Ultra-low Cost, Lightweight, Molded, Chalcogenide Glass-Silicon Oxycarbide Mirror Components

November 2015

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Talk Outline

- Project Background
- PDC(Polymer-Derived-Ceramics) Significance- X-MAT™ Development
- Large Mirror Blank Development
- Chalcogenide Sealing/Coating Development
- Conclusions

Who is Semplastics?

- 15 year old company focused on high performance plastics in electronics
- Recent development activities in novel high performance materials- X-MAT™
- US patent #8,961,840 issued 2/24/2015- Multiple Patents Pending
- Phase I NASA SBIR for lightweight optical mirrors granted in May 2015

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What are the Main Goals of the Project?

- Reduce areal costs to less than $500K/m² for UV/Optics and less than $100K/m² for IR systems
- Reduce the weight of mirror substrate through molding lightweighted structures using lighter X-MAT™ materials (SiOC)
- Make a High Performance Mirror Component that can meet NASA's requirements

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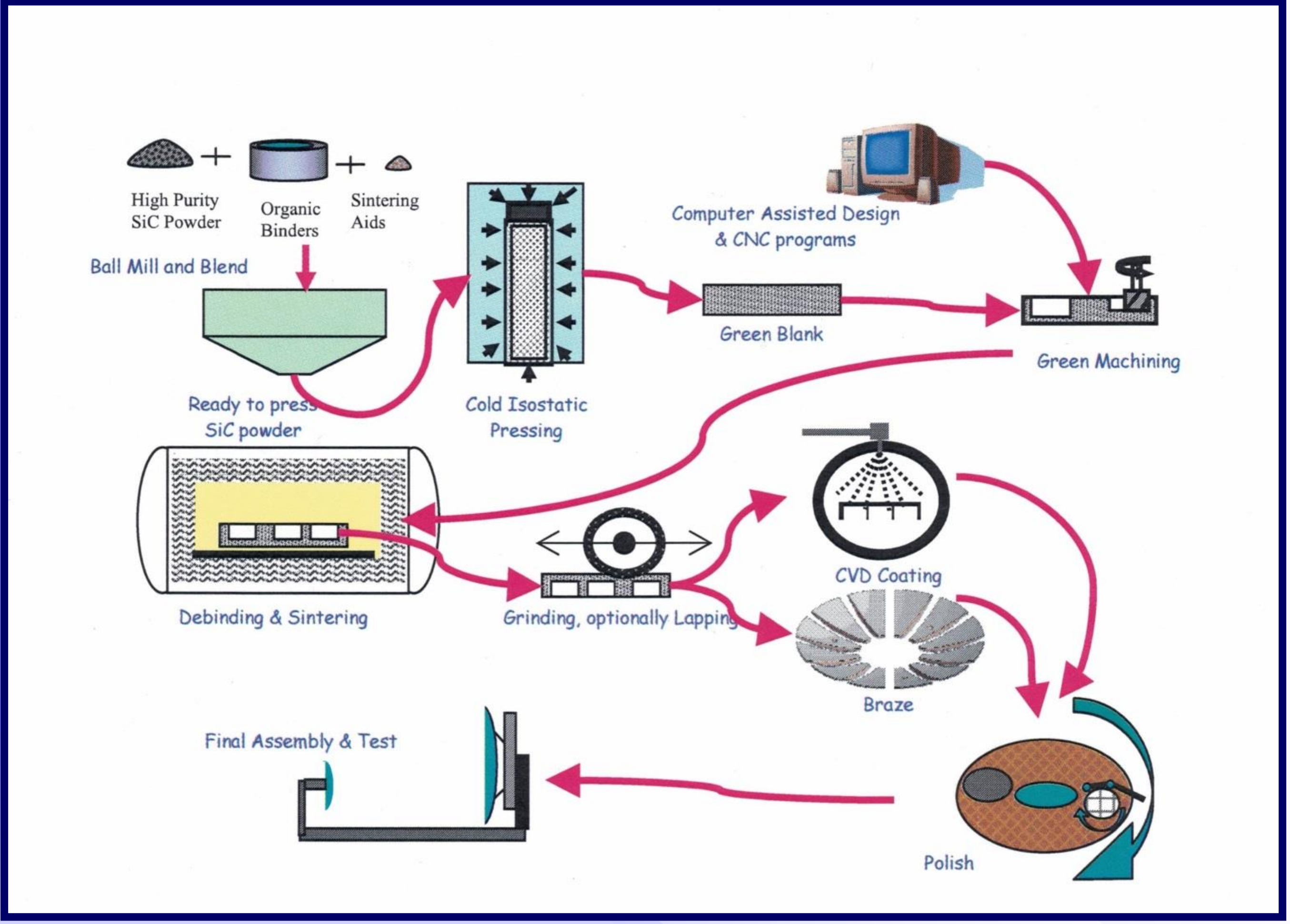
Advantages of X-MAT™ OC1

