



ARRADIANCE[®]

Scale-up: the Arradiance 8" ALD Tool

Presented to PSEC Collaboration Meeting
December 9, 2011
Neal Sullivan



Outline

- ◀ Project overview
 - ◀ DE-SC0004193 - PH II - Efficient Manufacture of Extreme Surface Area Microchannel Plate
- ◀ SBIR Phase I results summary
- ◀ SBIR Phase II
 - ◀ Objectives
 - ◀ Gantt
 - ◀ Milestones
- ◀ Arradiance ALD-MCP equipment development overview
- ◀ **GEMSTAR-8™** overview
 - ◀ Key design features
 - ◀ Standard features overview
 - ◀ Material handling options
 - ◀ Interface options
 - ◀ Software overview
- ◀ Summary
- ◀ Appendix – Product specifications



DE-SC0004193 - PH II

Efficient Manufacture of Extreme Surface Area MCP

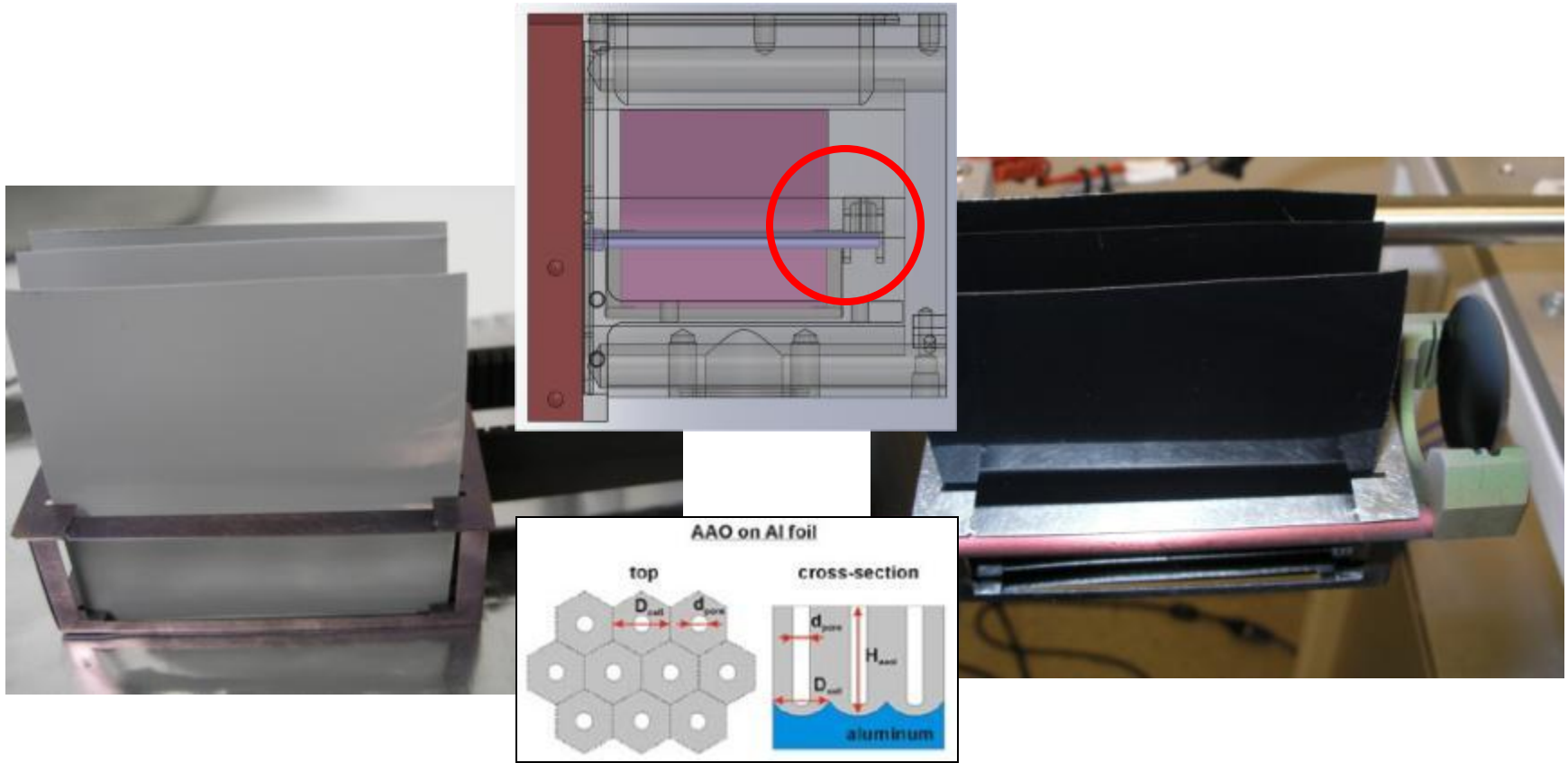
Project team:

- Arradiance (PI: N. Sullivan)
- Enrico Fermi Institute, University of Chicago (Prof. H. Frisch)

Project Summary

- The work proposed in this SBIR proposal is intended to follow the basic R&D effort of a consortium of national laboratories, universities, and industry, led by Argonne National Laboratory and the University of Chicago for the development of new, large area, photo-detector devices. **This project proposes to address the commercialization gap that exists between the proof-of-principle large area photo-detector (LAPD) program and the efficient manufacture of large area Microchannel plate devices using atomic layer deposition (ALD).** For programs such as the Deep Underground Science and Engineering Laboratory (DUSEL) project and other applications in high energy physics, medical discovery and diagnostics and homeland security applications this will be transformational.
- Arradiance, as the key commercial ALD component of the Argonne LAPD collaboration, will **develop productive recipes, without sacrificing MCP performance, for the LAPD device. In parallel, Arradiance will develop production equipment that can effectively and efficiently produce the large area MCP devices,** in which a single 8" square device comprises the same surface area as nearly 100 state-of-the-art 300mm integrated circuit wafers.
- The techniques required for large-scale commercial ALD production of LAPD a family of large-area robust detectors that can be tailored for a wide variety of applications for which large-area economical photon detection would be transformational. We believe that the success of this program, namely efficient coating of high surface area MCP devices, has the potential to extend far beyond this niche of ALD application and could impact other applications where ALD is used to coat extremely high surface area materials in technology areas such as: catalysis, fuel cell, energy storage and filtration.

