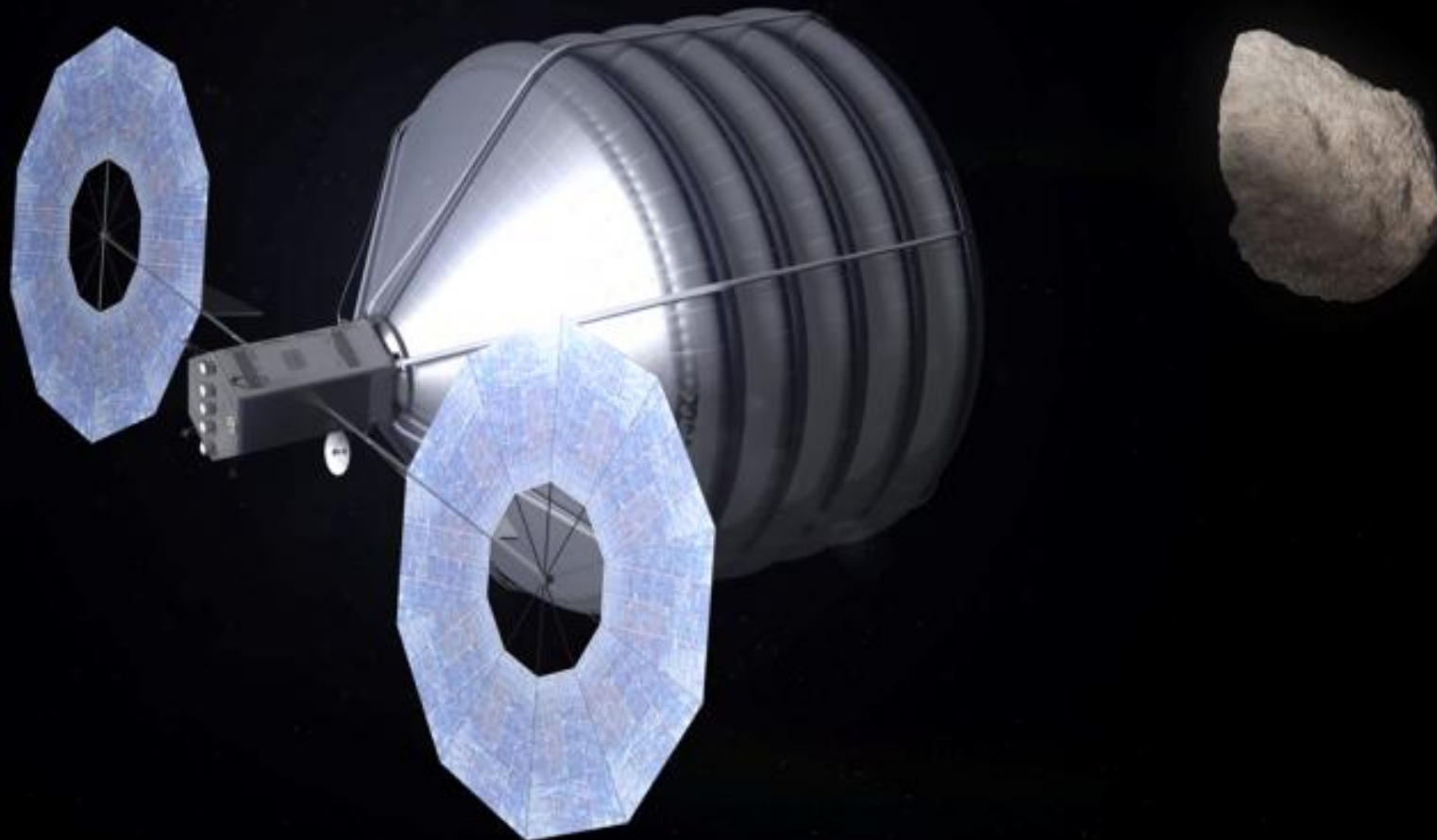


Flexible Graphene Composites for Human Space Flight Applications



Edward Sosa
ERC Inc.
MRS Fall Meeting
December 05, 2013

Graphene Properties and Applications

- **Excellent Electrical Properties**

↑ conductivity, ↑ carrier mobility, electron/hole carrier, zero gap
 $\sigma \sim 10^6 \text{ s/cm}$, $\mu \sim 200,000 \text{ cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$

- **Exceptional Mechanical Properties**

predicted strengths as high as 1TPa

- **Outstanding Thermal Properties**

$\kappa \sim 5.0 \times 10^3 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$

- **Ideal Gas Barrier Properties**

practically impermeable

- **Potential NASA Applications**

Bladder materials in inflatable structures

Lightweight liners in COPVs

Food & drug packaging

Filtration devices



Impermeable Nanocomposites

- **Flexible Composite Structures**

- elastomers: polyurethane, polyethylene, nylon, rubber, etc..
- laminate composites with graphene embedded between plies
- high transparency desirable
- Inflatable bladders, food packing

- **Rigid Structural Materials**

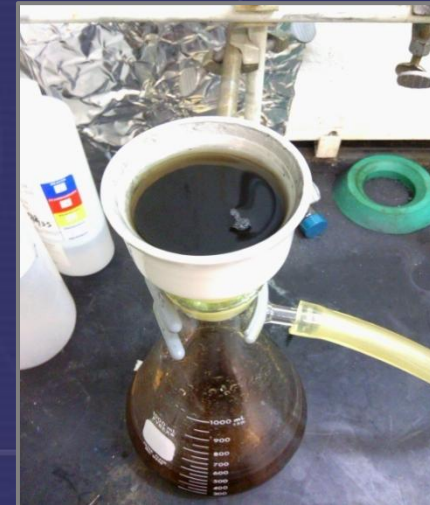
- carbon fiber composites: epoxy, polyethylene, cyanate ester
- graphene embedded between laminates or dispersed within matrix
- Pressure vessels, composite cabins

- **Methods of Graphene Deposition**

- spray coating
- spin coating
- rolling
- film transfer

Graphene Dispersions

- **Raw Graphene Nanoplatelets**
 - Variety of organic solvents
 - Tip probe sonicator @ 30% of 500W
- **Graphene Oxide**
 - 2:1 ratio sulfuric to nitric acid mixture
 - 24 hour reflux in bath sonicator
 - vacuum filtered and rinsed
 - Dispersed in water
- **Stabilized Graphene Solutions**
 - Used a variety of organic pyrene derivatives as stabilizing agents
 - Bath sonicated 4 hours to form graphene suspensions

