Coating hollow 3D objects with uniform low-density films by combining EPD and ALD


March 13, 2017
Goal

Fabrication of a foam liner on the inside of a hollow cylinder (=hohlraum)
- sub 50 mg/cm$^3$
- ~200 micron thick

Why do we need this?

Foam liner reduces wall motion and increases shape control

- low fill hohlraum (0.03 – 0.6 mg/cm$^3$ Helium)
- 20 mg/cm$^3$ Ta$_2$O$_5$, 300 um hohlraum liner
Approach: EPD + ALD

1) Deposit thin layers of foams on flat geometry

- Electrophoretic Deposition (EPD)
  - Electrophoresis
  - Electrode
  - PS bead layer
- Atomic Layer Deposition (ALD)
  - Coat template
  - Remove template
  - Burn/wash
- Low density foam with controlled density/pore size

![Diagram of EPD and ALD processes](image)

2) Transfer process to cylindrical geometry

- Counter Electrode
- Deposition Electrode
- Polystyrene Suspension

![Diagram of cylindrical geometry](image)

Projected density:

- 2 nm Al2O3
- 5 nm Al2O3
- 10 nm Al2O3

Density of templated material mg/cc vs. diameter of sphere in micron.
Tasks and Challenges

Deposit PS beads on Au flat by EPD
  • Finding the right beads and EPD conditions
  • Surface smoothness
  • Drying/cracking
  • Thickness (200 micron)

Homogeneously coat PS deposit with ALD
  • Diffusion of ALD precursor into polymer
  • ALD temperature (deformation, crack formation)

Remove PS template
  • Complete removal of PS
  • Crack formation

Transfer to cylindrical geometry
Proper bead selection is critical for coating uniformity

<table>
<thead>
<tr>
<th>vendor</th>
<th>X linked</th>
<th>carboxylated</th>
<th>bead size</th>
</tr>
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<tr>
<td>ST</td>
<td>X</td>
<td></td>
<td>1 μm</td>
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<td></td>
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<tr>
<td>ST</td>
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ST: Spherotech, PS: Polyscience

PS beads result in unconformal coating under various conditions:

Parameters explored:
- Bead concentration
- Flow rate
- pH
- Electrode pretreatment
- Voltage

Identifying the right beads for EPD requires testing
Control of EPD layer thickness

Spherotech beads (710 nm) deposit: thickness over deposition time

Good control of EPD layer thickness by linear growth rate
Prevention of crack formation during drying

Ambient drying:
ST/Bang beads after EPD (no ALD coating)
Dried in air (3 hours)

Slow drying/freeze drying:
ST beads after EPD (no ALD coating)
Dried in ethanol vapor (1 week)

Cracks can be avoided by carefully controlling the drying conditions (slow drying or freeze drying)
Surface roughness

Ra of ~1.8 um over mm² has been achieved for a 140 micron thick EPD film
PS beads: carboxylated 15c Al\textsubscript{2}O\textsubscript{3} after burnout
PS beads: not functionalized
15c Al$_2$O$_3$ after burnout
### ALD coating on different bead types
Mass gain analysis on bulk pieces after 15 c Al₂O₃ @ 90C (expected ~3 nm)

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ALD growth rate depends on the type of beads
(resulting foam density varies by a factor of 1.5 to 3.5 for same ALD conditions)
ALD on polymers: diffuse or abrupt interface?

Polyscience beads, non functionalized

Density: 157 mg/cm³ (~3x higher than expected) caused by subsurface deposition

“on surface”

“sub surface”

Mass gain analysis
15c Al₂O₃ @ 90C (expected: ~3nm)

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Reduce density by using larger beads
20c Al₂O₃ @ 90C (expected: ~4 nm)

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<td>18 nm</td>
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For 15c Al₂O₃ on 6.8 um beads: 22 mg/cm³
Burning leads to PS removal but also large crack formation

ST beads 6.8 um, 20c Al$_2$O$_3$, 450C/air/2h, residual PS mass 1.6%

- large cracks
- Some beads pop open,
- Some have “vent holes”
- Some beads “shrivel”
Air plasma drastically reduces cracks, but no complete PS removal (even after 72 h)

ST beads 6.8 um, 20c Al₂O₃, residual PS mass 16%

- Only minor cracks
- Some beads are not etched or only partially etched
- Beads at cracks are completely etched

Graph: Percent of PS Plasma Etched over Time (20c ALD)
Complete PS removal for thinner ALD coatings

ST beads 6.8 μ, **15c Al₂O₃**, air plasma for 48 hours, 1.3 % of PS mass left

Plasma etching efficiently removes PS without major crack formation

- no visible PS cores left
- Minor cracks (< 10 micron)
Cylindrical geometry
EPD successfully transferred to cylindrical geometry

Current Challenges:
• Inhomogeneous coating
• Side effects from inhomogeneous field
• Delamination of PS coating

3D printed sample holder
Polystyrene Suspension

Stainless steel tube with counter electrode

~50 micron thick PS bead deposit on inside of SS cylinder

Work in progress:
Demonstrated flat PS bead deposits by EPD on Au electrodes, 200 microns thick, without cracks

Templated PS films with ALD

Successfully removed the PS core with only minor crack formation

Achieved a Al$_2$O$_3$ foam density of $\sim$20 mg/cm$^3$

Working on optimization of 3D setup

Need: PS bead synthesis capability in-house