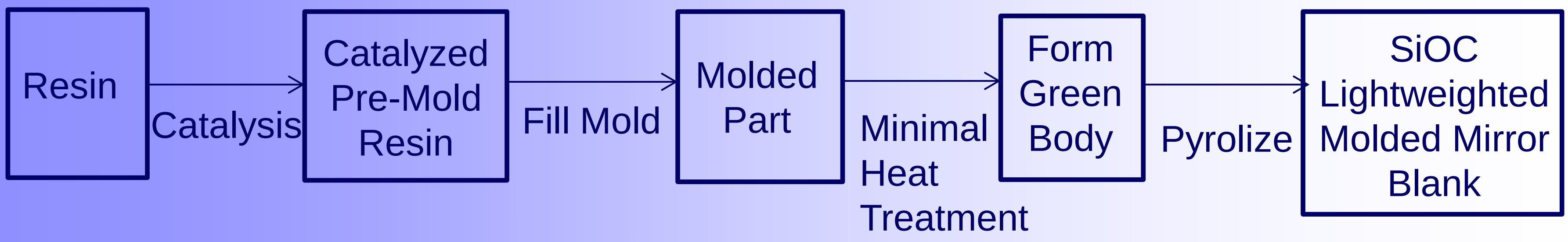
- Lightweight- 1.69 g/cc (SiC- 3.1 g/cc)
- High Temperature Range- -150C – 1100C Continuous Usage
- Low Coefficient of Thermal Expansion- 0.60-1.27 x10E-6 in/in C (-150C - 300C)- Similar to Quartz
- Amorphous structure provides isotropic properties
- Very Green technology- Uses 20X less energy than typical SiC manufacturing processes!!

SiC Manufacturing Process*



***Overview of the production of sintered SiC Optics and optical sub-assemblies**, S. Williams, CoorsTek, Inc.; P. Deny, BOOSTEC Industries (France) [5868-04]

X-MAT™ Mirror Blank Process



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So What is the Big Deal with X-MAT™?

- Polymer resin instead of ceramic powders
- Typical Plastic Processes (molding, etc.) Available
- Shorter Manufacturing Intervals
- Chemical Bonding of the Materials rather than Sintering (Significantly Lower Energy)
- Tailored Material System Properties
- 3D Systems Rather than Traditional 2D(Sheet) PDC Systems

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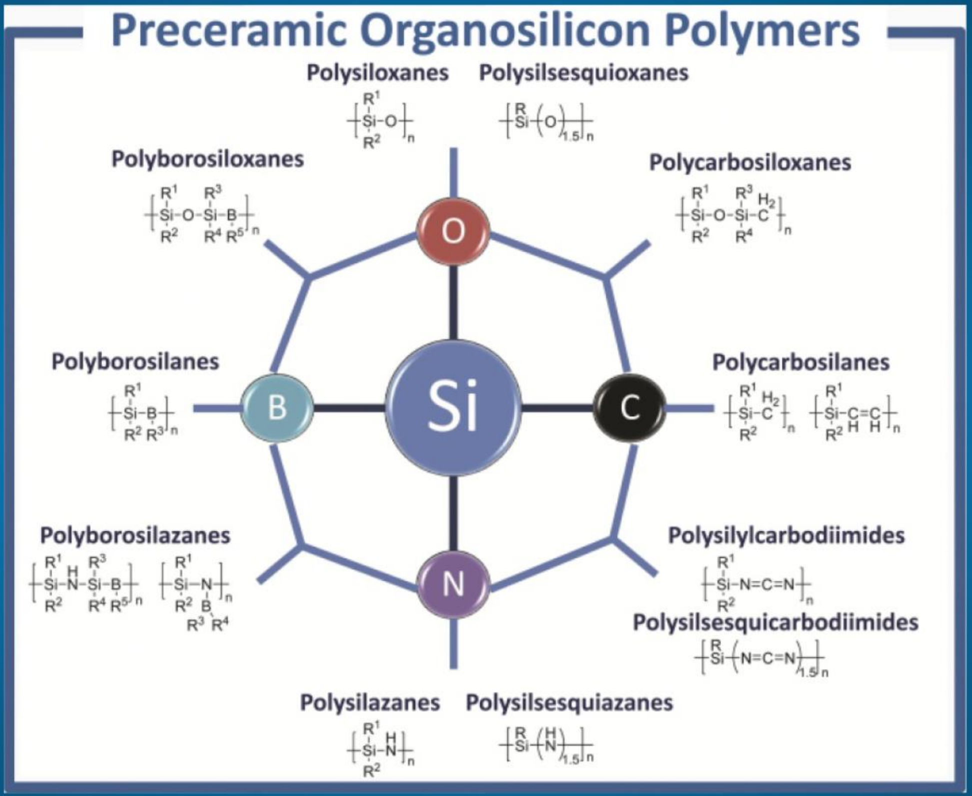
Significance/Review of Polymer-Derived Ceramics (PDCs)

