High Surface Area (HAS) /High Aspect Ratio (HAR) ALD Process Optimization Using Anodic Aluminum Oxide

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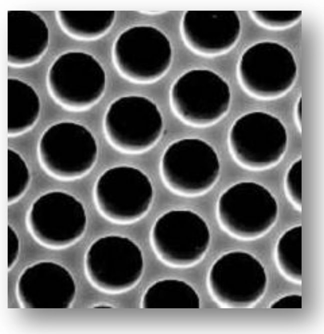
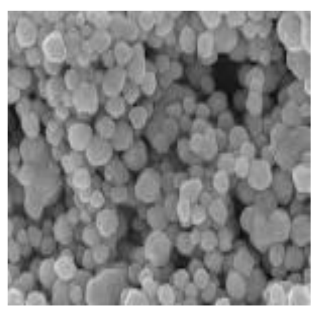
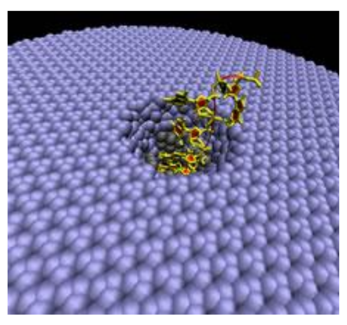
HAS/HAR ALD Overview



Microchannel plate (MCP)
pore diameter ranges from 5-10 um in with an AR of >50:1

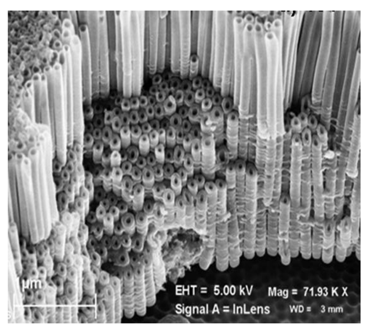


Nanoparticles (NP)
Surface area ranges from 1-100 m^2 per gram with AR of >50:1

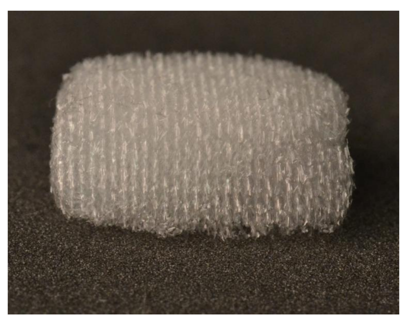


Nanopore
Pore diameter can be as low as 2 nm with AR up to 10

Aerogel
Surface area ranges from 20 -100 m^2 per gram with AR upto 1200:1



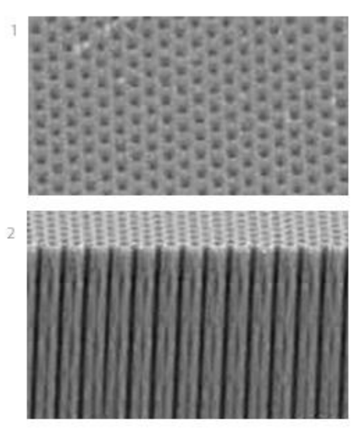
TiO_2 nanotube membrane
pore diameter of 150 nm um in with an AR of 2000:1



Microlattice
pore diameter ranges from 100 to 400 um in with an AR up to 100:1

More and more devices incorporate high surface area (HAS)and high aspect ratio (HAR) components into their device structures such as microchannel plate (MCP) for night vision goggle, nanopores for DNA sequencing, nanoparticles for fuel cell, etc. while at the same time these components require functionalization by atomic layer deposition. The benefits of ALD functionalization include fine tuning the properties over very large area; very conformal coating , and defect free. Optimizing ALD conformality in these HAS/HAR structures is challenging due to the cost of the substrates. Here we present HAS/HAR ALD process optimization using anodic alumina oxide (AAO).

