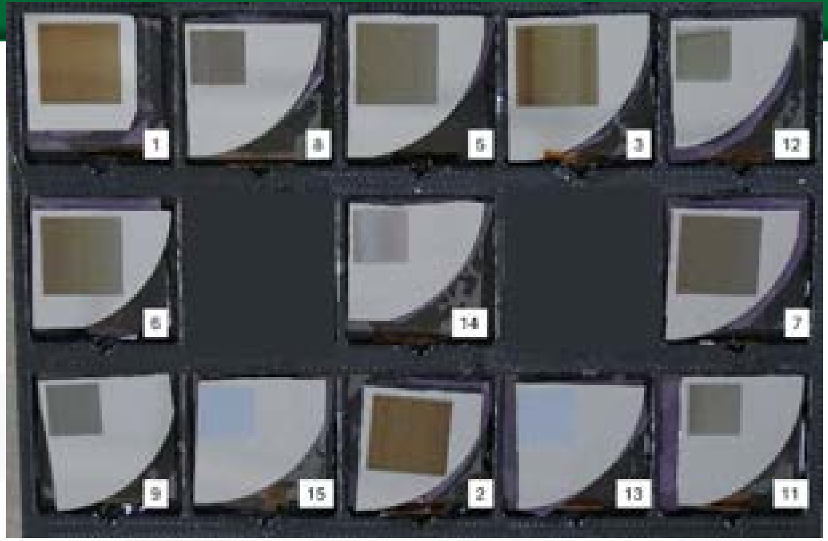
- Developed aerosol jet printable nanocomposite inks containing Polyimide (PI) and hexagonal boron nitride (h-BN)
  - ❖ Ink Concentrations:
    - PI
    - PI-25 wt% h-BN
    - PI-50 wt% h-BN
    - PI-75 wt% h-BN
    - h-BN
- Characterized printed nanocomposite thin films using Fourier-transform infrared spectroscopy (FTIR)
- Exposed thin films to Cobalt-60 source for a total dosage of 80 krad (Si) and recharacterized using FTIR

Materials used for the nanocomposites' formulations:

- ➢ Commercial off the shelf (COTS) Polyimide solution (P84 Type 70 in LTM1) from Evonik Industries
- ➢ Organic solvents used: Cyrene (99%) – Sigma-Aldrich, Cyclohexanone (≥99.0%) – Sigma-Aldrich, and α-Terpineol – Sigma-Aldrich
- ➢ Boron Nitride Powder (98%), 1 µm average particle size – Sigma-Aldrich
- ➢ Ethyl Cellulose (5% in toluene/ethanol) – Sigma-Aldrich
- ➢ Substrate - Undoped double sided polished silicon wafers