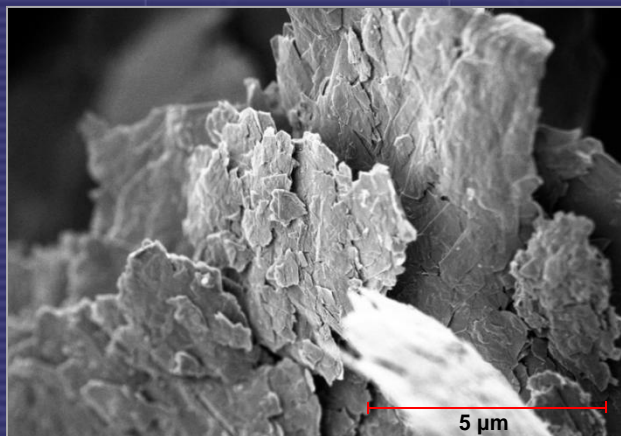


10 min sonication

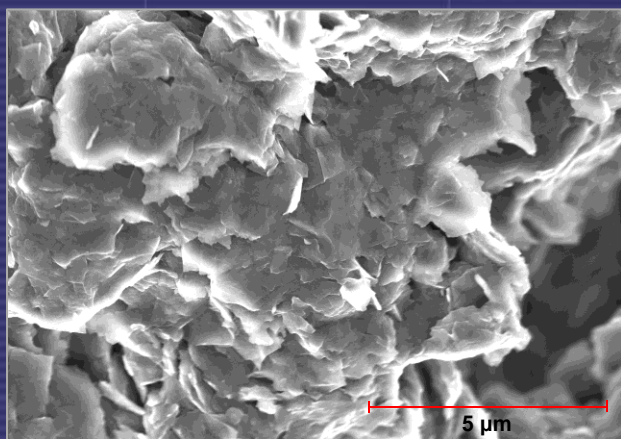


after 3 weeks

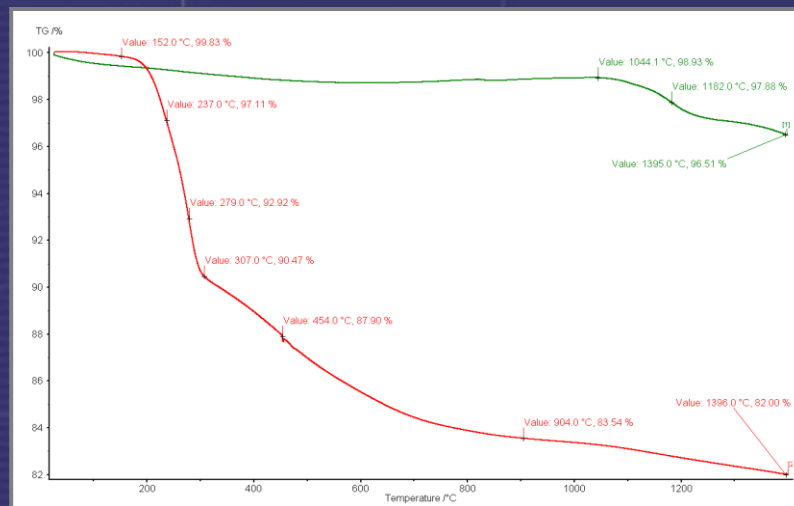
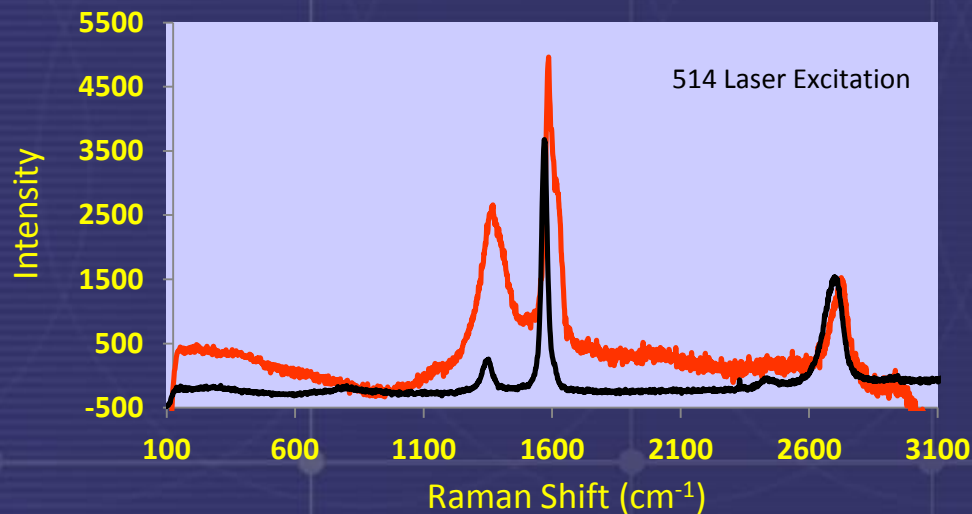
Graphene vs Graphene Oxide



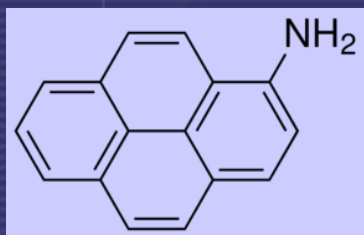
graphene



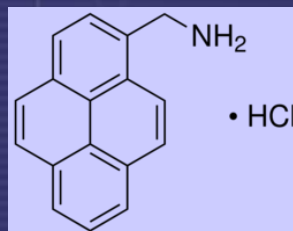
graphene oxide



Graphene Stabilization

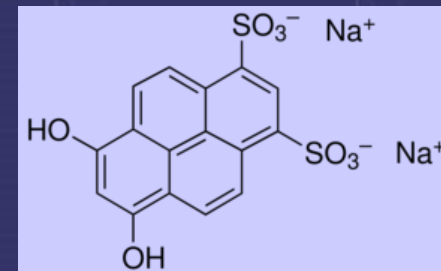


1



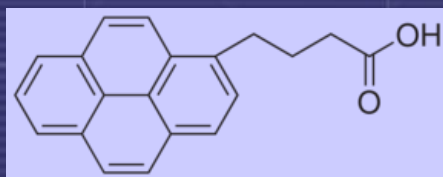
1-Pyrenemethylamine hydrochloride

2



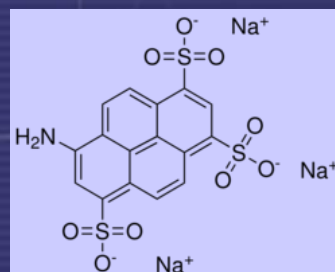
6,8-Dihydroxy-1,3-pyrenedisulfonic acid disodium salt

