Atomic Oxygen-Resistant, Static-Dissipative, Pinhole-Free Coatings for Spacecraft

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Motivation

- **Replacing metal satellite components with polymeric composites results in substantial weight savings**

BUT…

- **Polymeric composites are susceptible to erosion by atomic oxygen that is present in Low Earth Orbit (LEO)**
  - 10^{12} – 10^{16} O atoms/cm^2-sec at 5 eV translational energy for a satellite moving at 8 km/sec
- **Most polymers are insulators, with the potential to accumulate significant static charge**

SOLUTION:

- **Apply a coating that is resistant to atomic oxygen (AO) and can dissipate static**

Degradation of Spacecraft Materials

*S.K.R. Miller & B. Banks, MRS Bulletin, **35** (2010) 20-24.*

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- **Baseline: Kapton**
  - Erodes at a rate of $3x10^{-24}$ cm^3/incident O atom

Protective Coatings

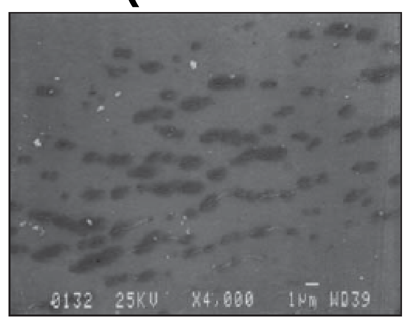
- **PVD Aluminum**
  - ✓ Surface is fully oxidized
  - ✗ Has pinholes
- **PECVD SiO_2**
  - ✓ Fully oxidized
  - ✗ Has pinholes
  - ✗ Insulator, builds static charge
- **Surface modification e.g., Photosil** *J. Kleiman, MRS Bulletin, **35** (2010) 55-65.*
  - ✗ Insulating surface

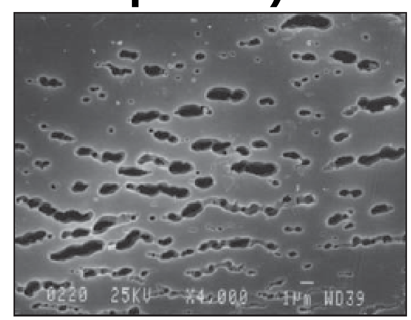
Al-backed Kapton Sheet (Kapton side exposed)

Before Flight **On-orbit**

O'Neal et al., NASA SP-531 (1996).

Al-coated Kapton Sheet After Orbit (Aluminum side exposed)





Defects in Al **Kapton remaining after chemical removal of Al**

*de Groh & Banks, J. Spacecr. Rockets **31** (1994), 656.*

Requirements for Spacecraft Coatings

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- **Resistant to atomic oxygen**
- **Static dissipative** Sheet resistance < 10^5 Ω/square
- **UV-resistant**
- **Pinhole-free**
- **Thin** To reduce weight
- **Conformal** To protect areas out of line-of-sight
- **Optical properties requirements depend on application**
  E.g., transparent for solar arrays, reflective for other uses
- **Mechanically robust** Adhesion, wear, stretch, bend, …

⇒ ***A new deposition method is needed***

ATOMIC LAYER DEPOSITION

Atomic Layer Deposition (ALD)

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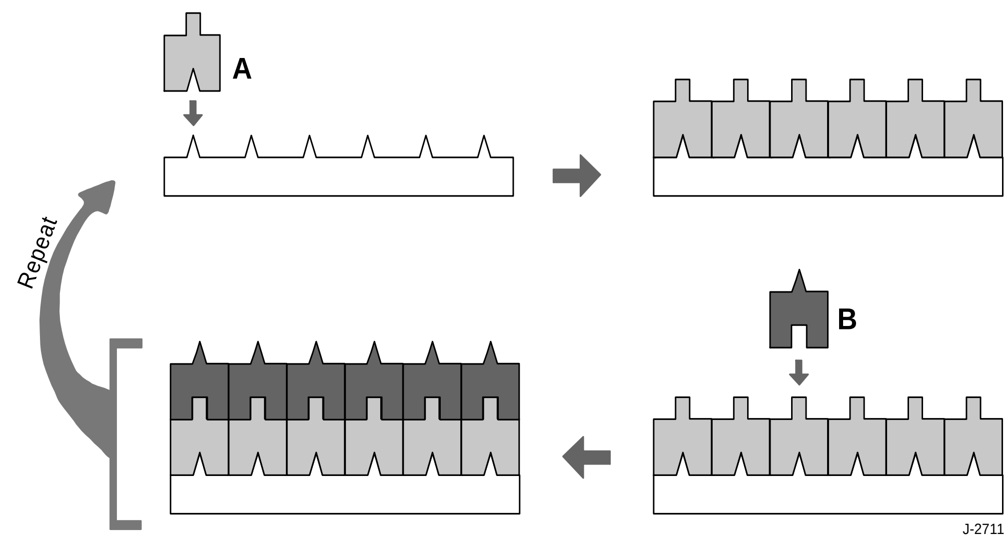
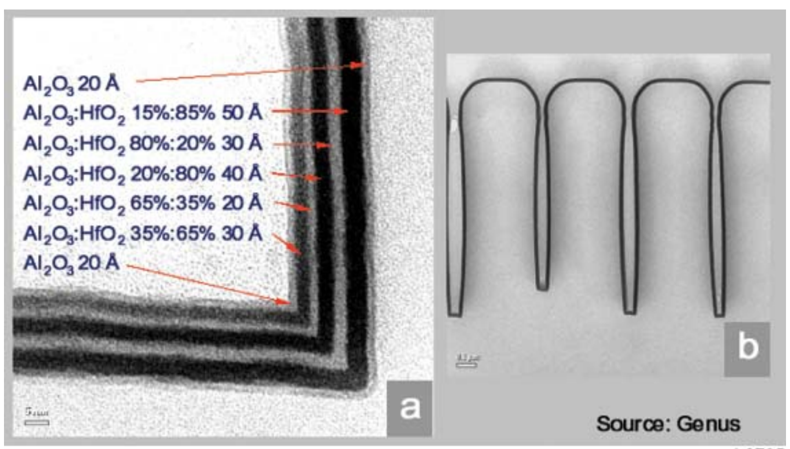
- **Similar to Chemical Vapor Deposition (CVD)**
  – Thin film growth on a substrate from gas phase precursors
- **EXCEPT… precursors are delivered sequentially rather than simultaneously**
- **Purge in between $\Rightarrow$ No gas phase reactions**
- **Self-limiting surface adsorption**
- **Monolayer-by-monolayer surface-mediated growth**
- **All exposed surfaces are coated**