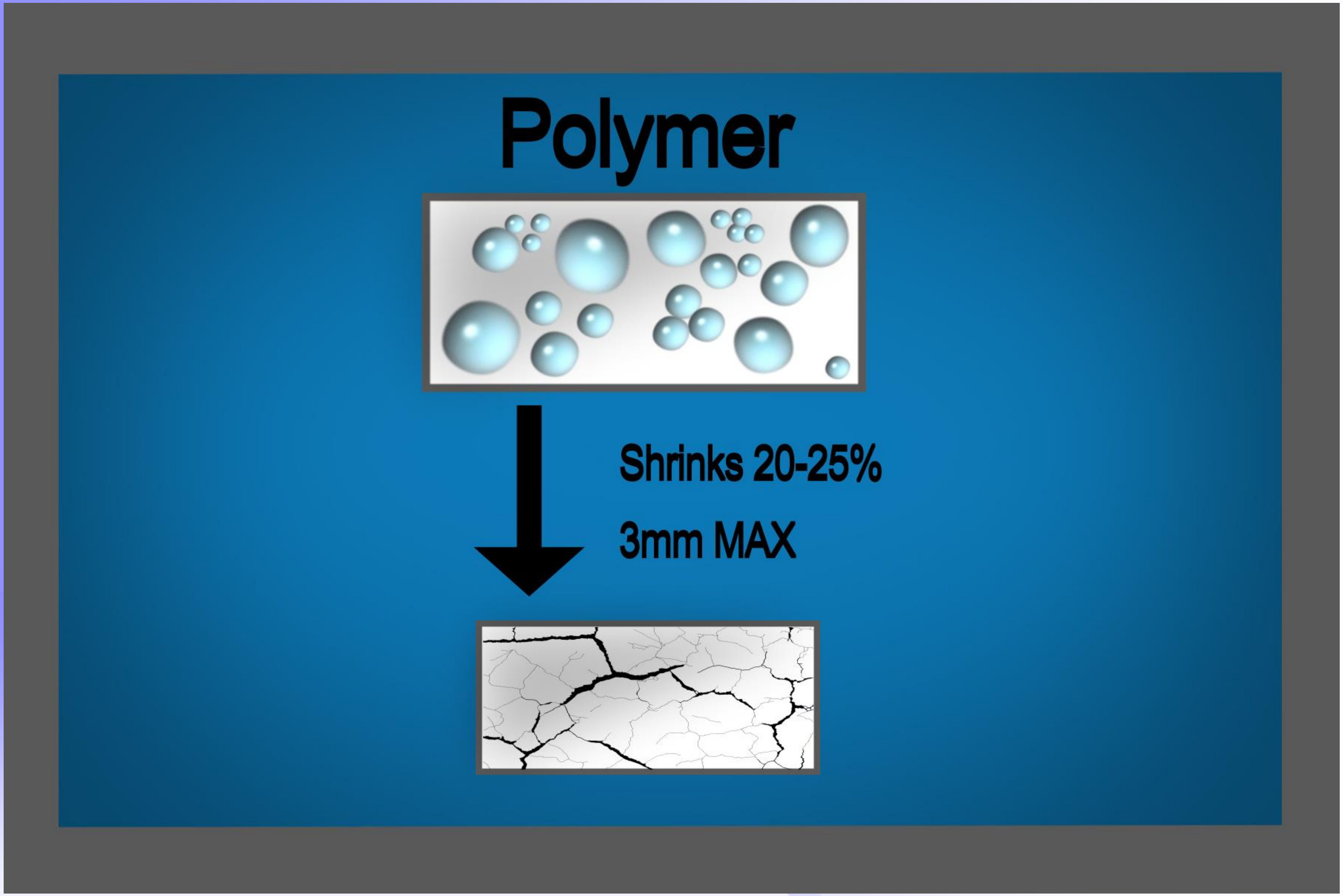
- 40 year history of PDC Development activities
- Commercially Available Resins
- Current commercial usage limited to ceramic fibers, polymer coatings and thin ceramic films
- Multiple resin types and processes produce unique ceramic types and properties

PDC Technologies



J. Am. Ceram. Soc. 93 [7] p.1807 (2010)

Polymer to Ceramic Processing



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Current PDC Limitations

Can only produce thin films or fibers due to cracking and degradation of films thicker than several hundred microns

“ The polymer to ceramic conversion occurs with gas release which typically leads to cracks or pores which make the direct conversion of a preceramic part to a dense ceramic **virtually unachievable**, unless its dimension is typically below a few hundred micrometers(as in the case of fibers, coatings, or foams.) J. Am. Ceram. Soc. 93 [7] p.1811 (2010)

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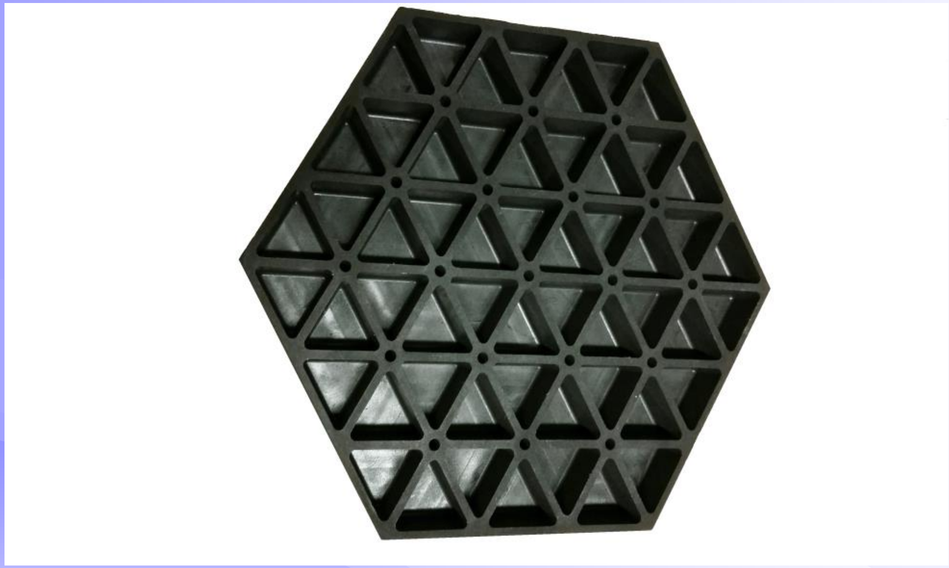
“Virtually Unachievable” Largest Bulk PDC Ever Made (No Fibers!)