3

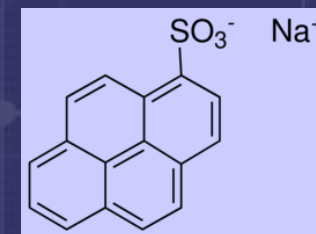


8-Aminopyrene-1,3,6-trisulfonic acid trisodium salt

4



5

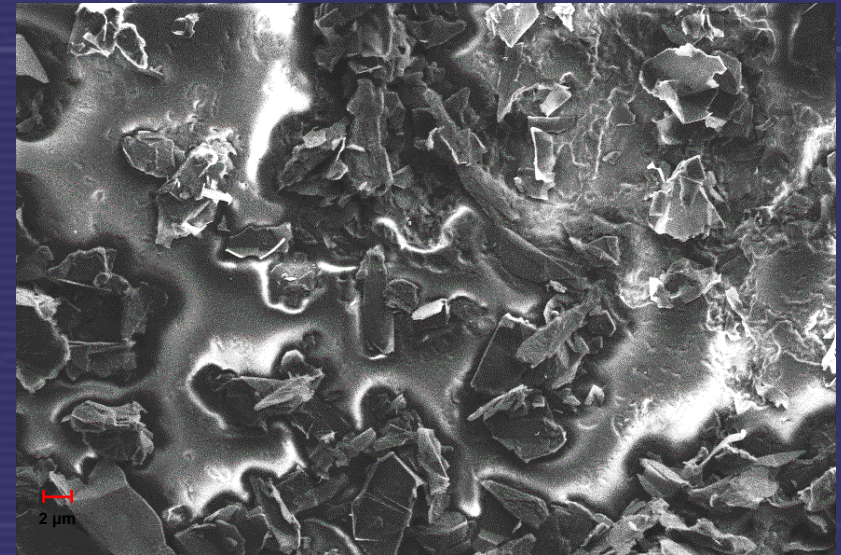
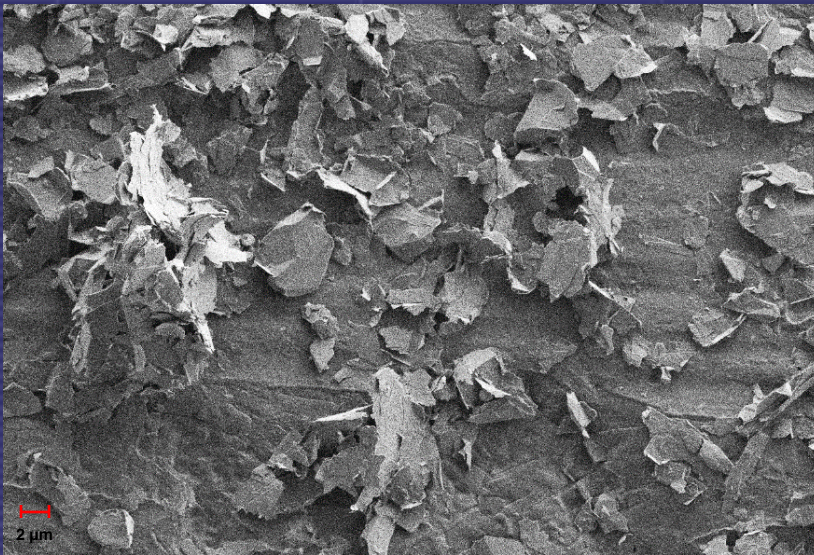


1-Pyrenesulfonic acid sodium salt



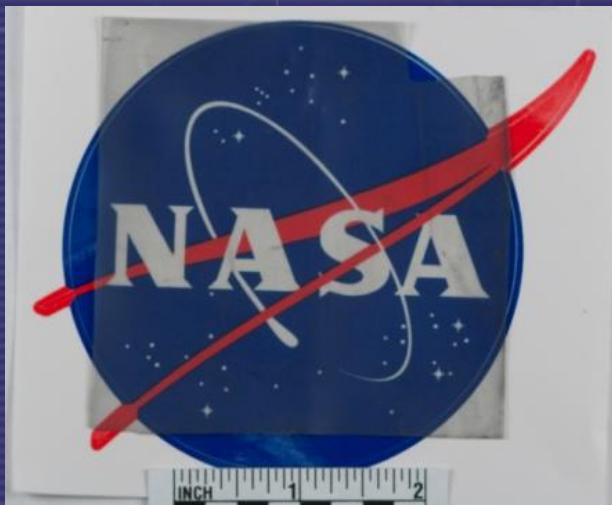
Graphene Polymer Coating

- Graphene platelets dispersed in methanol or isopropal alcohol deposited on polyurethane or polyethylene
- Hand held sprayer used to coat polyurethane or polyethylene
- Coverage is non-uniform
- Coated films hot pressed to form laminate composite