Benefits of ALD

- **No pinholes**
- **Extremely conformal**
- **Extremely thin (nm)**
- **Low temperature process**
  - Can deposit on plastics
- **Commonly used in the semiconductor industry**



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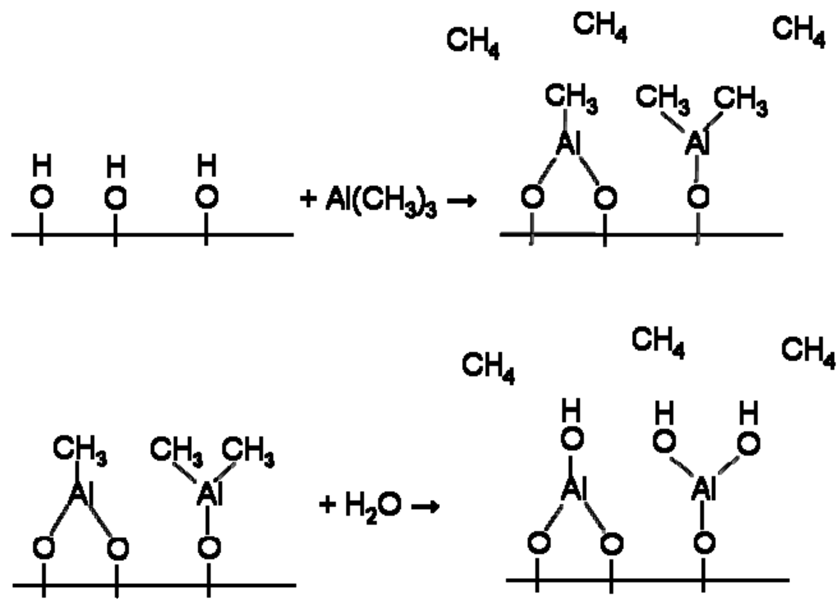
Typical ALD Reaction:

$$2\,Al(CH_3)_3 + 3\,H_2O \rightarrow Al_2O_3 + 6\,CH_4$$



Screening Process for ALD-grown Spacecraft Coatings

- **Select a variety of oxide and other coatings to evaluate in simulated space conditions**
  - Some optically transparent, some reflective
- **Apply coatings to Kapton coupons with ALD**
  - Process temperatures ≤ 300°C
  - Coating thicknesses ranged from 6 – 100 nm
  - Both single layer and dual layer coatings
- **Expose to simulated Low Earth Orbit conditions in PSI's FAST™ AO source**
  - Over 60 samples with 10 different coatings have been evaluated
  - Uncoated Kapton used as control
- **Look for erosion (SEM), changes in conductivity, optical properties, etc.**