Ink Formulations and Printed Samples

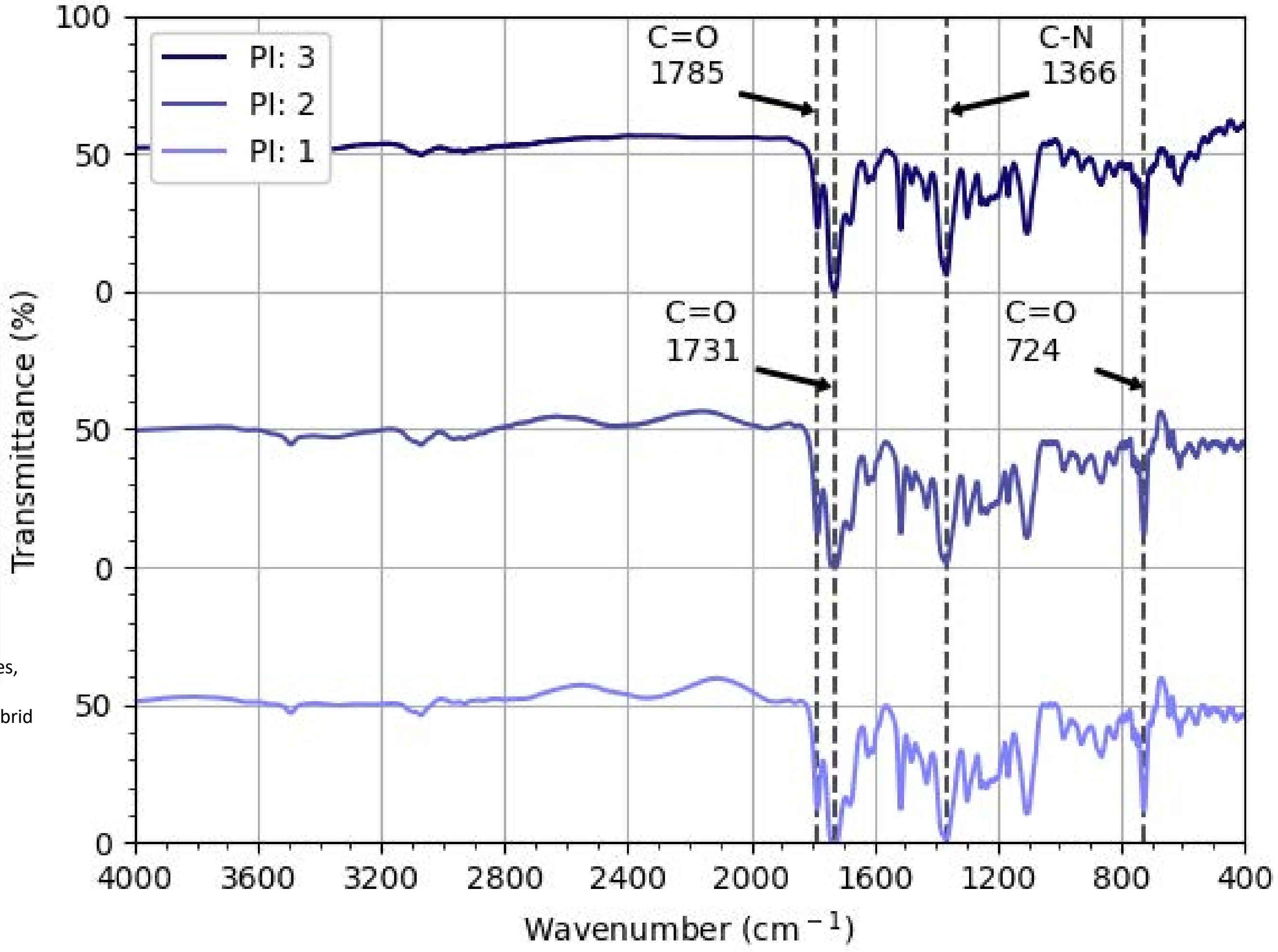
- Polyimide Ink:
  - P84 was diluted with Cyrene
- h-BN/Polyimide Inks:
  - Sonication of h-BN flakes in Cyrene
  - Polyimide combined with h-BN/Cyrene Solution
- h-BN Ink:
  - h-BN and ethyl cellulose were sonicated in isopropyl alcohol
  - Solvent exchanged using salt water and supernatant
  - h-BN pellet was washed with deionized water
  - Deionized water was replaced with isopropyl alcohol
  - Solution was placed in an evaporation dish to dry into flakes
  - h-BN flakes were added to cyclohexanone and α-terpineol solution



Sample Type	Sample ID	Thickness (μm)
PI	1	5.7
PI	2	5.2
PI	3	1.5
PI-25 wt% h-BN	4	1.4
PI-25 wt% h-BN	5	1.5
PI-25 wt% h-BN	6	2.4
PI-50 wt% h-BN	7	2.2
PI-50 wt% h-BN	8	1.0
PI-50 wt% h-BN	9	1.4
PI-75 wt% h-BN	10	2.0
PI-75 wt% h-BN	11	1.8
PI-75 wt% h-BN	12	2.1
h-BN	13	1.9
h-BN	14	6.2
h-BN	15	1.9