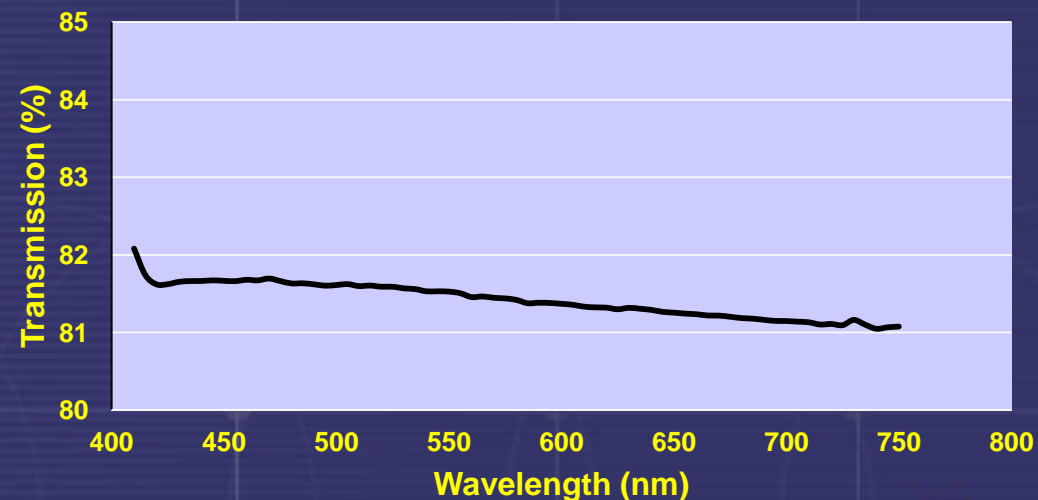
Graphene Polymer Composites

- Laminate composite consist of 2 or 3 ply of polymer sheets hot pressed
- Polyurethane 0.0015", 0.005", 0.02"
- UHMW Polyethylene 0.005"
- Graphene solutions range in concentrations from 0.25 – 1.0 mg/ml
- Semi-transparent laminate composites over visible light spectra



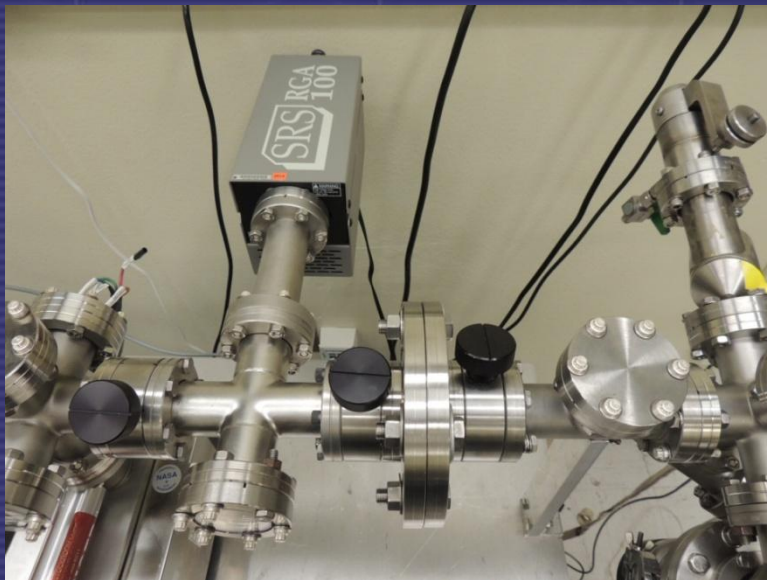
0.75 mg/ml graphene in isopropanol
9 layered coating

Optical Transmission 0.75 mg/ml Graphene on
Polyurethane

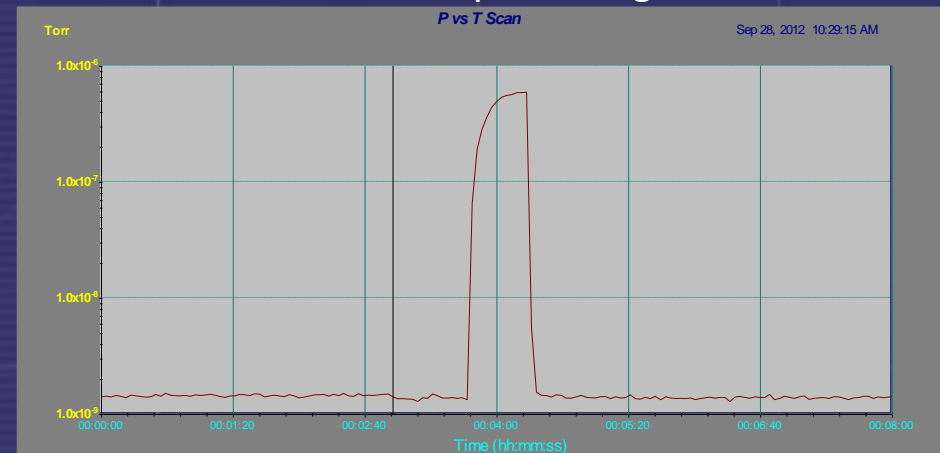


Permeation Testing

- Permeation testing in custom built vacuum apparatus
- System differentially pumped with turbo and ion pumps
- Composite films placed between ASA flanges to make vacuum seal
- One side held at high vacuum the other side 1 atm He
- Residual gas analyzer used measure He partial pressure increase
- Composite permeation calculated from slope of permeation curve