Atomic Oxygen Testing of Spacecraft Coatings: PSI FAST™ O Atom Source

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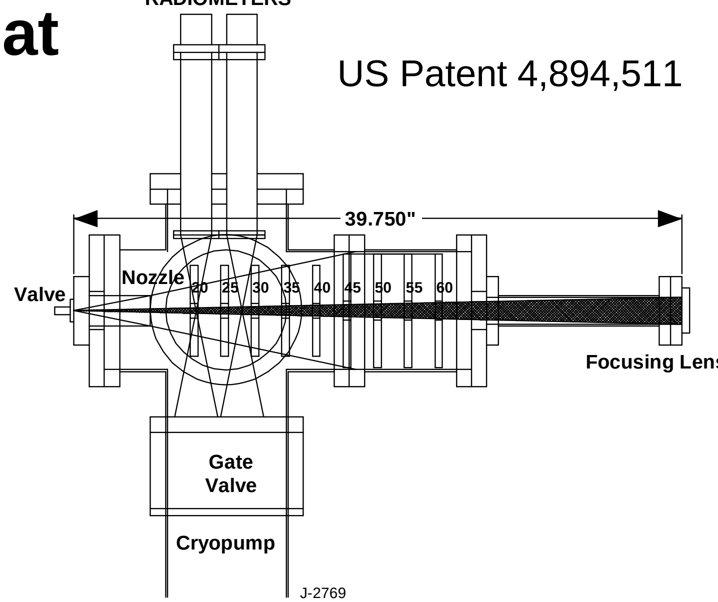
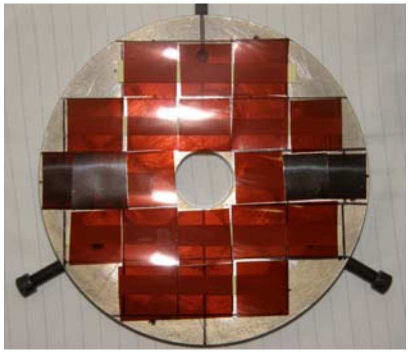
- **High power pulsed CO_2 laser to heat and dissociate O_2**
- **Closest approximation to LEO of any available O atom source**
  - **Energy distribution**
    Velocity and Boltzmann spread
  - **Ion content**
    ~1% O^+, matches LEO at 600 km
  - **UV/VUV photon flux**
    10^{-4} photons/O atom, matches LEO at 230 km
- **Large beam area to test many samples**
- **Typical exposure: $10^{19} – 10^{20}$ O atoms/cm^2 in 16 hours** Equivalent to weeks to months on the International Space Station

US Patent 4,894,511

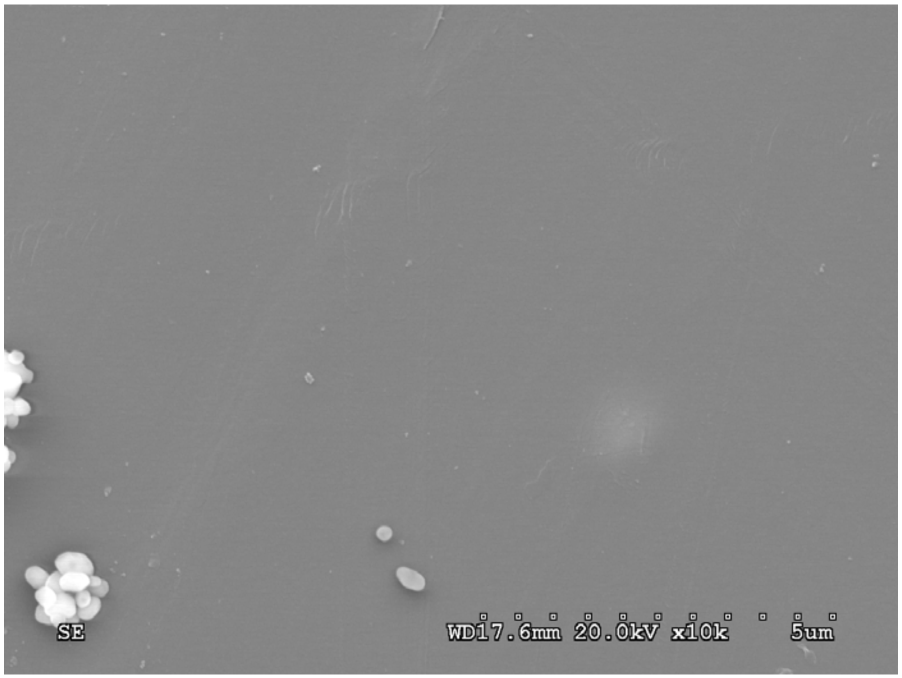




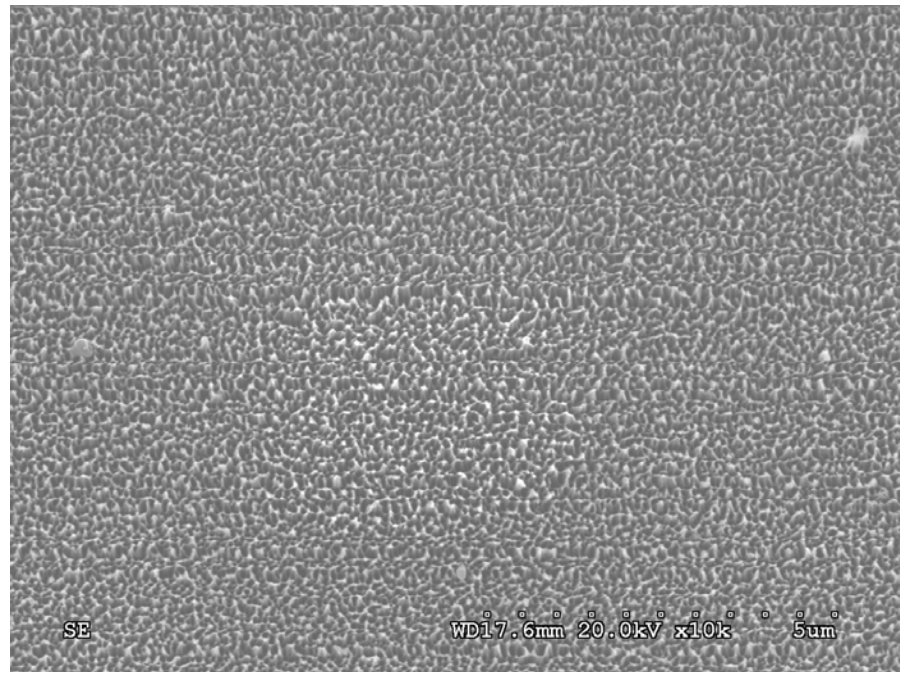
Sample size ~1" x 1"