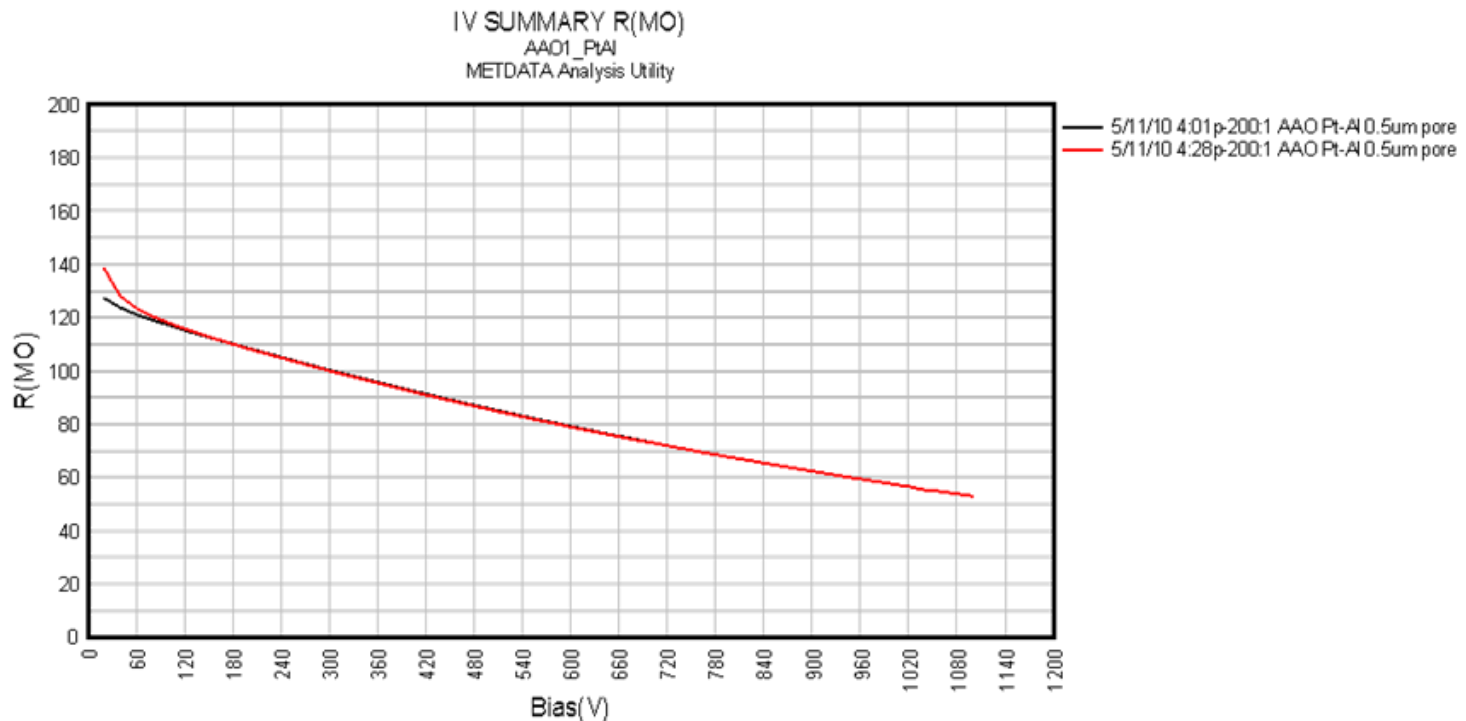
DE-SC0004193 - PH I: Experimental design



- ◀ As-received AAO plates (Synkera)
- ◀ Pore diameter = 150nm
- ◀ Pore pitch = 250nm
- ◀ Pore length = 50 μ m
- ◀ Image shows plates after deposition of resistive ($1e7 \Omega \cdot \text{cm}$) film
- ◀ Each plate has 3 m² of available surface area (or ~43 300mm wafers per plate)