FTIR: Polyimide – Before Exposure

- Polyimide Common Peaks:
  - ≈1775 cm^{-1}: Asymmetric Stretching of C=O Bonds
  - ≈ 1720 cm^{-1}: Symmetric Stretching of C=O Bonds
  - ≈ 1370 cm^{-1}: C-N Bond Stretching
  - ≈ 720 cm^{-1}: Bending of C=O



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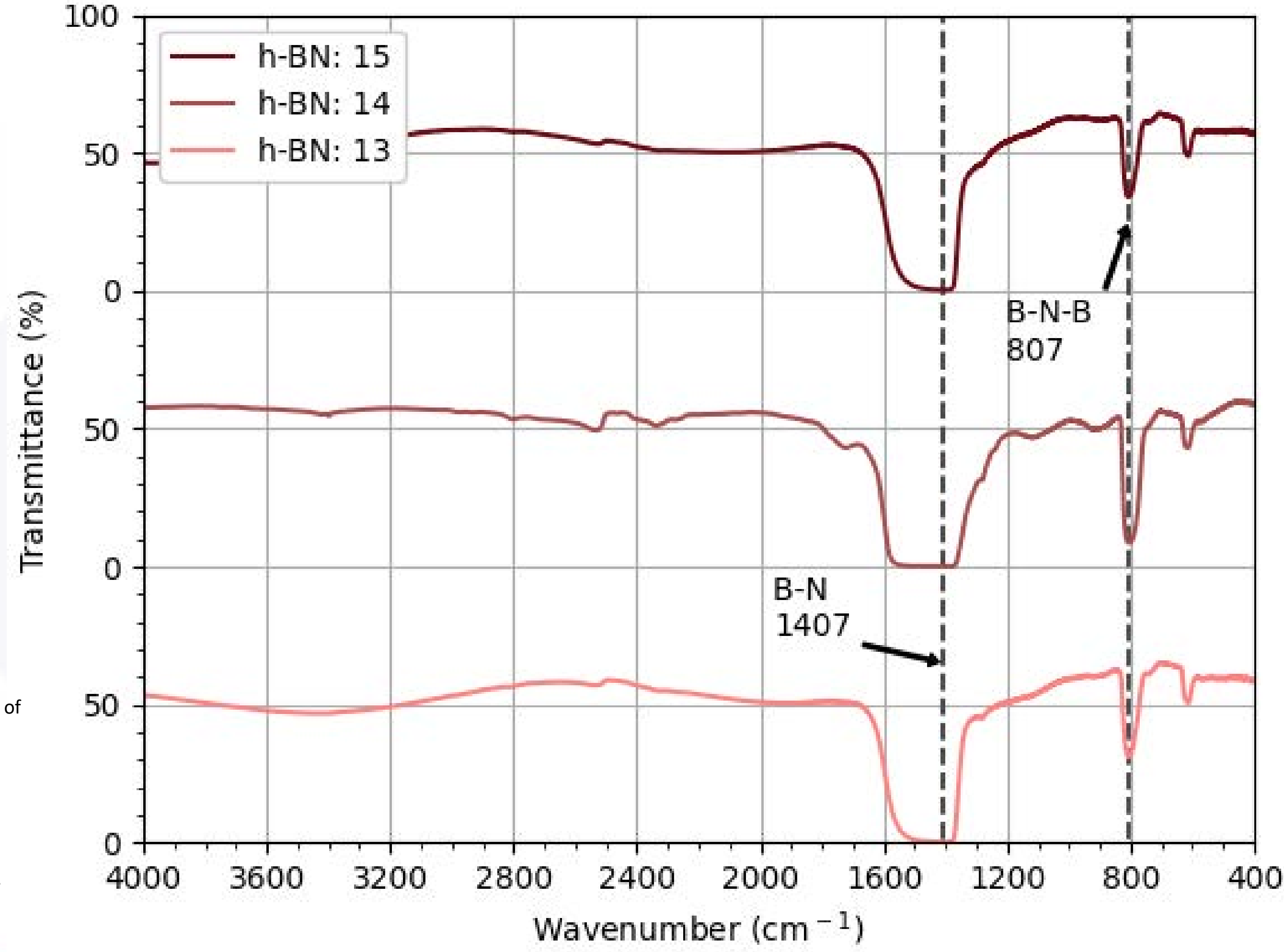
FTIR: h-BN – Before Exposure