Mirror Blank Deliverable: 0.25 meters

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Properties of X-MAT™ OC1

TEST	VALUE	UNITS
Fracture Toughness	.96	Mpa-m^1/2
Flexural Strength	43.5	Mpa
CTE	0.75	1E-6in/in˚C
Young's Modulus	56	Gpa
Poisson's Ratio	.53	-
Density	1.69	g/cc

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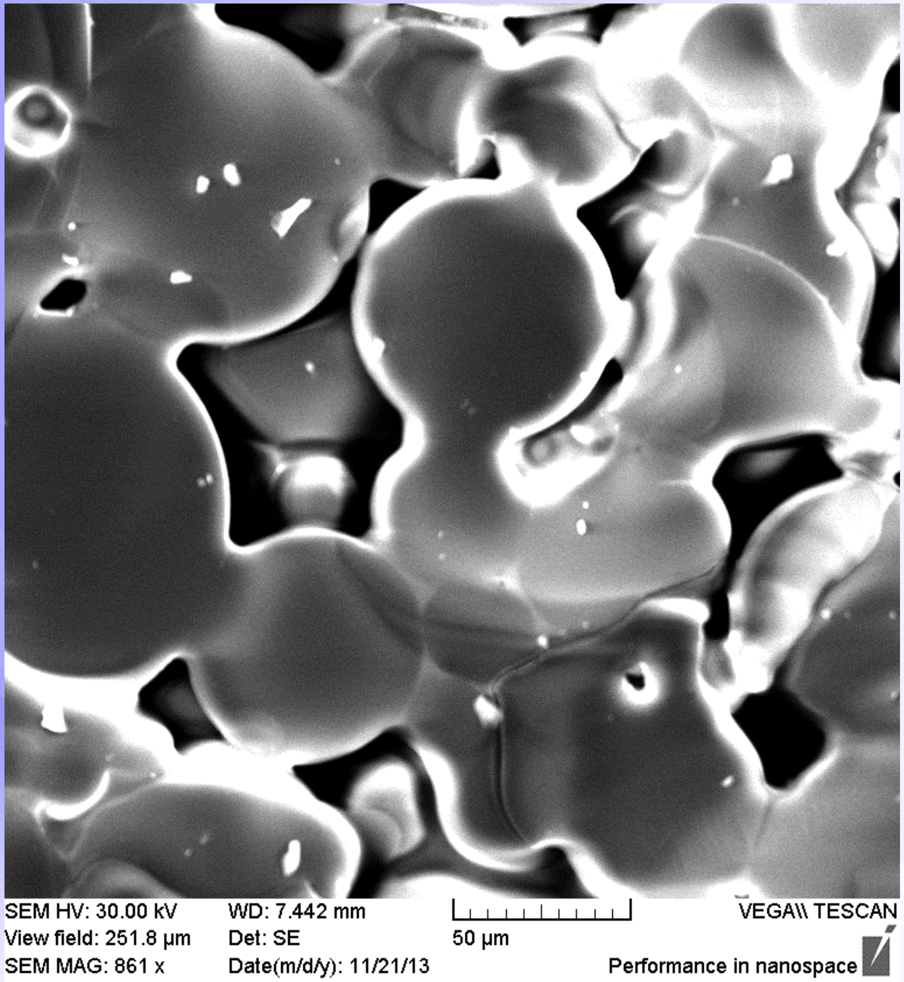
SEM of X-MAT™ OC1



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Chalcogenide Sealing/Coating Development Activities