DE-SC0004193 - PH I Results: ALD of Synkera Prototype AAO-MCP sample

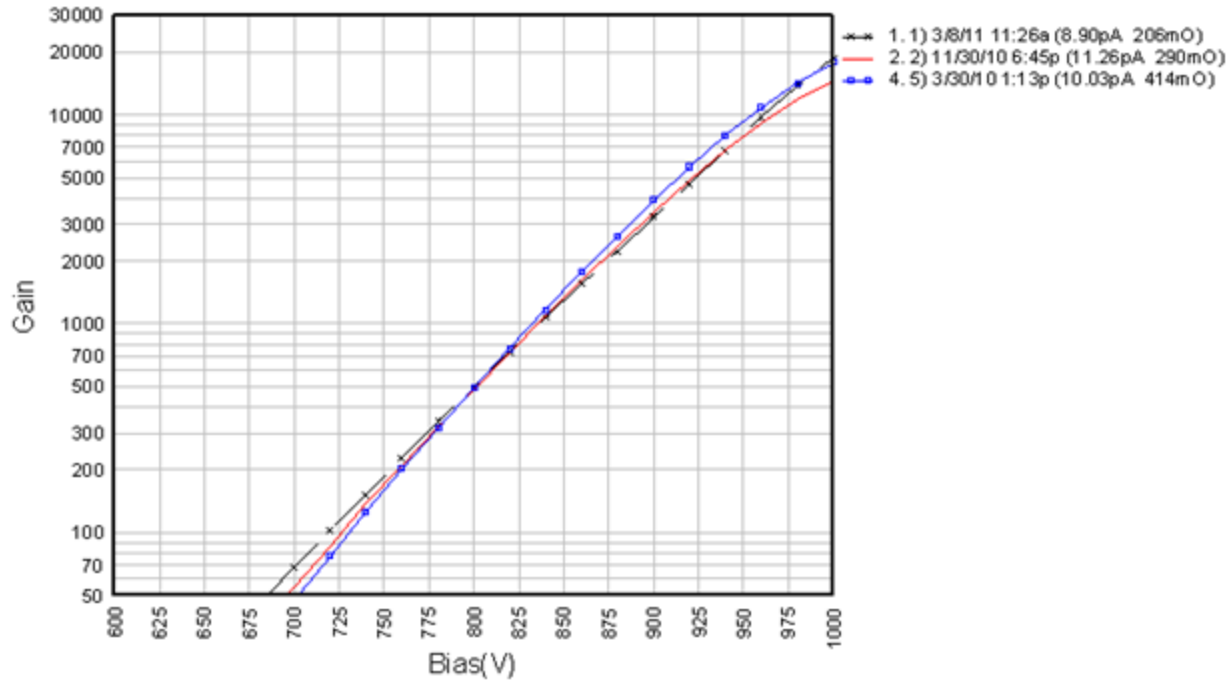
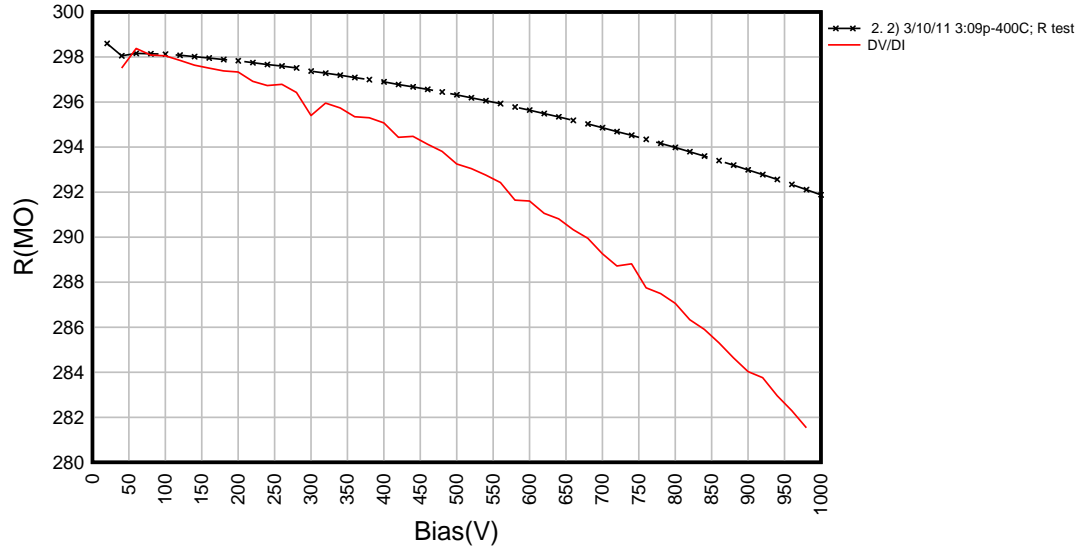
- ◆ Pore size target $\sim 500\text{nm}$, actual $\sim < 300\text{nm}$
- ◆ 100 – 200 nm NiCr electrode processing (100nm 3 sigma variation).
- ◆ Estimated MCP turn on (unity gain) voltage $> 1800\text{V}$.