Erosion of Kapton Control

Bare Kapton, As-Received



Bare Kapton, AO-Exposed



5 µm

- **Bare Kapton is severely eroded by exposure to AO**
  - Dust particles are included in the "as-received" image to provide a focal point

ALD Coatings Can Protect Kapton

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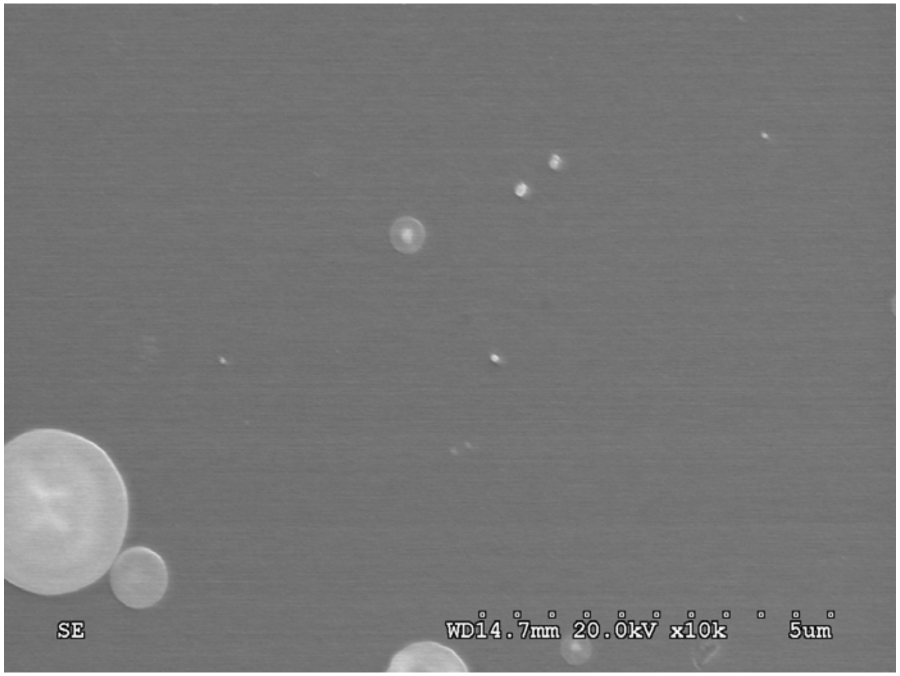
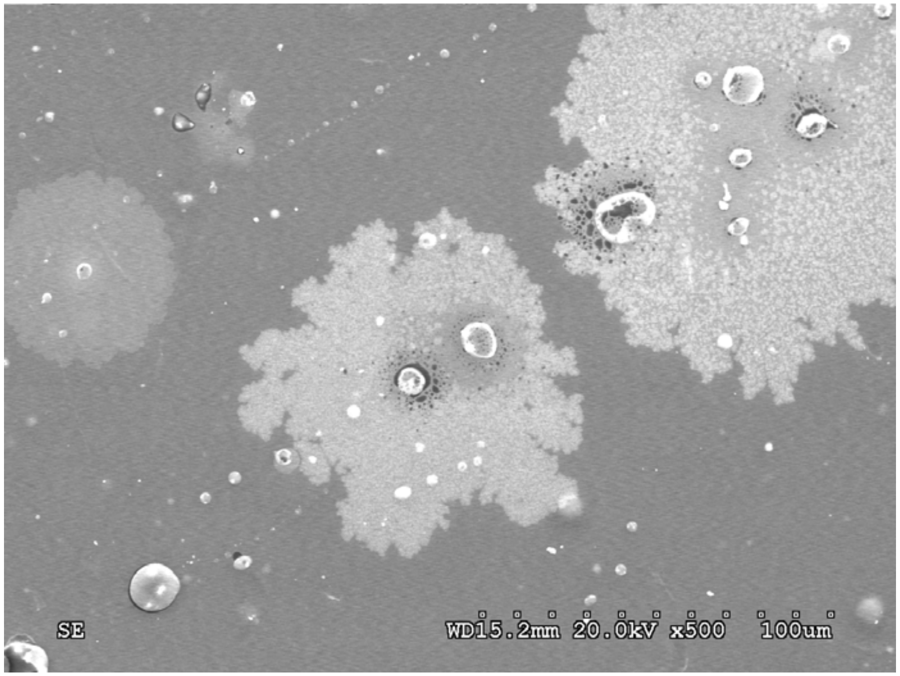
Successful coating, after thermal cycling and AO exposure