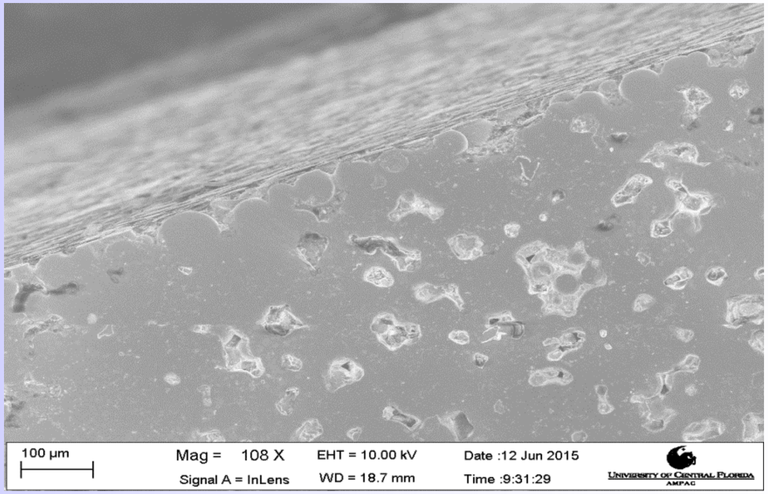
Design Matrix

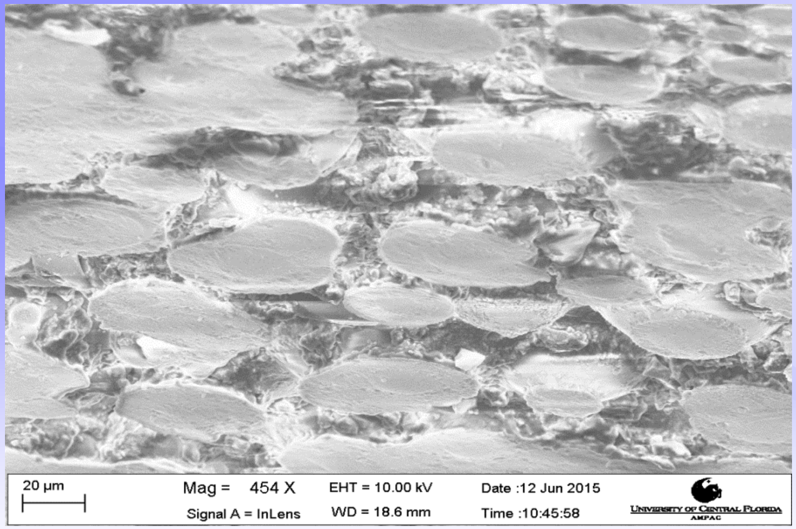
			Zygo Imaging	SEM		Thermal Cycle	
Coating Type	Coatings #	Progress	RMS Roughness (µm)	Film Thickness (µm)	Penetration Depth (µm)	Temperature Change (°C)	Damage
Uncoated							
Uncoated	0	×	12.159				
Spin Coating							
0.4 mol	1						
	2	✓	7.579			25-150	×
	4	✓	5.927			25-150	×
0.6 mol	1						
	2	✓	8.460			25-150	×
	4	✓	6.022			25-150	×
0.8 mol	1	✓	8.0214			25-150	×
	2	✓	6.371			25-150	×
	4	✓	4.390				×
Dip Coating							
0.4 mol	1						
	2	✓	6.435			25-150	✓
	4	✓	8.385			25-150	✓
0.6 mol	1						
	2	✓	9.369			25-150	✓
	4	✓	8.301			25-150	✓
0.8 mol	1	✓	9.581			25-150	×
	2	✓	19.560			25-150	✓
	4	✓	23.464			25-150	✓

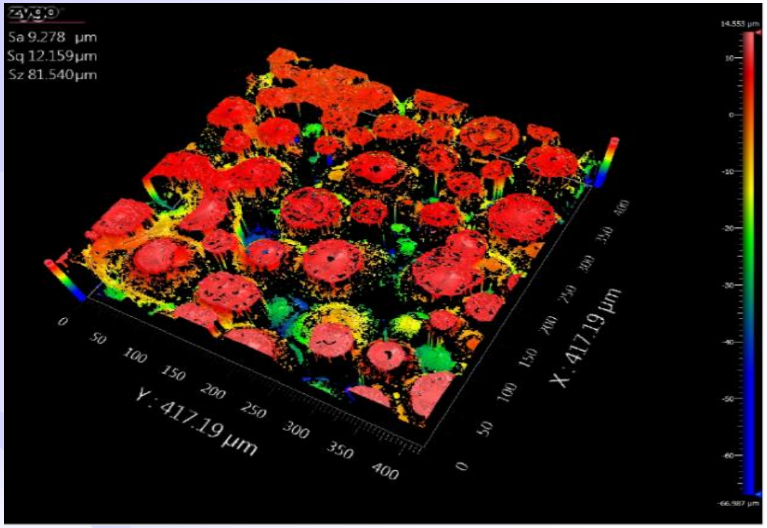
SiOC PDC Uncoated Substrate

Uncoated SiOC PDC

- Highly porous
  - ~80% dense
- Highly Rough Surface
  - RMS roughness of ~12 µm