DE-SC0004193 - PH I MCP test results

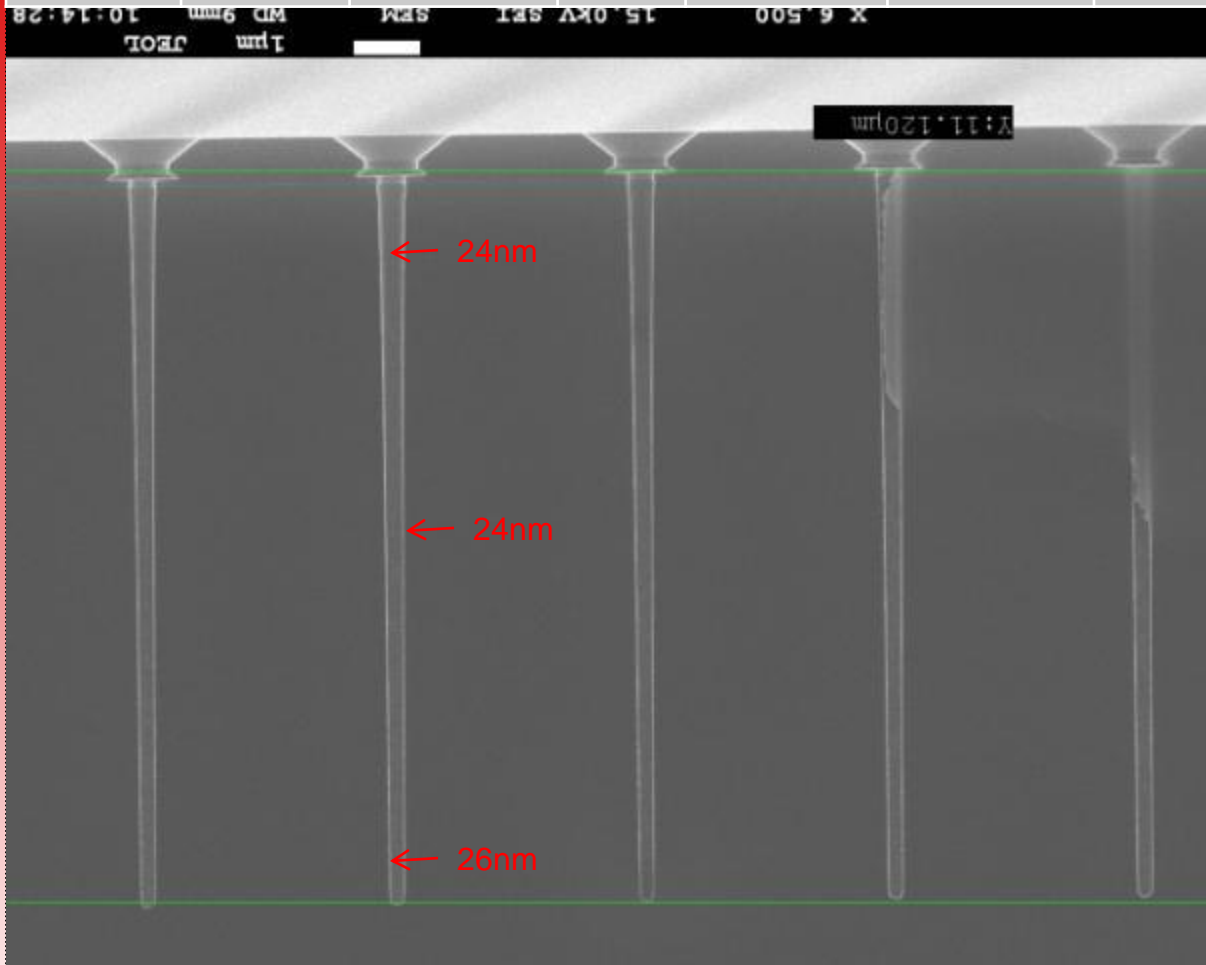
◀ Compares favorably with PSEC year 2 MCP Resistance and gain-voltage results

RV
S000943_PTAL_INCOM
MetData™ MCP Data Analysis (2.10.9)



High-Aspect Ratio Pt Deposition: Full Trench Image*

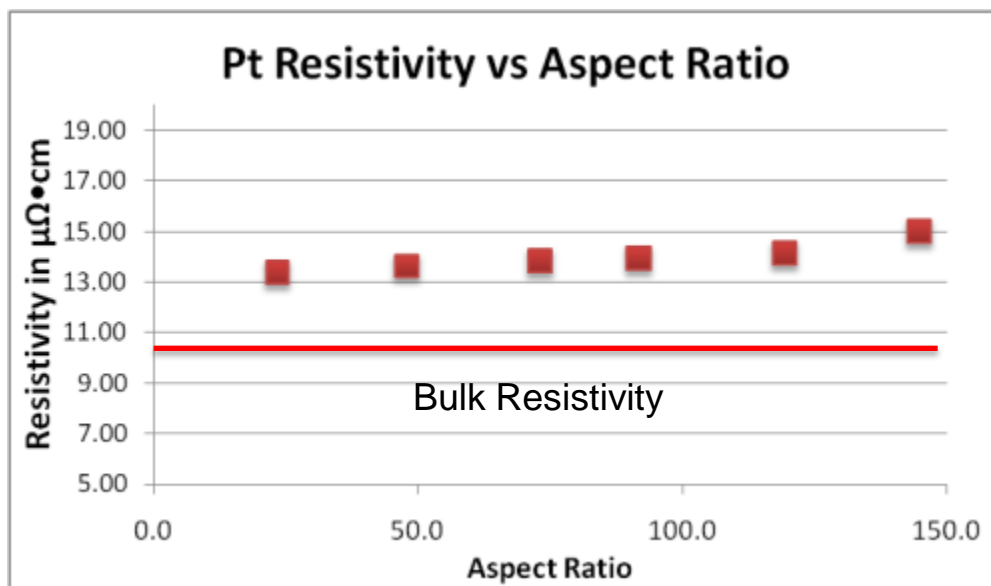
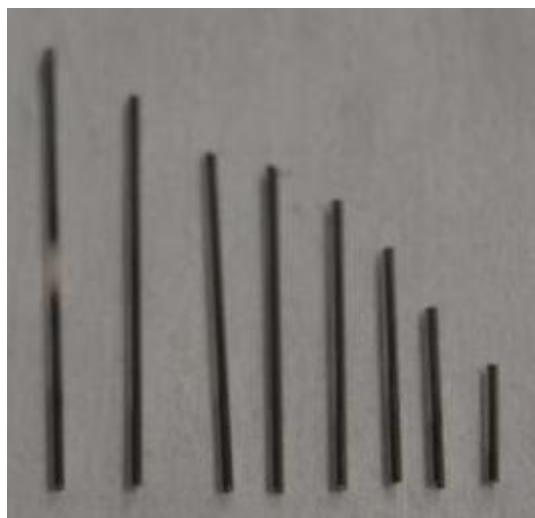
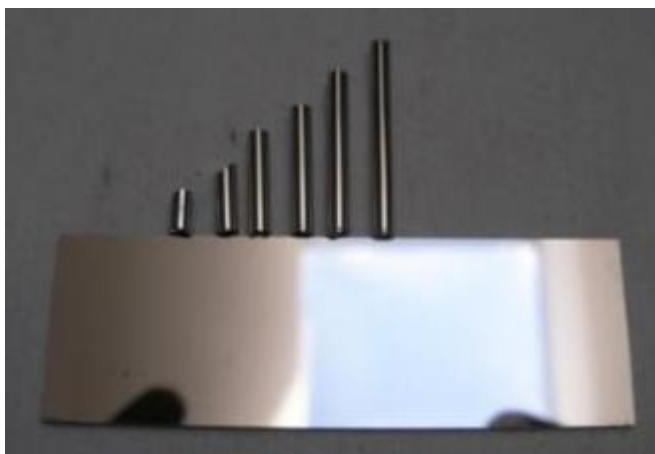
Date	Cycles	Pt Dose	# Pt	Pt Expo	Pt Purge	O2 Dose	O2 Expo	O2 Purge
3-10-11	400	250ms	x4	3 s	25 s	25 ms	1 s	30s