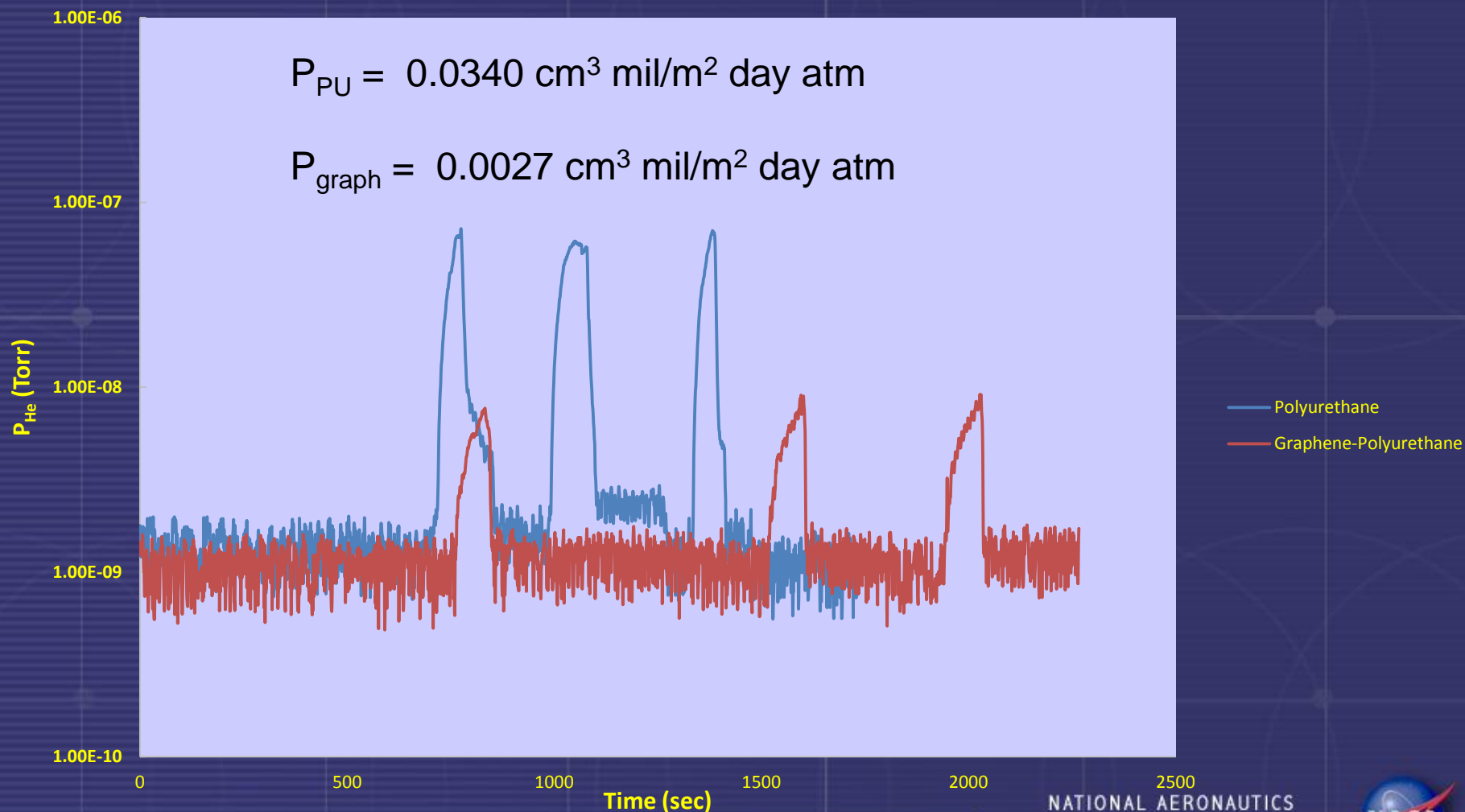


$$\frac{\text{Volume (STP)} \times \text{thickness}}{\text{Area} \times \text{time} \times \text{pressure gradient}}$$