- h-BN Common Peaks:
  - ≈1380 cm^{-1}: In-plane stretching vibration of B-N bonds
    - Broad Band ranging from 1300-1500 cm^{-1}
  - ≈ 810 cm^{-1}: Out-of-plane bending vibration of B-N-B bonds



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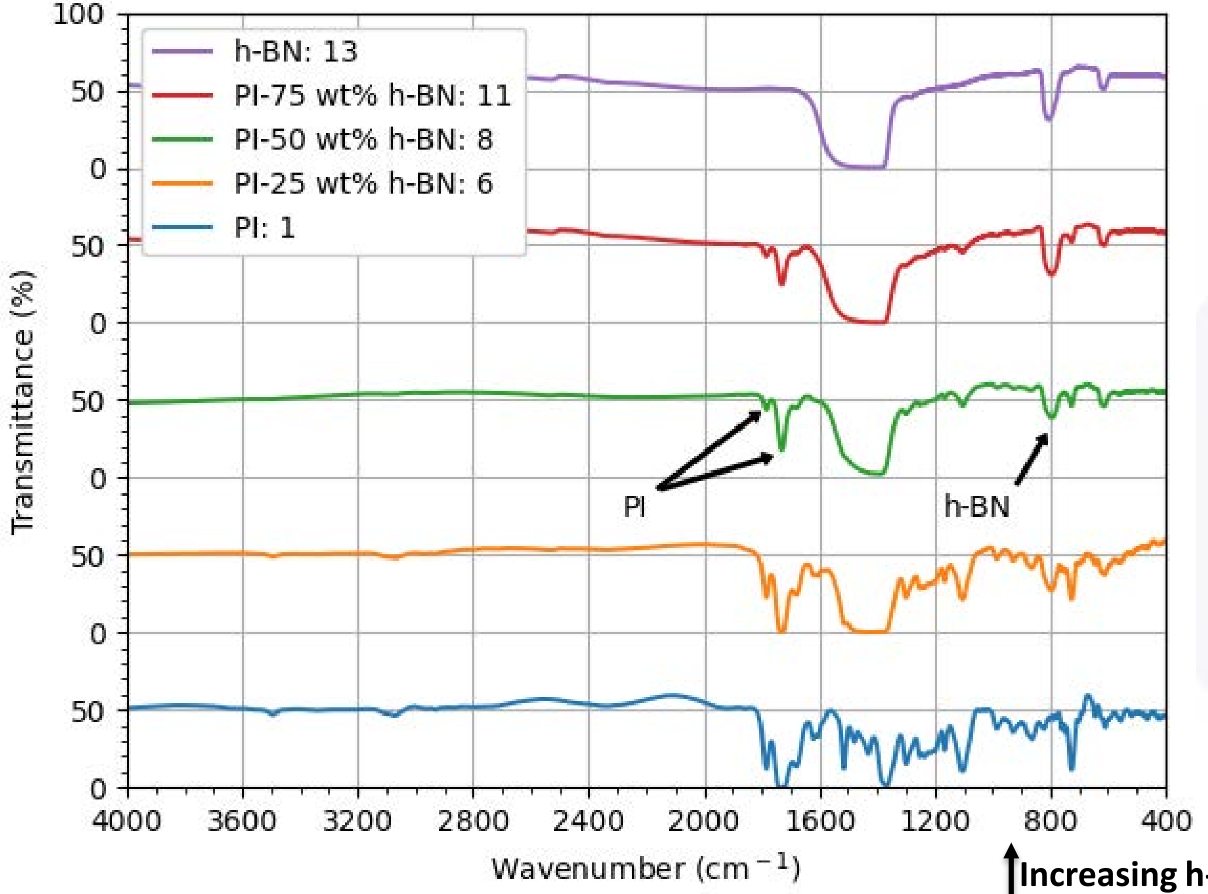
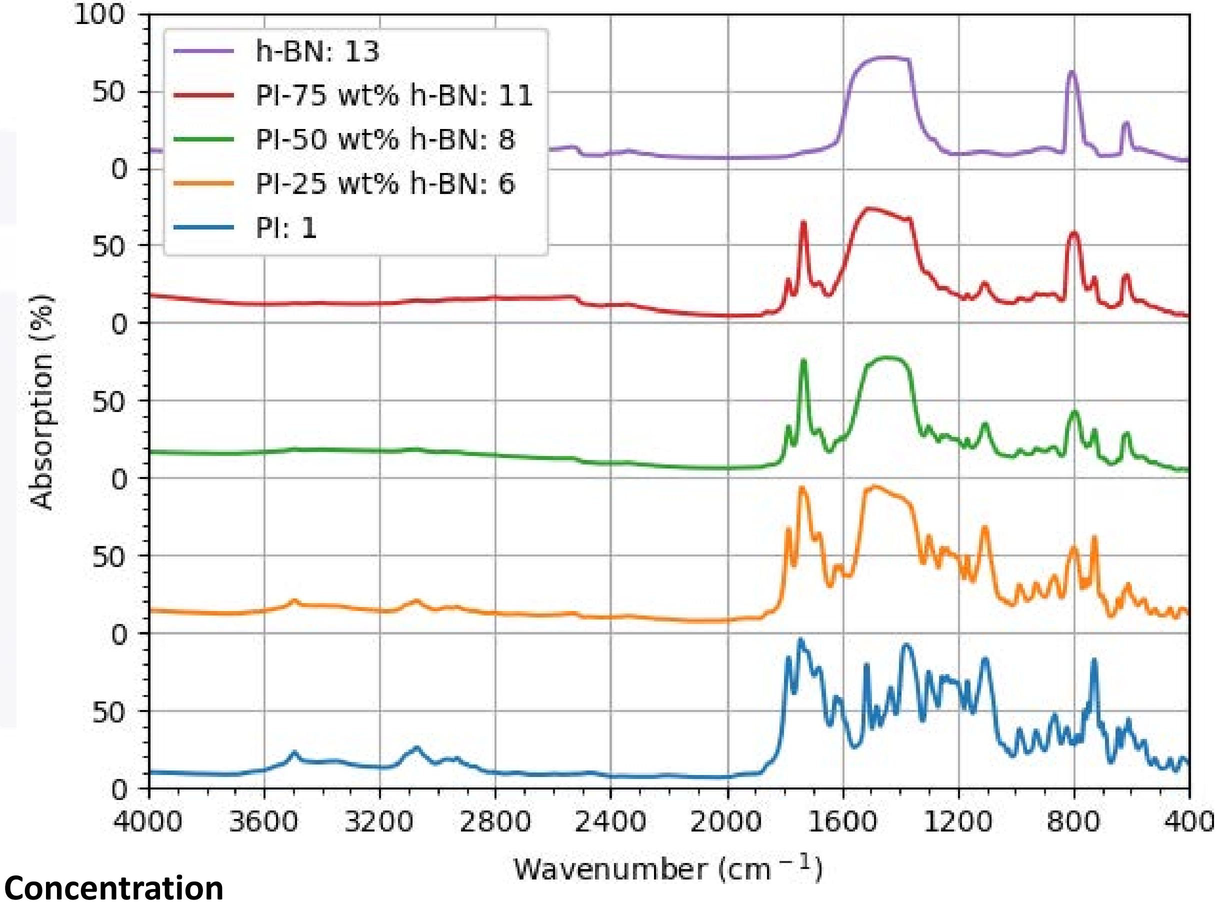
FTIR: Nanocomposites – Before Exposure