- ↖ 11.1 μm hole depth
- ↖ Pt film coats entire pore
- ↖ AR at center = 34
- ↖ AR at bottom = 46
- ↖ GR = 0.6 Å/cycle

High-Aspect Ratio Pt Deposition: Capillary tubes*

- ◀ 300 cycles of Pt on high aspect ratio capillary tubes



- ◀ 300 cycles of Pt on high aspect ratio capillary tubes



DE-SC0004193 - PH II First Year Objectives:

- ◀ Complete design & build of ALD chamber & Anneal furnace
- ◀ Evaluate high surface area process window for stable operating points in Exposure & dose to minimize within run and run-to-run variation. Opportunities to minimize process cycle time will also be evaluated.
- ◀ Optimize “finished” MCPs for subsequent tube processing: High temperature bake, electron scrub, tube seal & lifetime.
- ◀ Optimize all non-ALD processing for large substrates
- ◀ Transfer ALD process to new chamber, using existing (phase II improved) baseline ALD Process.
- ◀ Characterization of ALD process in 200mm chamber
- ◀ 8” plates to Argonne collaboration beginning Q2 2012 (1 plate / Qtr following)



DE-SC0004193 - PH II Gantt

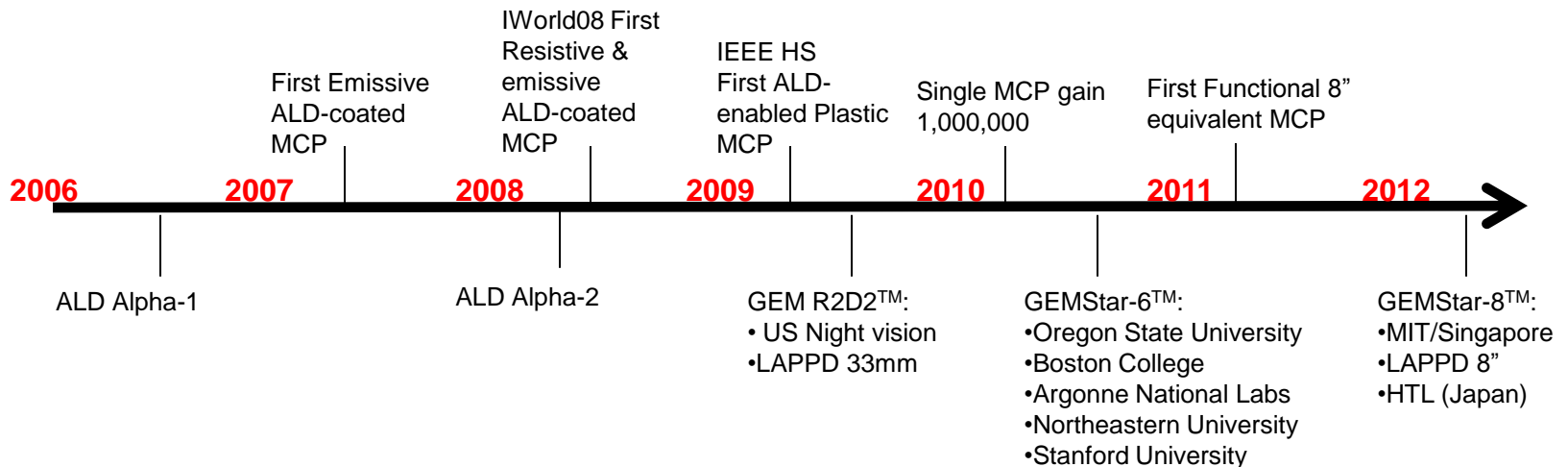
Task	Q1			Q2			Q3			Q4		
	M1	M2	M3	M1	M2	M3	M1	M2	M3	M1	M2	M3
Objective 1- 200mm ALD process & anneal equipment	[Light orange bar]											
Architecture	[Dark orange]	[Dark orange]										
Detailed Design	[Dark orange]	[Dark orange]	[Dark orange]									
Procurement			[Dark orange]	[Dark orange]	[Dark orange]							
Build					[Dark orange]	[Dark orange]	[Dark orange]					
Objective 2- ALD Process window study	[Light orange bar]											
Precursor disbursement optimization: Expo / Dose	[Dark orange]											
Cycle time reduction		[Dark orange]										
Objective 3- Optimized MCPs for Tube processing	[Light orange bar]											
Scrub/ Charging optimization	[Dark orange]	[Dark orange]	[Dark orange]	[Dark orange]								
Thermal optimization			[Dark orange]	[Dark orange]	[Dark orange]							
Seal optimization					[Dark orange]	[Dark orange]						
Lifetime optimization			[Dark orange]	[Dark orange]	[Dark orange]	[Dark orange]						
Objective 4- optimize non-ald processing for large substrates	[Light orange bar]											
Cleans							[Dark orange]					
Anneals								[Dark orange]	[Dark orange]			
electroding								[Dark orange]	[Dark orange]	[Dark orange]		
Objective 5- bring up ALD process on new chamber	[Light orange bar]											
Baseline existing Phase I process on equivalent surface area								[Dark orange]	[Dark orange]	[Dark orange]		
Optimize Phase I process for new chamber									[Dark orange]	[Dark orange]	[Dark orange]	
Objective 6- ALD Process characterization on new chamber	[Light orange bar]											
Precursor disbursement optimization												[Dark orange]
Verify MCP performance												[Dark orange]
Scrub/ Charging optimization												[Dark orange]
Thermal optimization												[Dark orange]
Seal optimization												[Dark orange]
Lifetime optimization												[Dark orange]
Objective 7- Provide 8" plates to Argonne collaboration	[Light orange bar]											
First articles												[Dark orange]