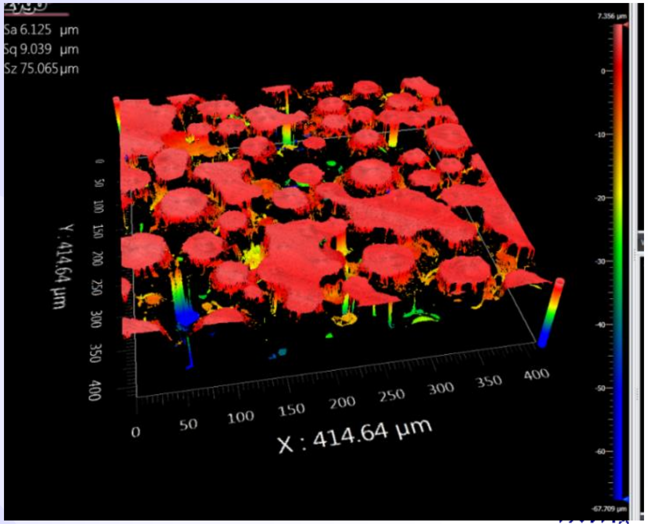


As_2Se_3/PDC Experimental Parameters

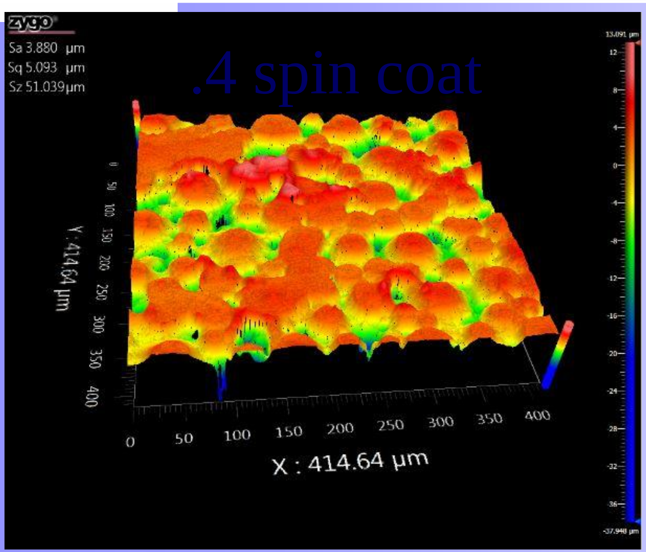
- **Solutions:** made by dissolving powdered As_2Se_3 in Ethylenediamine.
- **2x coats:** Deposit onto PDC in nitrogen atmosphere glovebox, anneal at 75 °C for ~24 hours after each deposition. Bake at 150 °C for ~15 hours after all layers applied.
- **4x coats:** Deposit onto PDC in nitrogen atmosphere glovebox, anneal at 75°C for ~6 hours and bake at 150 °C after each deposition for ~15 hours.
- **Thermal reflow:** After 4 coats were applied, put in oven again at 240 °C for 30 min to reflow the deposited glass.

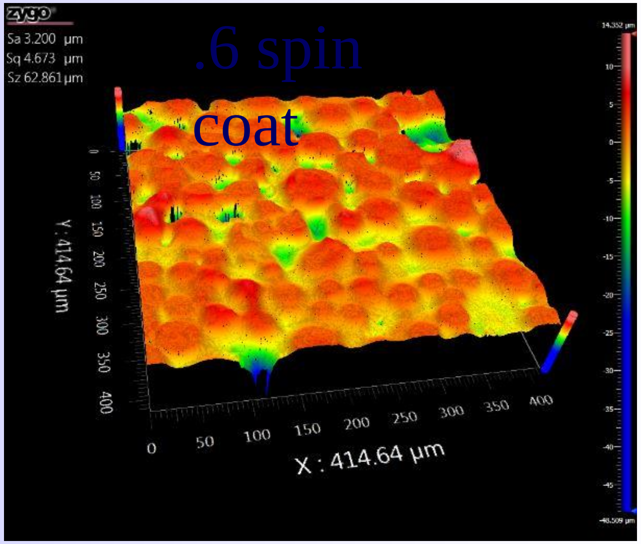
Starting substrate (PDC):

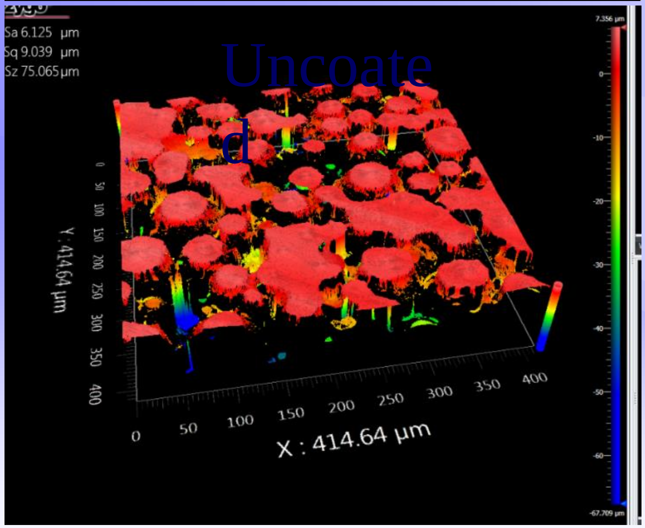
Avg RMS Roughness: >10.129 µm (≈ 12 µm)
Target RMS roughness: ~5nm

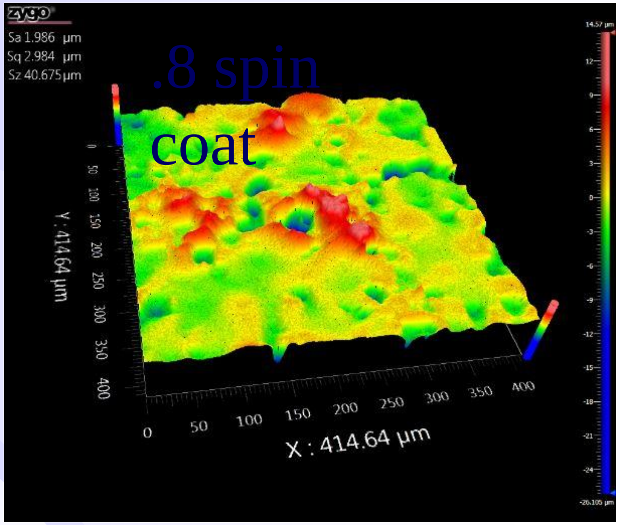


As_2Se_3 4x Spin Coating (Zygo)