Circles are contamination



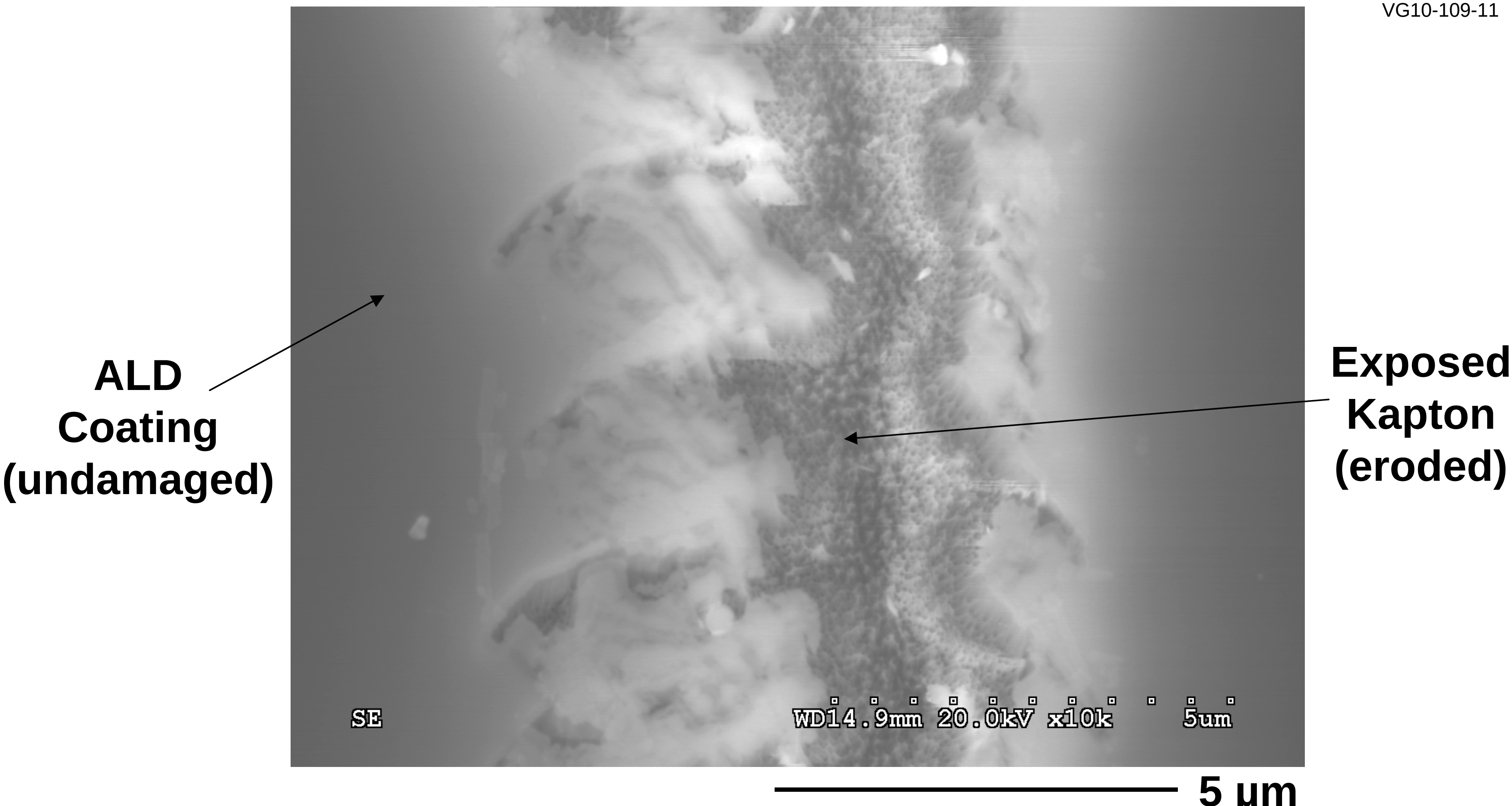
5 µm

Unsuccessful coating, after AO exposure



100 µm

Erosion of Exposed Kapton Area on ALD-Coated Coupon



- ALD coating was scratched prior to AO exposure
- Erosion is limited to abraded area

Results of Screening Matrix

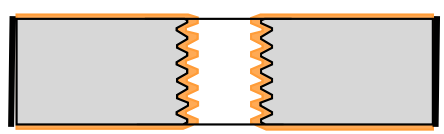
- ***SEM:***
  8 of 10 coatings showed no degradation after AO exposure.

- ***Static Dissipation:***
  6 of 10 coatings had sheet resistance < 10^5 Ω/square both before and after exposure to atomic oxygen.

- ***Optical Properties:***
  8 of 9 transparent coatings showed no change in UV-Vis reflectance. The reflective coating became more reflective.

- ***Adhesion:***
  Of 3 coatings tested, 2 passed a scribed tape adhesion test.

- ***Conformality:***
  The one coating tested demonstrated conductivity down a threaded hole



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Down-Selection of ALD Coating

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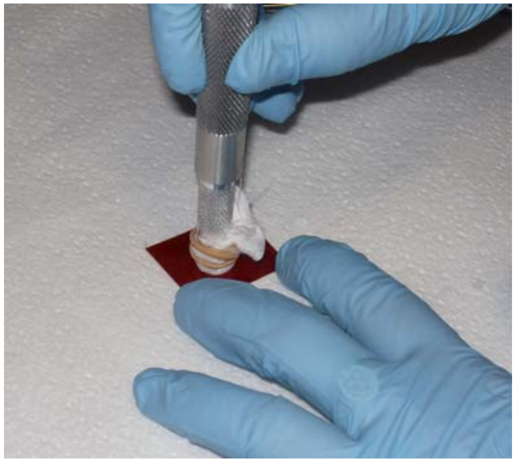
- **One single-layer transparent coating has been selected**
  - Focus on solar array applications
- **ALD process temperature reduced to 120°C**
  - A novel precursor has been synthesized
- **Minimum coating thickness determined to be 17 nm for Kapton substrate**
- **Further testing:**
  - Evaluate mechanical properties: Bend, stretch, wear, adhesion
  - Expose coatings to thermal cycles in vacuum
  - Demonstrate deposition on model spacecraft materials
    - Carbon-epoxy composite