Major Program Milestones

- ❖ 200mm Process and anneal equipment build – January 2012
- ❖ First 200mm MCP samples delivered to Argonne LAPD collaboration – May 2012
- ❖ LAPD Production support for Argonne LAPD collaboration begins – October 2012
- ❖ Production plan for Phase III delivered to Argonne LAPD collaboration – July 2013

Arradiance ALD-MCP Technology & Equipment Development



US Patents:

- ◀ 5,729,244 (Filed 04/1995) Field Emission Device with Microchannel Gain Element
- ◀ 6,522,061 (Filed 08/2000) Field Emission Device with Microchannel Gain Element
- ◀ 7,408,142 (Filed 09/2006) Filed MCP Amplifier with Tailored Pore Resistance
- ◀ 7,759,138 (Filed 09/2008) Silicon MCP Devices With Smooth Pores and Precise Dimensions
- ◀ 7,855,493 (Filed 02/2008) MCP Devices With Multiple Emissive Layers
- ◀ 8,052,884 (Filed 02/2008) Method of fabricating MCP devices with multiple emissive layers
- ◀ 7,977,617 (Filed 04/2009) Image intensifying device having a MCP with a resistive film for suppressing the generation of ions

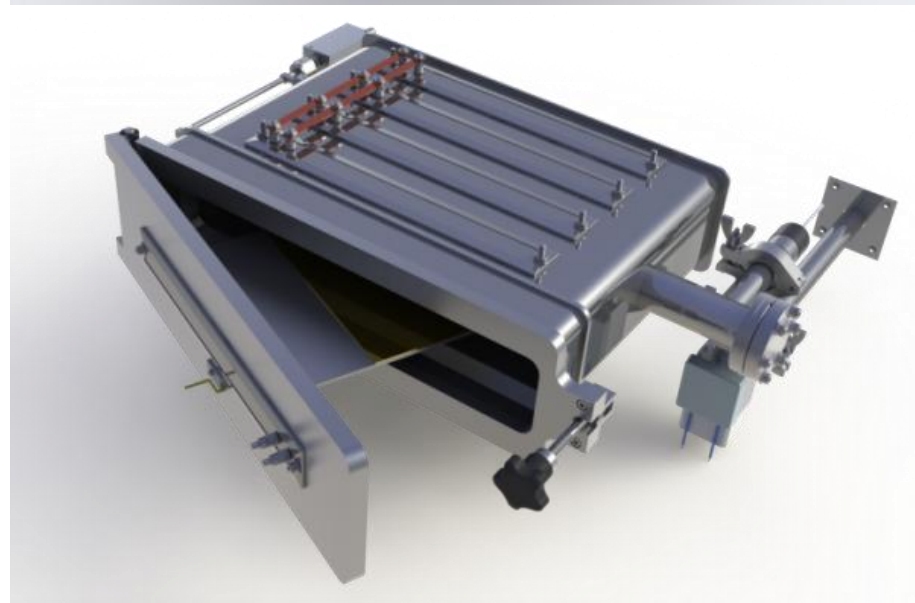
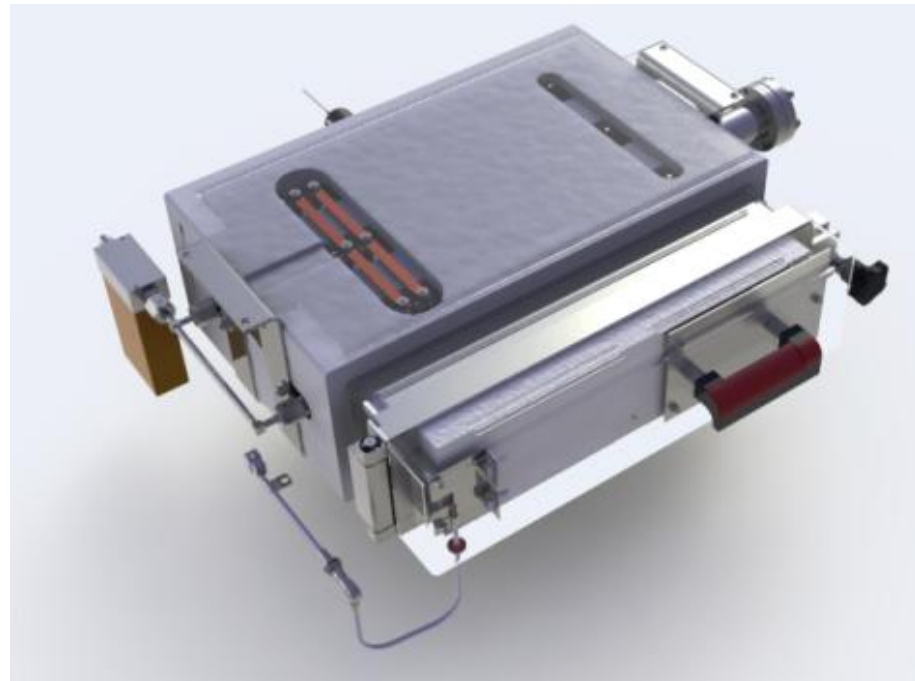
◀ 1 US Patents Allowed - Image intensifying device (Filed 06/2011)