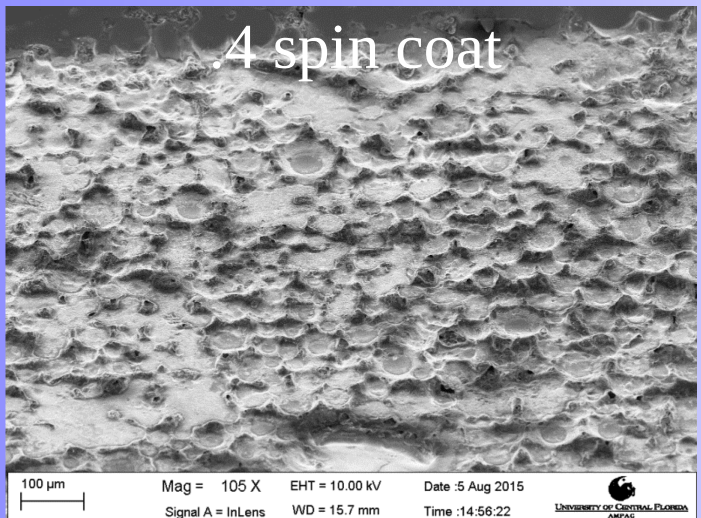


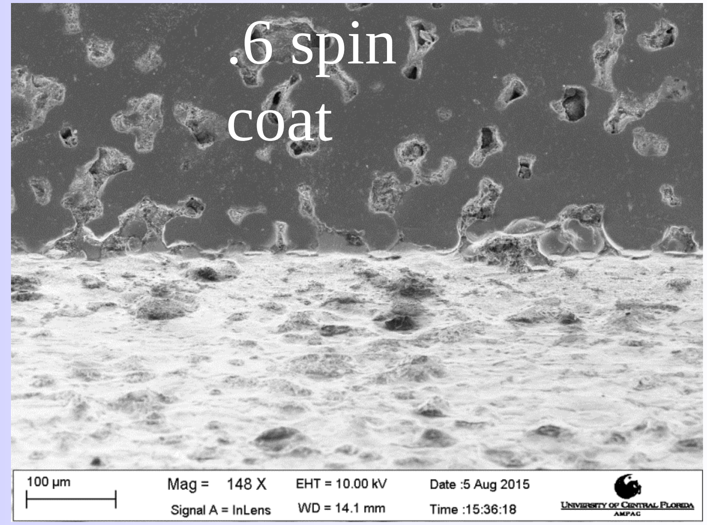


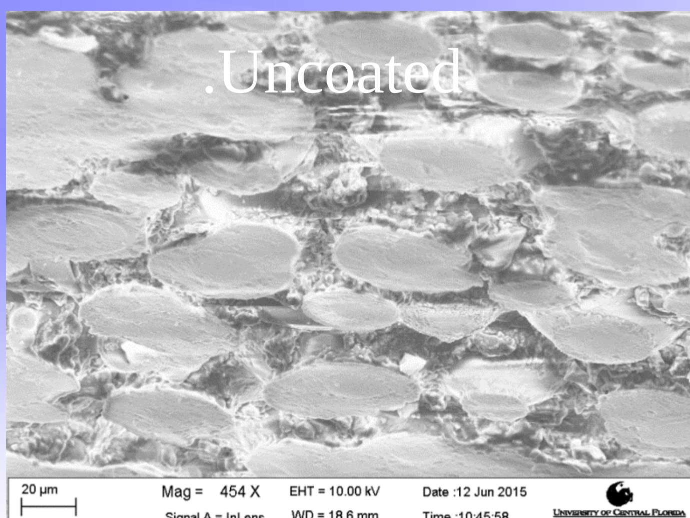


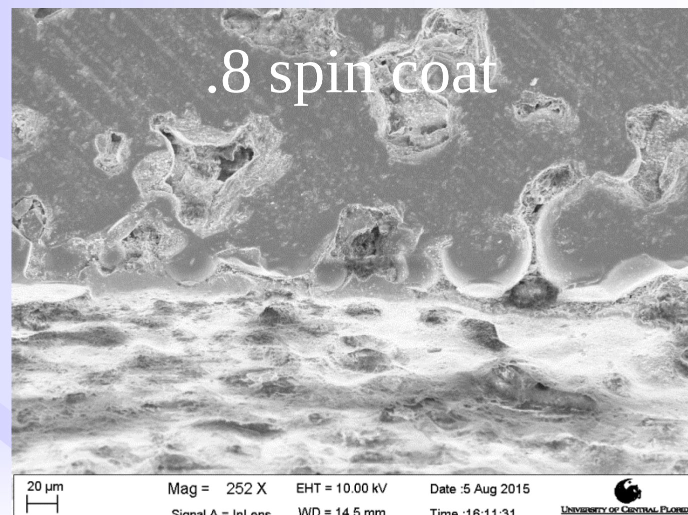


As_2Se_3 4x Spin Coating (SEM)