AAO as HAS/HAR ALD Process Optimization Tool:



Commercial available anodic aluminum oxides (AAOs) have diverse applications in prevention of corrosion of metal substrates from their service environment, forming capacitor dielectrics, templating nanomaterials and in many other fields such as catalysis, optics and electronics. AAO can be used as template for preparing various nanoparticles, nanowires and nanotubes because of the nanoporous structure. Using AAO as process optimization tool for HAS/HSA ALD is a little new. AAO possesses very high pore density (>10E8) and high aspect ratio (>300:1), which are similar to aforementioned devices such as MCP. Those 3D devices are sometimes hard to prepare and hard to analyze by electron beam imaging techniques because of the charging. So AAO will be a good alternate testing vehicle for HAS/HAR ALD process optimization.

Conformal coatings are becoming increasingly important as various industries move towards the nanoscale technology. The exceptional atomic level thickness control and conformality of ALD made it the process of choice for numerous applications found in microelectronics and nanotechnology. Process optimization for 3D high aspect ratio nanopores is usually difficult as process parameters, such as dosage, purging, temperature and pressure are often interdependent with one another. Therefore, processes must be optimized to achieve self-limiting saturated growth and avoid parasitic CVD-like reactions in order to maintain thickness control and conformality at the atomic level while preserving the desired materials' properties (electrical, optical, compositional, etc.). This work investigates AAO as a versatile platform to optimize ALD process when transitioning from a planar system to a high aspect ratio nanopore using a cross-flow wafer-scale reactor.

Experimental Method: GEMStar-8 ALD Equipment

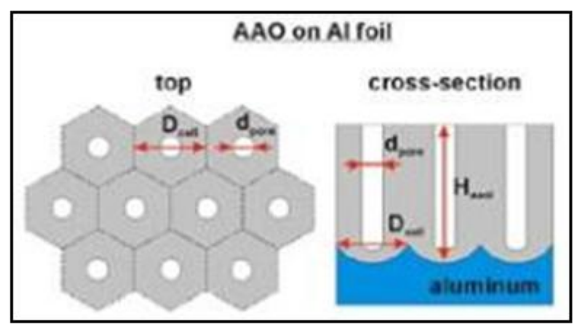
GEMStar-8 system is designed for extreme surface area, high aspect ratio structures: Multi-channel precursor delivery system isolates & distributes precursors combine with a tapered exhaust to provide exceptional nanofilm uniformity.

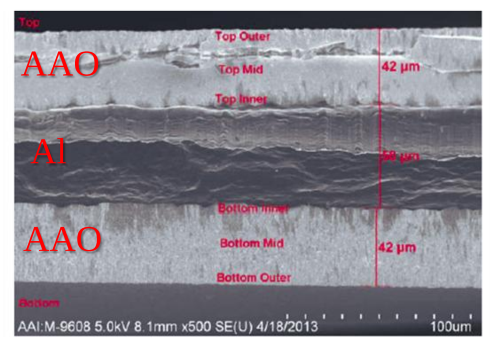
The differentially pumped system seals eliminate gas permeation which along with separate and actively heated Oxidant and Metal-Organic manifolds eliminate parasitic nanofilm production.

Metrology Interface for QCM, ellipsometry , FTIR, OES and room for up to six high capacity precursor cylinders (up to 4 heated) with 2 independent gas lines, maximizes system productivity.

Experiment

Surface Area Surrogate: AAO
- 2- sided; AR = 420:1
- 50nm diameter pores
- 251 nm pore pitch
- 42µm pore thickness
- surface area: 2.5 m^2





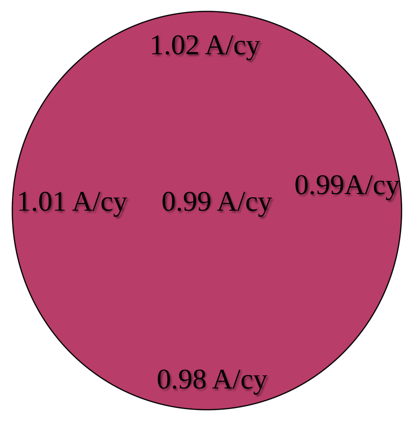
❖ **Two pieces of AAO with aspect ratio (AR) 300:1 = one MCP (8”X8”) of AR of 60:1 in terms of surface area.**
❖ **The uniformity and growth rate of the process was gauged by 8” Si wafer.**
❖ **The SEM image of AAO was used to see the conformality.**
❖ **We generally optimized the process parameters such as dosage, purging, pressure to meet a non-uniformity less than 5% and conformality greater than 80% before running real 3D pore samples and devices made of nanoparticles.**