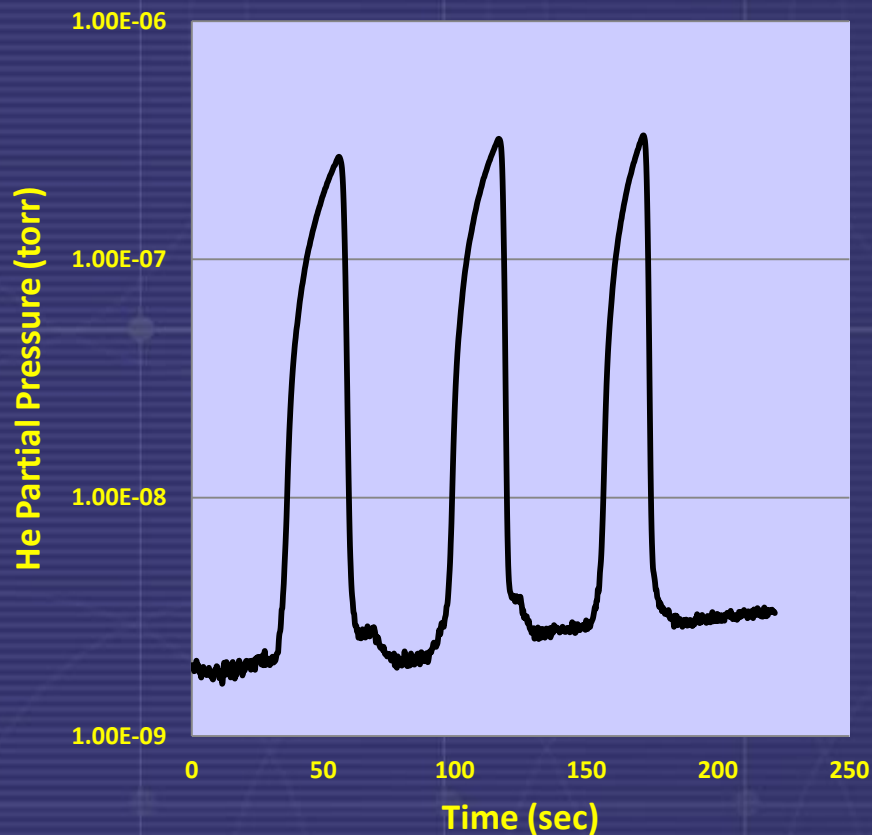
Polyurethane Composite Permeation

Helium Permeation

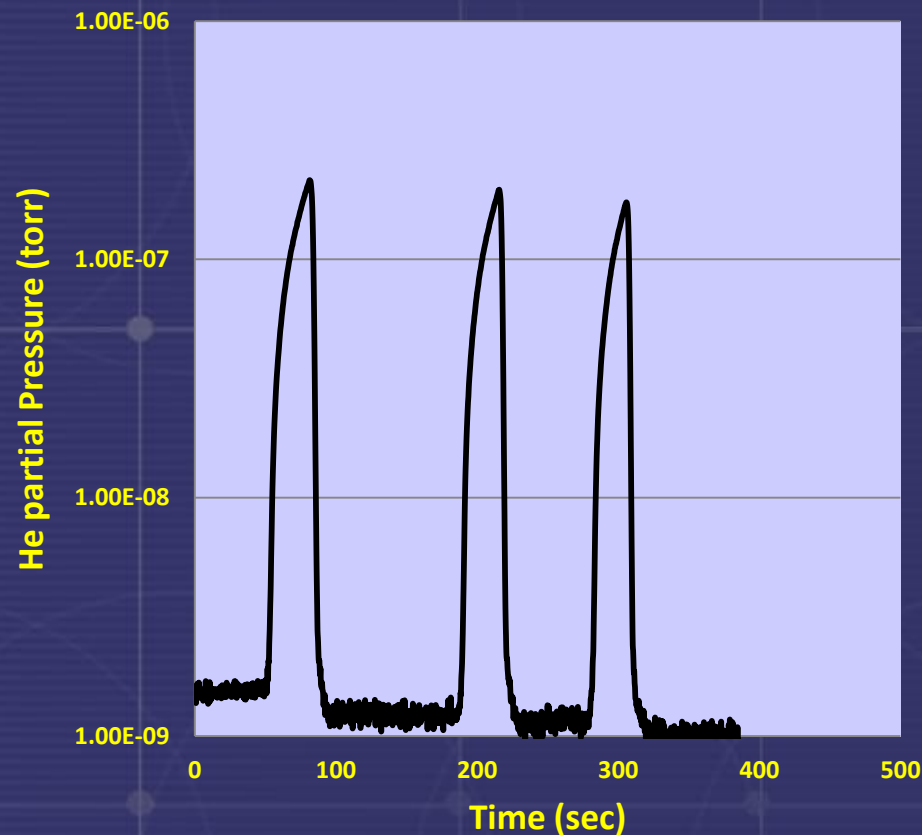


Polyethylene Composite Permeation

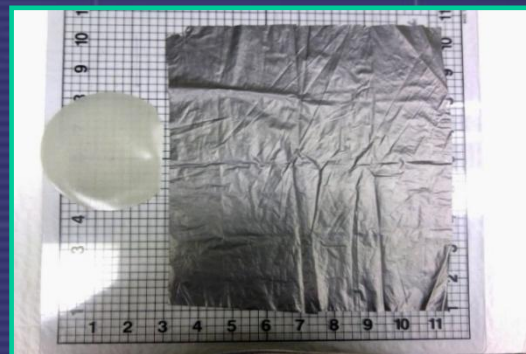
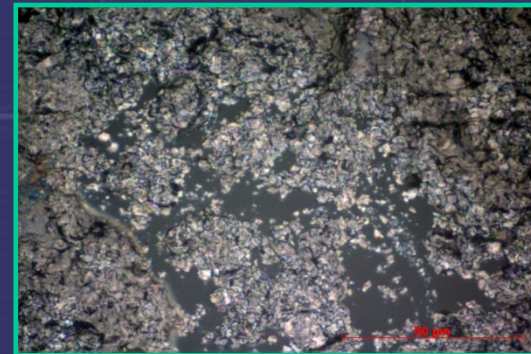
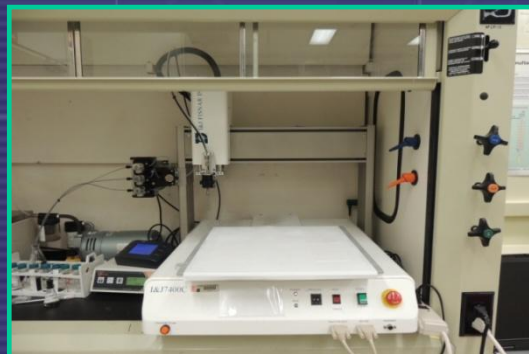
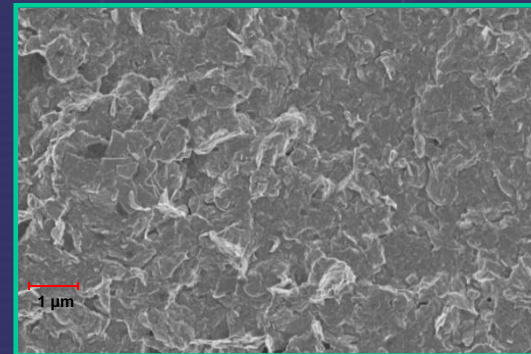
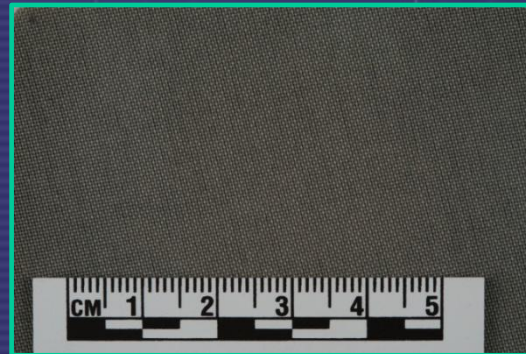
Polyethylene Helium Permeation