◀ US Patents Pending:

- ◀ Microchannel Plate Devices With Tunable Resistive Films (filed 06/2008)
- ◀ Microchannel Plate Devices With Tunable Resistive Films (Filed 02/2009)

GEMSTAR-A: Anneal Chamber Preliminary Specifications

- ◀ Chamber
 - ◀ 3"x 15" x 10" Thermally Controlled
 - ◀ Substrate to 500C +/-1%
 - ◀ Customizable End Effector Interface
 - ◀ MFC Controlled User Selectable Gas Input
 - ◀ All Metal Seal Gas Handling
 - ◀ System Pressure Monitoring
- ◀ 20A 120V 50/60 Cycle Power
- ◀ Heated Vacuum Isolation Valve
- ◀ Operating Controls Environment:
 - ◀ Custom USB Control Module
 - ◀ Dell Vostro Laptop with Windows 7 (64 bit OS)
 - ◀ GEMFlow Software User Scripting and logging
- ◀ Size: 13.32"x30.16" x19.64
- ◀ System Weight: 150lbs

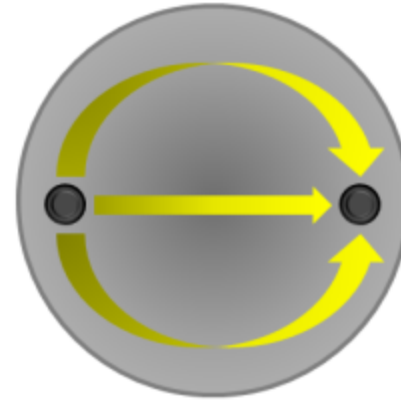




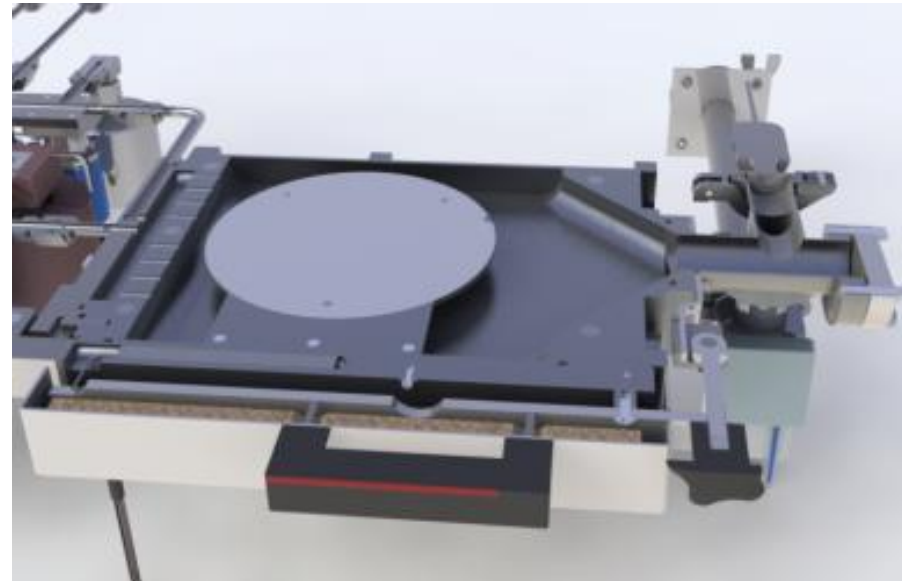
GEMSTAR-8™: Innovative Precursor Delivery



◀ First in-house system (ALD Alpha-1) had a single input & output ports resulting poor film uniformity.