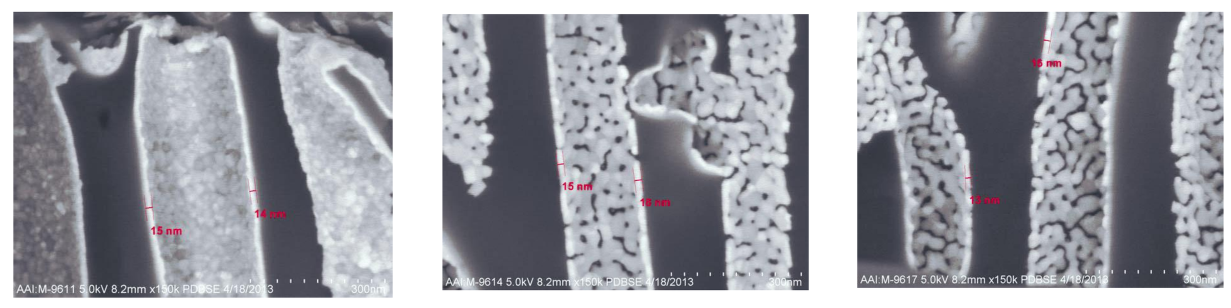
Results

❖ **Generally large surface samples require:**
- **more doses of precursor to saturate the surface**
- **more time for precursor to penetrate into nanopores**
- **longer time to purge away the unreacted precursors.**

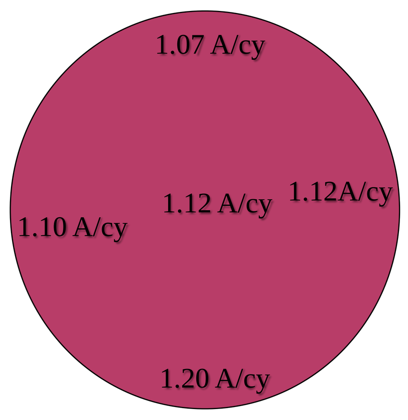
❖ **GEMStar 8 tool and GEMFlow software can allow researchers to easily change ▪ doses, ▪ pulsing, ▪ residence time, ▪ purging time.**

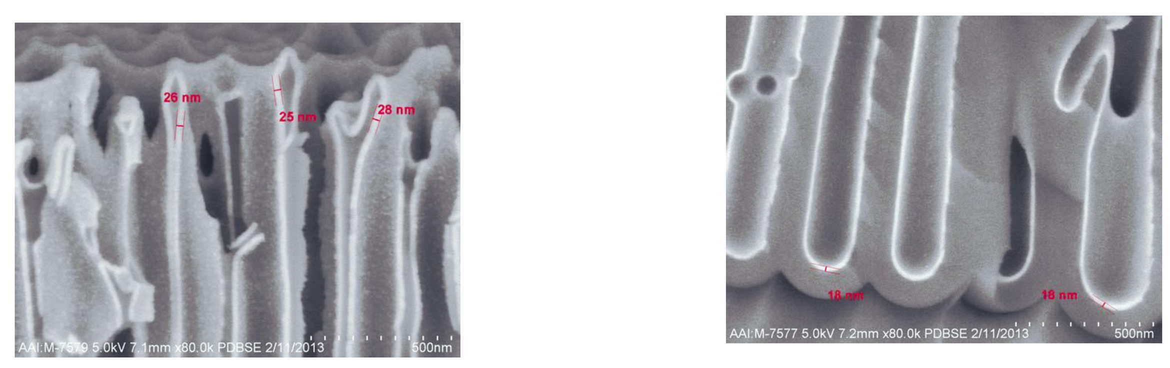
❖ **Some typical films developed for 3D structures on GEMStar 8”:, Pt, Yttria stabilized zirconia (YSZ), ALD nanofilms**