Evaluation of Mechanical & Thermal Properties

- **Coated Kapton coupons subjected to mechanical and thermal stresses**
  - **Stretch**: Apply tension in Instron mechanical test apparatus
  - **Bend**: Wrap around mandrel
  - **Wear**: Cheesecloth abrasion test
  - **Adhesion:** Scribed tape test
  - **Thermal Vacuum**: 5 cycles from -30°C to 70°C at 10^{-6} torr
- **Expose to AO after stretch, bend, wear, thermal vacuum**



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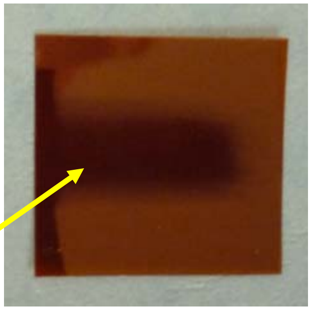
Results of Mechanical & Thermal Testing

- **Coating passed 4-level scribed tape adhesion test**
- **Stretching, bending, and thermal cycling had no effect on the coatings**
  - No change in sheet resistance
  - No cracks or delaminations detected by SEM
  - No erosion after AO exposure
    - Fully coated coupons had mass losses < 0.3% of bare Kapton
    - Essentially inert within resolution of microbalance
- **Wear test removed the coating**
  - Mitigation: Apply sacrificial clean ablating coating in areas to be handled during satellite assembly



Worn region

Protection of Carbon-Epoxy Composite: Bare Sample is Severely Eroded by AO

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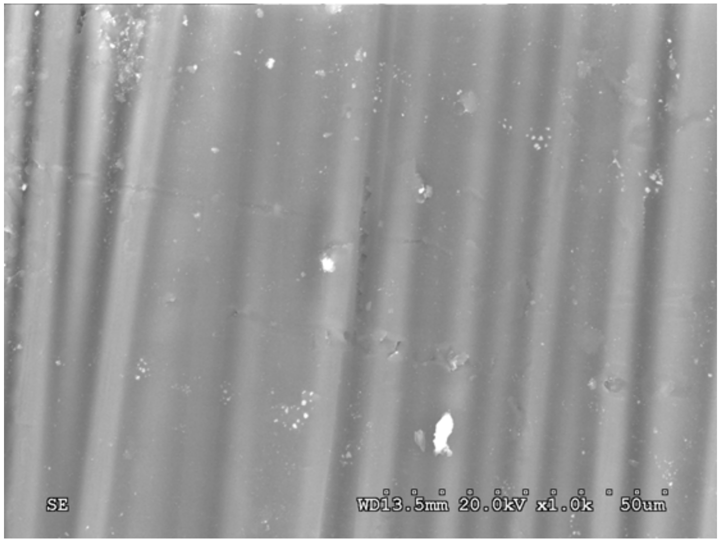
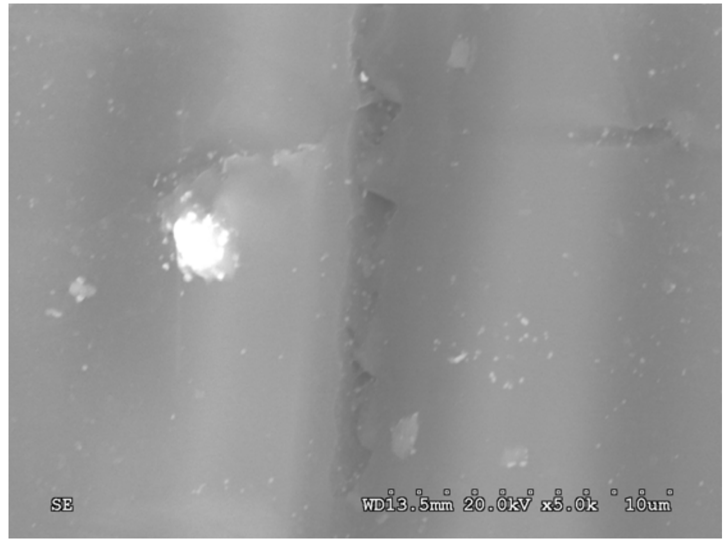
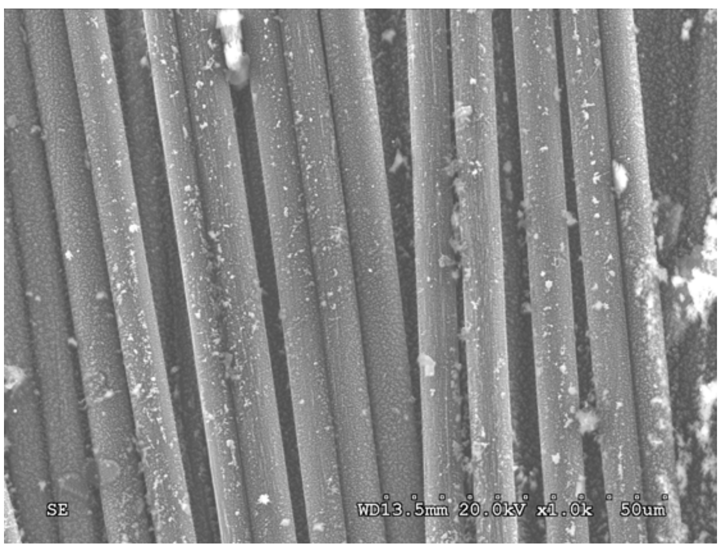