Graphene Composite Helium Permeation

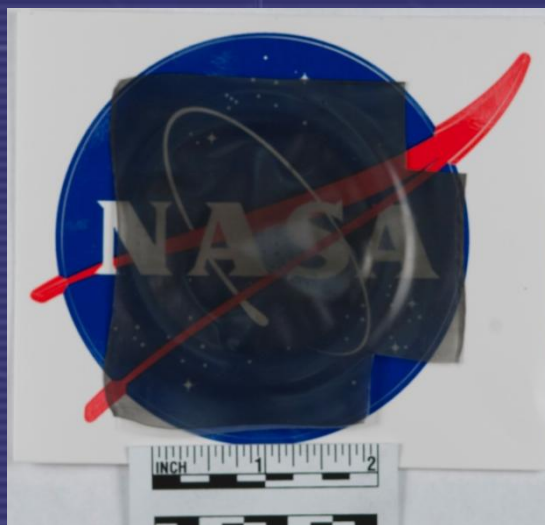


Graphene-Oxide Deposition on Polymer



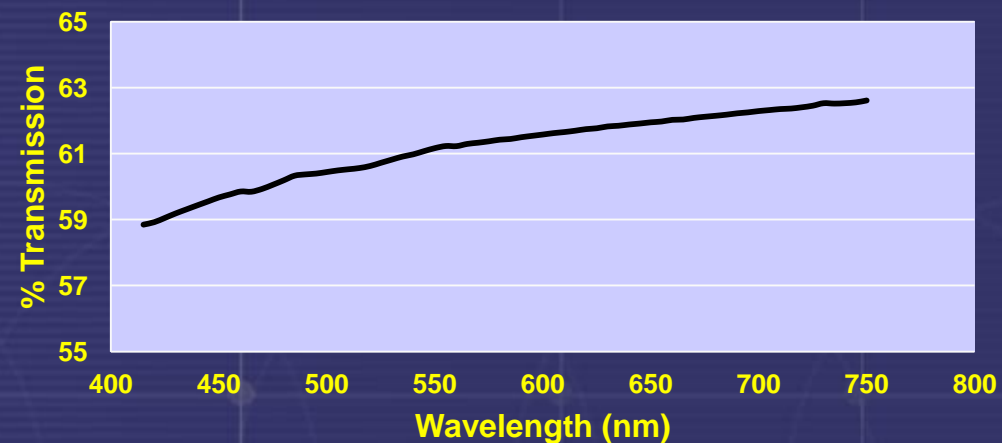
Graphene-Oxide Polyurethane Composites

- Graphene oxide composite consisted of 9 coats from 1.0 mg/ml solution
- Coating are much more uniform across due to better dispersion stability
- Same number of coats relative to plain graphene result in thicker coatings, result in lower transmission in visible region