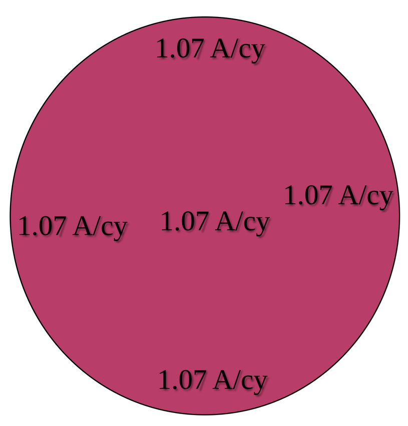


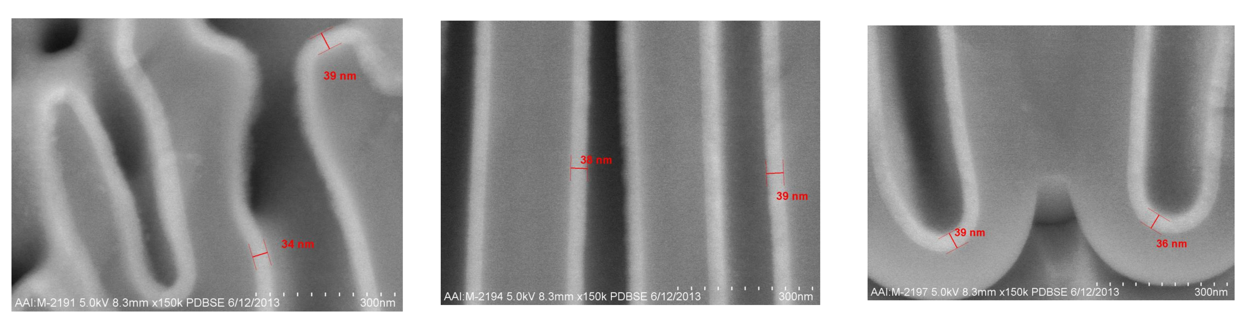
High penetration Pt ALD :1.0 A/cycle GR, 2% unif, >90% conformality in AAO



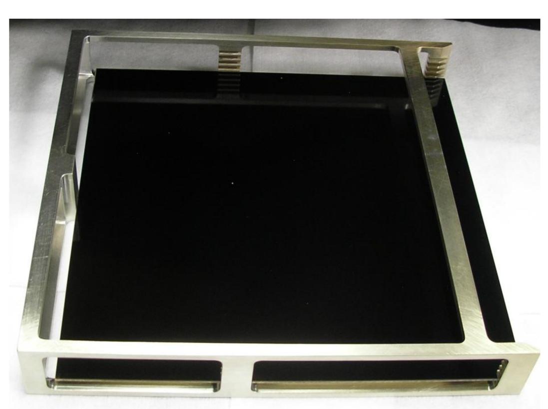


High penetration YSZ ALD :1.1A/cycle GR, 5% unif, 75% conformality in AAO

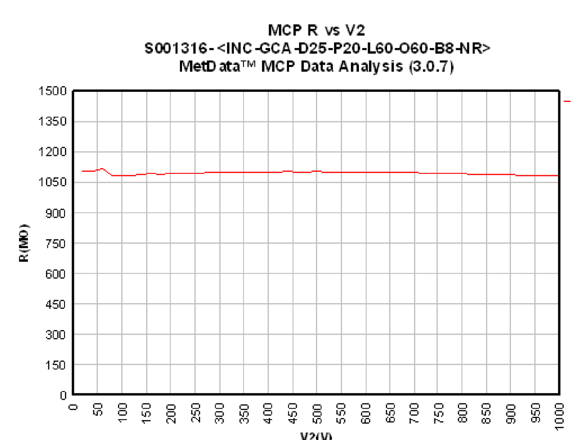




High penetration ALD nanofilms for MCP :
2% unif, ~100% conformality in AAO



Fully processed 8” MCP



1 GΩ R of Pilot MCP Processed alongside 8” MCP
Provides R of 10 MΩ for 8” equivalent resistance

Summary

To realize ALD on some 3D pore substrates and nanoparticle samples, AAO was used as an alternate process optimization tool to gauge the effect of surface area and high aspect ratio. It was found that more doses of precursor, residence time and purging were required to have a true ALD. In this way we have developed suitable YSZ, Pt, ALD nanofilms for those 3D devices to have good film uniformity across a 200mm substrate and good conformality over 400:1 in the extreme surface area and aspect ratio environment.

Acknowledgements

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