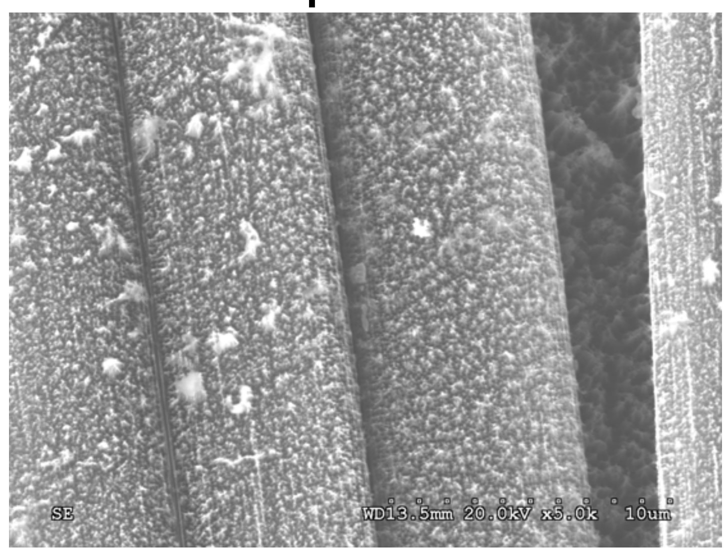


As Received: C fibers buried in epoxy matrix

50 µm

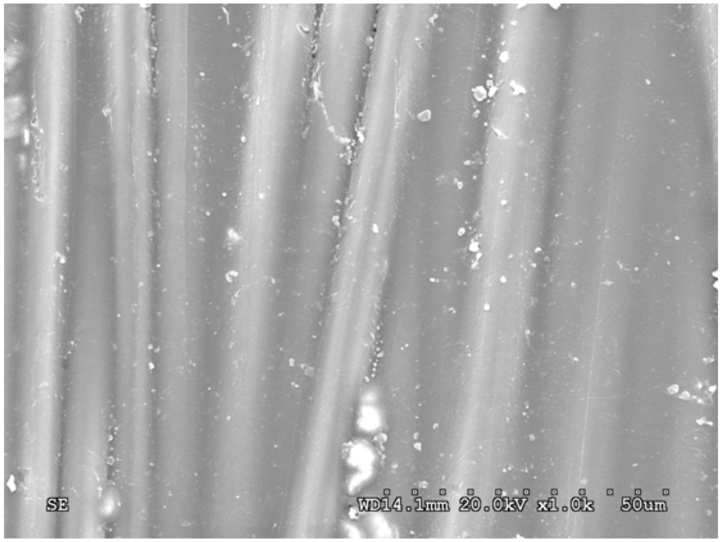
10 µm

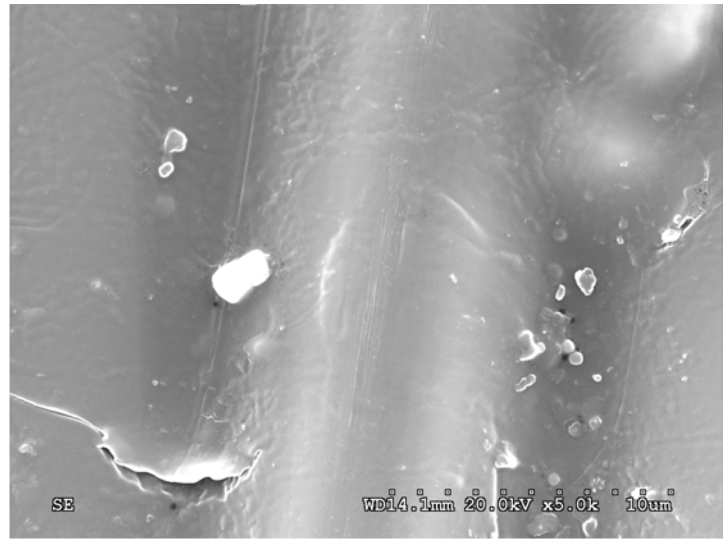




After AO: Epoxy matrix is gone, fibers are damaged

Protection of Carbon-Epoxy Composite: ALD-Coated Sample is Unchanged

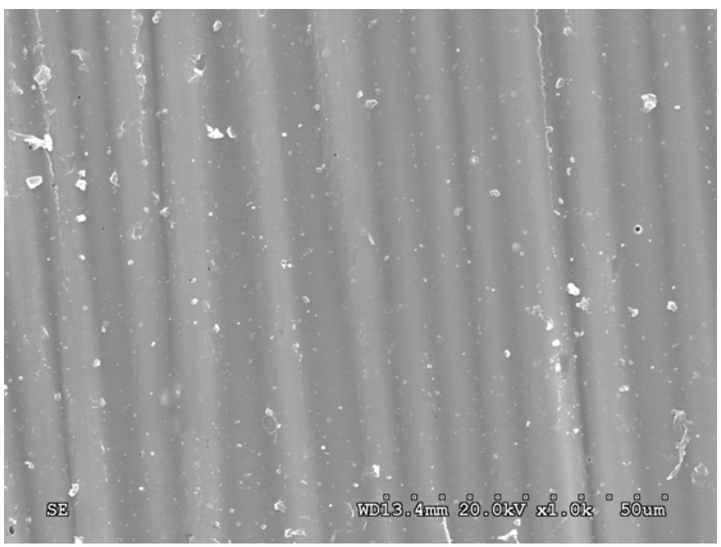


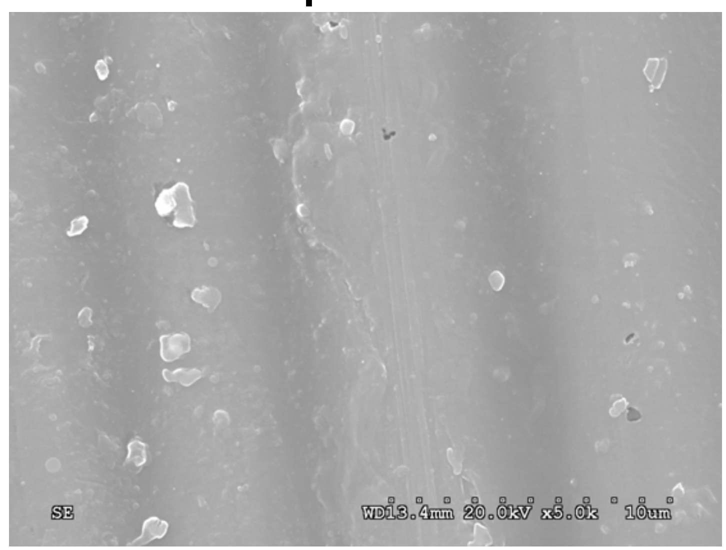


After ALD coating is applied

50 µm

10 µm





After AO: Matrix and fibers are intact. No damage detected.

Conclusions

- **We have developed a pinhole-free, static dissipative ultra-thin ALD-grown coating that protects polymeric and composite materials from erosion by atomic oxygen in a simulated Low Earth Orbit environment**
- **The minimum coating thickness to protect a Kapton substrate is 17 nm**
- **The coating survives bend, stretch and thermal stresses and exhibits good adhesion**
- **Current work: Scale up the process to coat model satellite components**