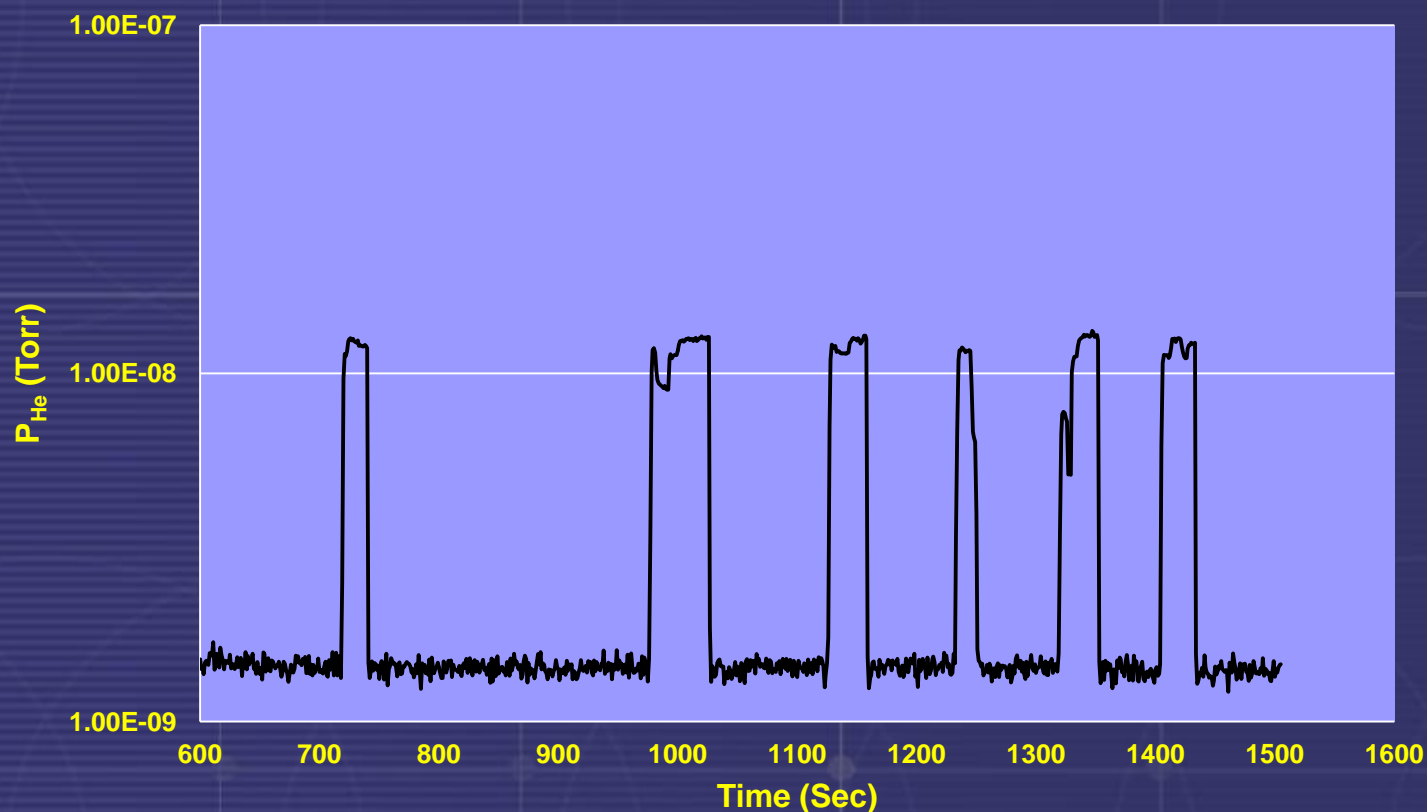
1.0 mg/ml graphene oxide in methanol

Optical Transmission 1.0 mg/ml GO on Polyurethane



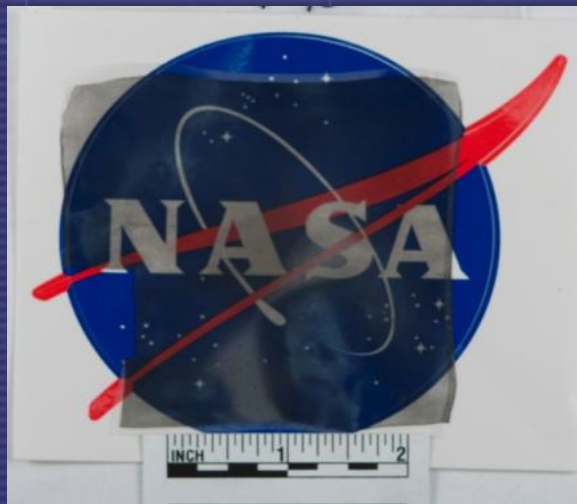
Graphene-Oxide Composite Permeation

Permeation 1.0 mg/mL Graphene Oxide on Polyurethane ST3655
9 Coats



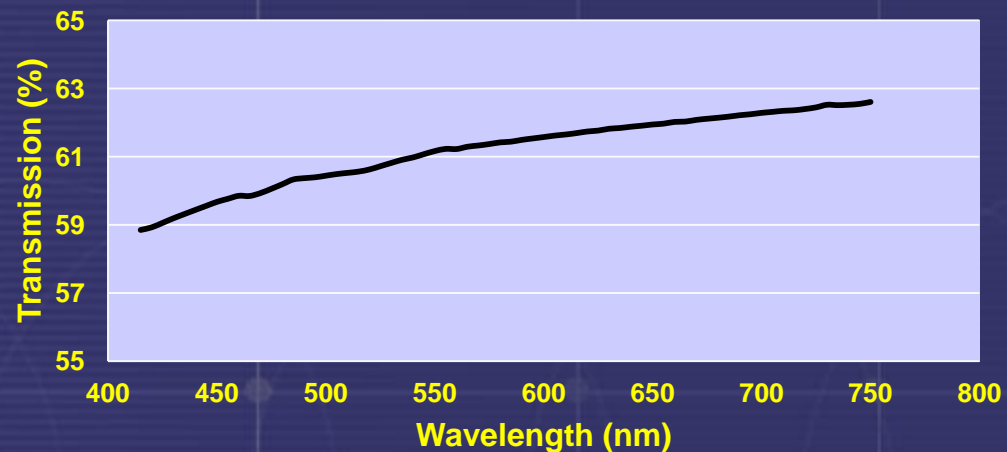
Graphene/GO Composite Fabrication

- Use GO as a seed layer to allow better deposition of the pure graphene
- Polyurethane sheet coated with 6 layers GO followed by 6 layers graphene
- Optical transmission on the same order as laminate composites composed of 9 layers of GO



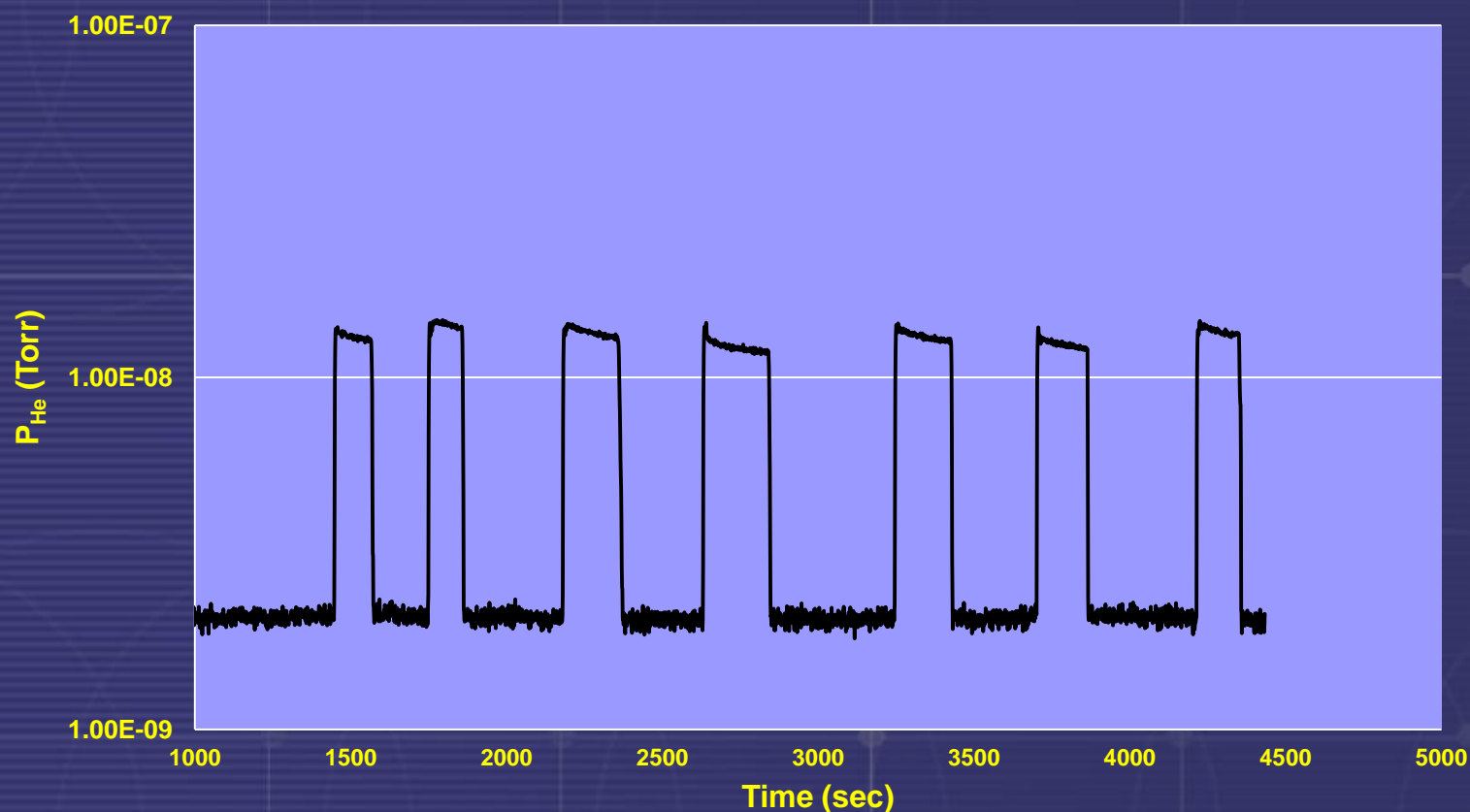
1.0 mg/ml graphene oxide and 0.75 mg/ml graphene in methanol

Optical Transmission of 0.75 mg/mL Graphene and 1.0 mg/ml GO on Polyurethane



Graphene/GO Composite Fabrication

Permeation 0.75 mg/mL Graphene on Polyurethane ST3655
9 Coats



Conclusions

- Graphene oxide allows for better dispersion stability in aqueous and organic solvents
- Stabilizers provide dispersion of pristine graphene
- Roll coating provide the best coverage of polyurethane sheets
- Graphene and GO coated polyurethane used to fabricate flexible laminate composite
- Permeation testing indicates that pristine graphene acts as a better gas barrier material
- Continuous graphene films are expected to provide even better gas barrier properties

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