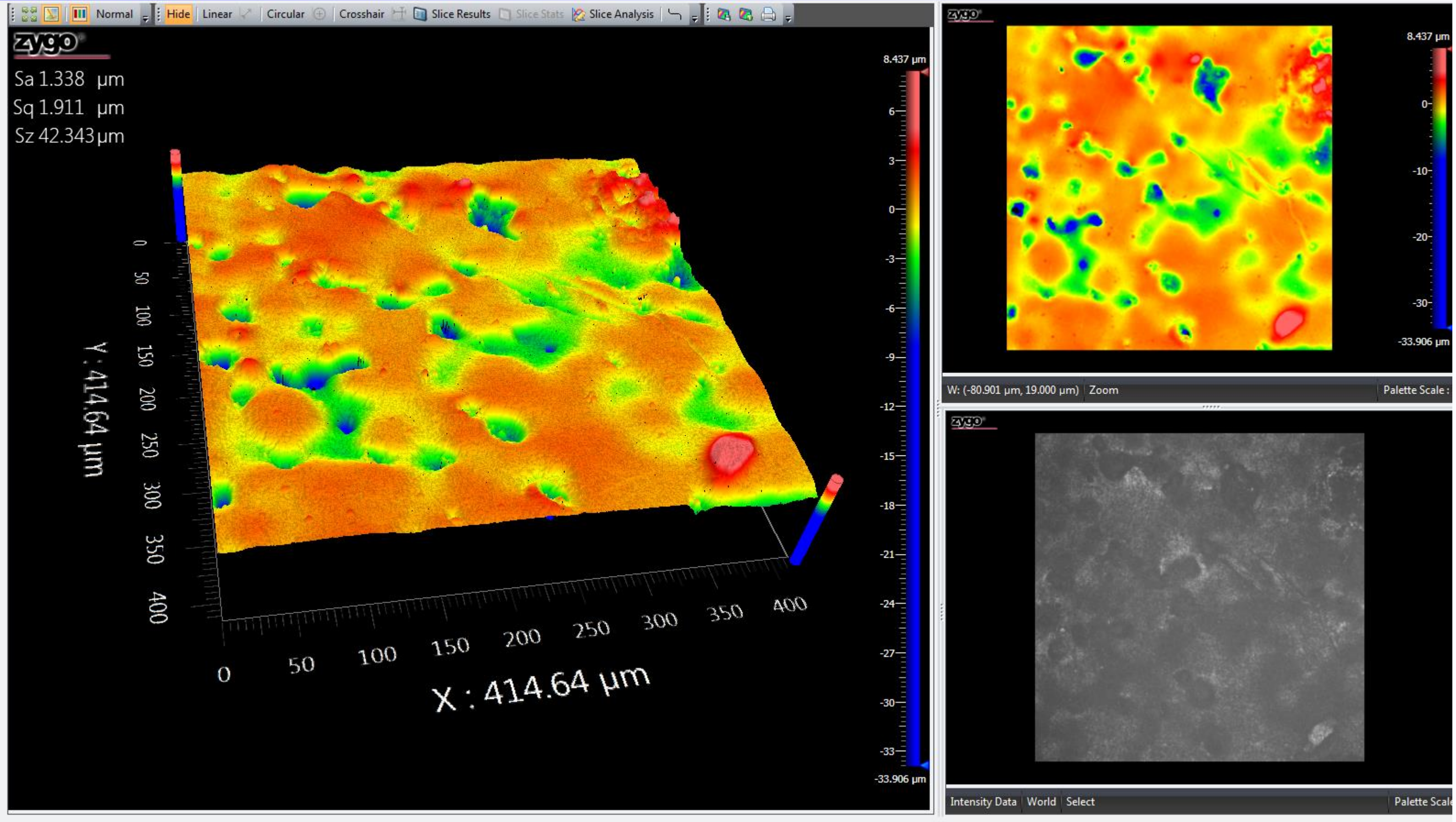








0.8 g/mL Spin Coat After Reflow



Roughness (Not Averaged): 1.911 µm

Coating Conclusions

- **Spin coating** shows better reduction in RMS roughness for all solutions and all coating layers.
  - 1x & 2x coatings of 0.4, 0.6, & 0.8 mol solution do not lead to complete coverage and pore sealing
  - 4x coatings of 0.6 & 0.8 mol solutions lead to complete coverage and sealing with significantly reduced roughness
    - 0.8 mol 4x spin coat has been down selected for future work.

- **Dip coating** shows better coverage and pore sealing for all solutions and all coating layers.
  - Except for 1x dip coatings, gas evolution through thick uneven coating layer resulting in damage to on the surface.
  - Successive dip coatings leads to increased RMS roughness

- First round of **thermal reflow** conducted at 240°C for 30min
  - Most samples show reduction in RMS roughness

Future Work

- A matrix of thermal reflow experiments will be outlined and experiments conducted with varying temperatures and times for 0.8 mol 4x spin coatings.
- A new series of dip coatings with thermal reflow after each coating will be performed with 0.8 mol solutions
- Once average roughness has been reduced below the 1 μm mark
  - work will be started on coating whole disks .
  - Cryo cycling experiments will temperatures ranging from -50 to 50°C

Internal R&D Sealing and Coating Effort-Breaking News!

- Low CTE Coating- Negligible CTE Mismatch with X-MAT™ OC1
- Seals Porous Surface
- Easily Integrated into Manufacturing Flow
- Easily Polished
- Excellent Aluminum Adhesion Properties

Coated X-MAT™ Sample(no metal)

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Conclusions

- Largest Free Standing Bulk PDC Component Ever Produced- 0.25M Mirror Blank- 3D X-MAT^{TM} OC1 Material System
- Sealing/Coating Systems are being developed
- On Track for Phase 1 Deliverables
- Making a 1.0M Diameter Mirror is the next step in the Technology Development Progression

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Acknowledgements

NASA- Ron Eng, Phil Stahl

UCF- Kathleen Richardson, Jason Lonergan

Semplastics- Arnie Hill, Todd Hubert, Matthew Stephens, Barbara Hopkins

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