Supplemental Information

ISN'T ALD SLOW??

Yes, but….

- ***<u>ALD can make coatings that can't be made by any other technique</u>***

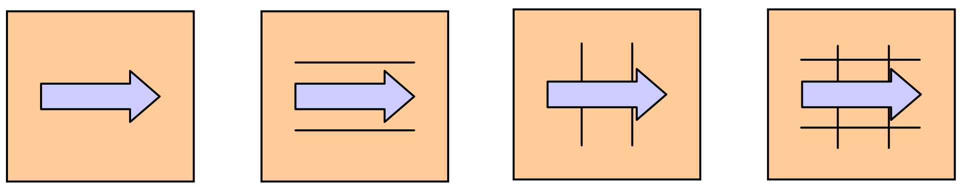
- **Lower deposition temperatures enables coating of polymeric materials**
  - Temperature uniformity requirements are less stringent than for CVD

- **ALD is well-suited to batch processing**
  - Non-line-of-sight

- **For thin coatings, the overall throughput may be comparable to PVD or PECVD processes**
  - Including sample loading, pump-down, etc.

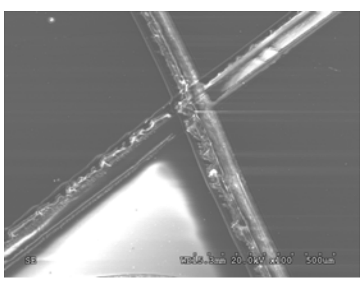
Adhesion: Four Level Scribed Tape Test

- **Scratch the coating according to 4 patterns**
- **Apply Scotch tape and peel off quickly**
- **Look for delamination**

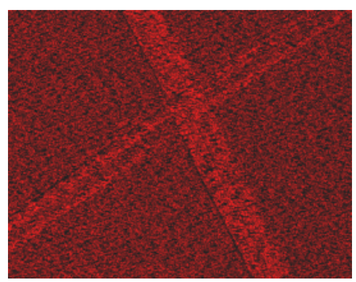


1 2 3 4

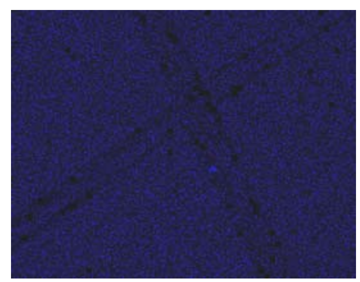
Successful adhesion test of ALD coating



500 µm



Carbon (Kapton)



ALD Coating

Coating remains intact after 4th level tape test