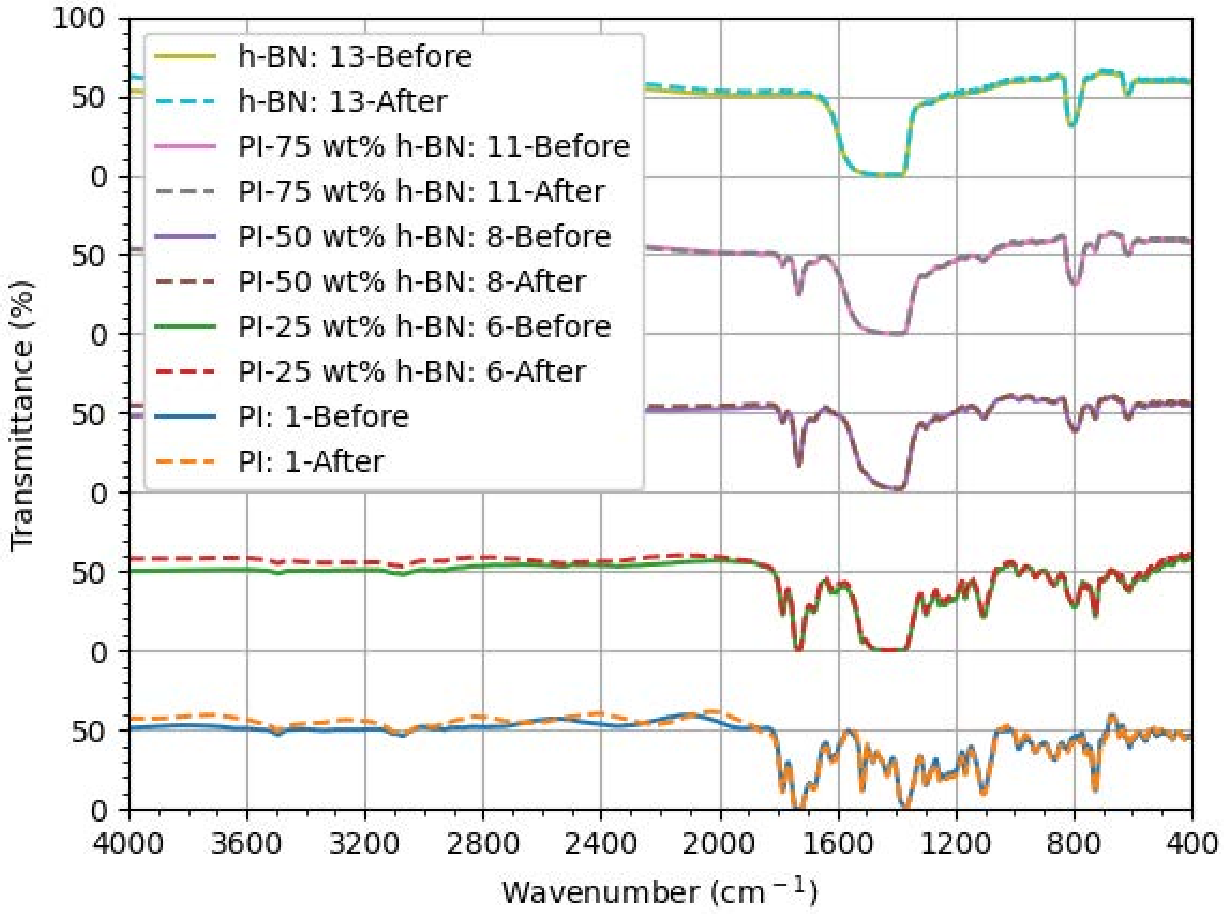


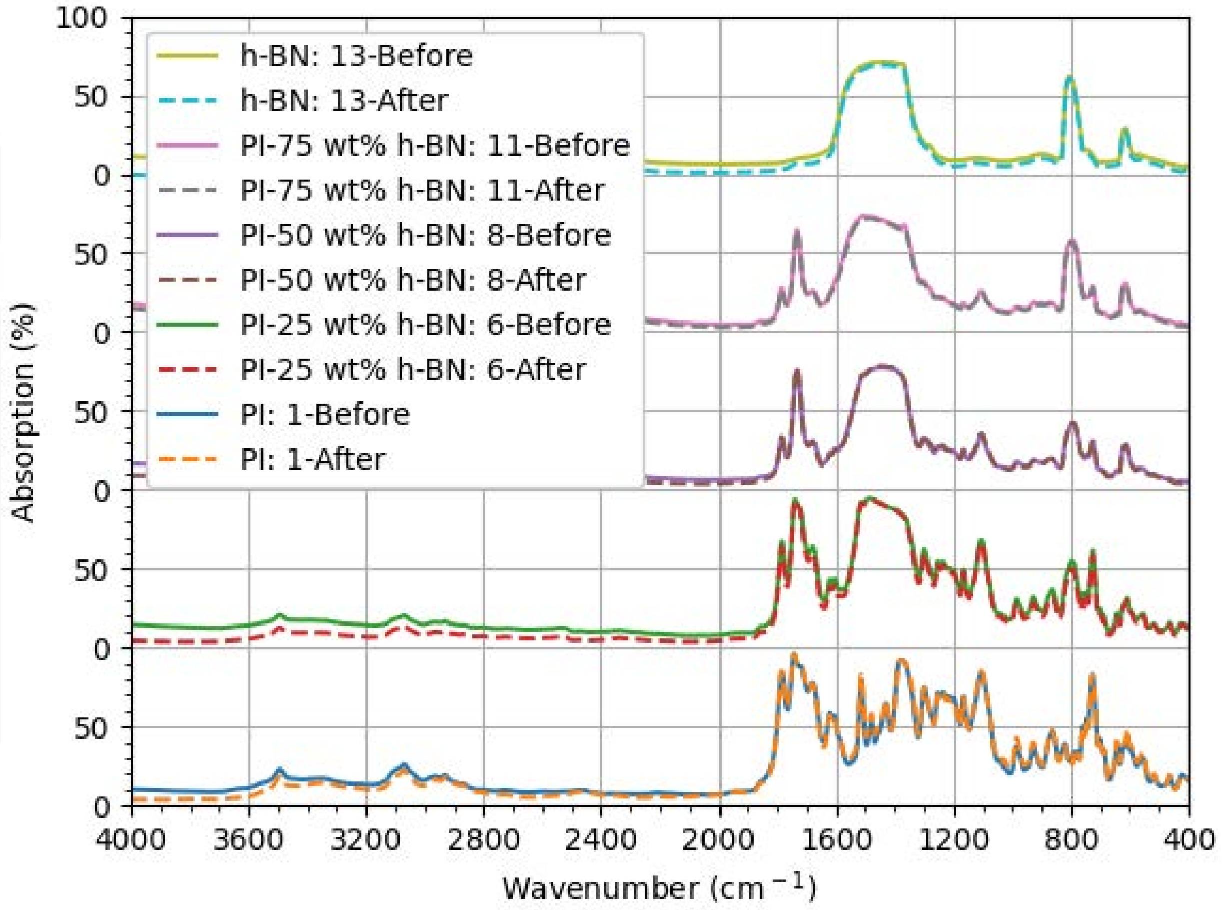
Increasing h-BN Concentration

Decreasing Polyimide Concentration

FTIR: Nanocomposites - After Exposure

Exposure: University of Massachusetts Lowell - Gamma Cave Facility - Cobalt-60 Source -Total Dosage: 80 krad (Si)





No change in peak location or intensity

Conclusion

- Formulation of PI/h-BN Nanocomposite Inks For Aerosol Jet Printing
  - Ink Concentrations:
    - PI
    - PI-25 wt% h-BN
    - PI-50 wt% h-BN
    - PI-75 wt% h-BN
    - h-BN

- Printed nanocomposite thin films were exposed to a Co-60 source up to 80 krad (Si)
- Analysis of FTIR data before and after Co-60 exposures show the materials under this study were stable up to the applied dosage as there was no change in peak locations or intensities

- **Future Work**: Measure attenuation coefficients of the nanocomposites and determine effectiveness as specifically designed radiation spot-shielding materials