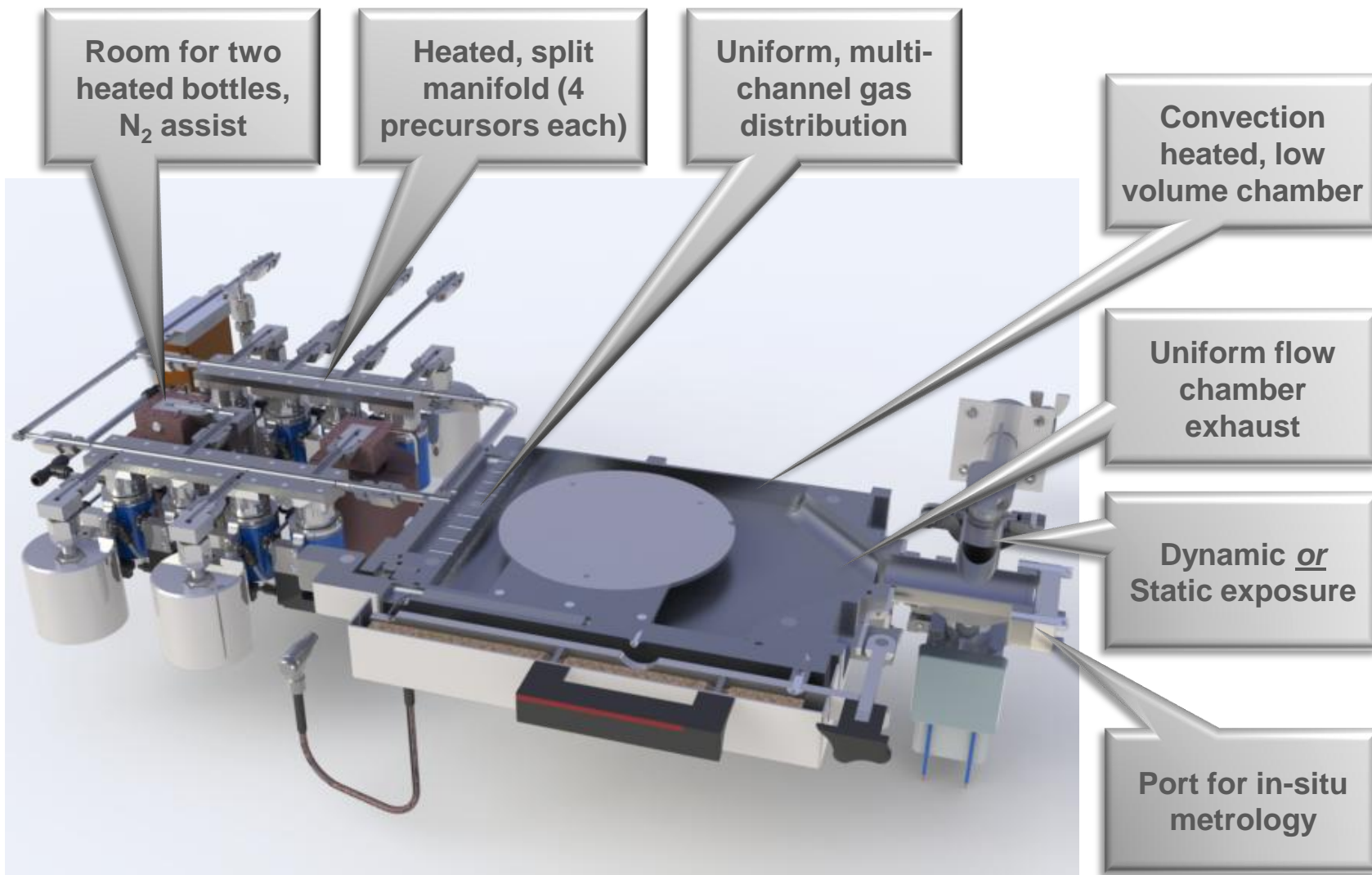


- ◀ **GEMSTAR™-8** design incorporates:
- ◀ Multi-channel precursor delivery system to isolate & evenly distribute precursors into process chamber
 - ◀ Tapered exhaust maintains uniformity through chamber





GEMSTAR-8™: System Internals

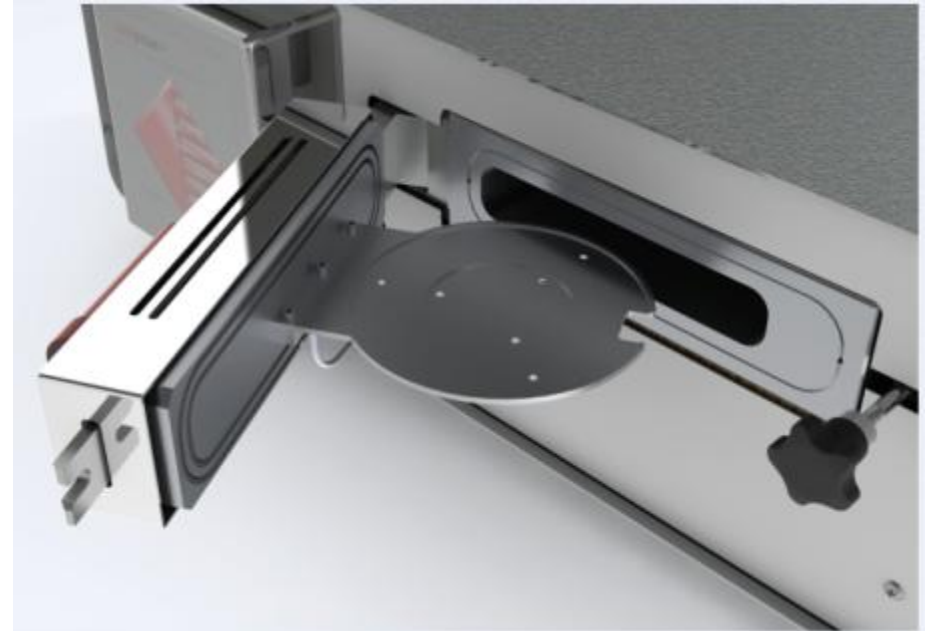




GEMSTAR-8™: **Zero permeation**



- ◀ Permeation of molecular species (e.g. H, H₂O, O₂ & etc.) through a single O-ring is caused by differential pressure across the O-ring.
- ◀ O-ring permeation is a function of both temperature & cross section.
- ◀ The permeated species can be active in the process chamber, resulting in parasitic CVD, poor metal film growth and other non-ideal ALD characteristics



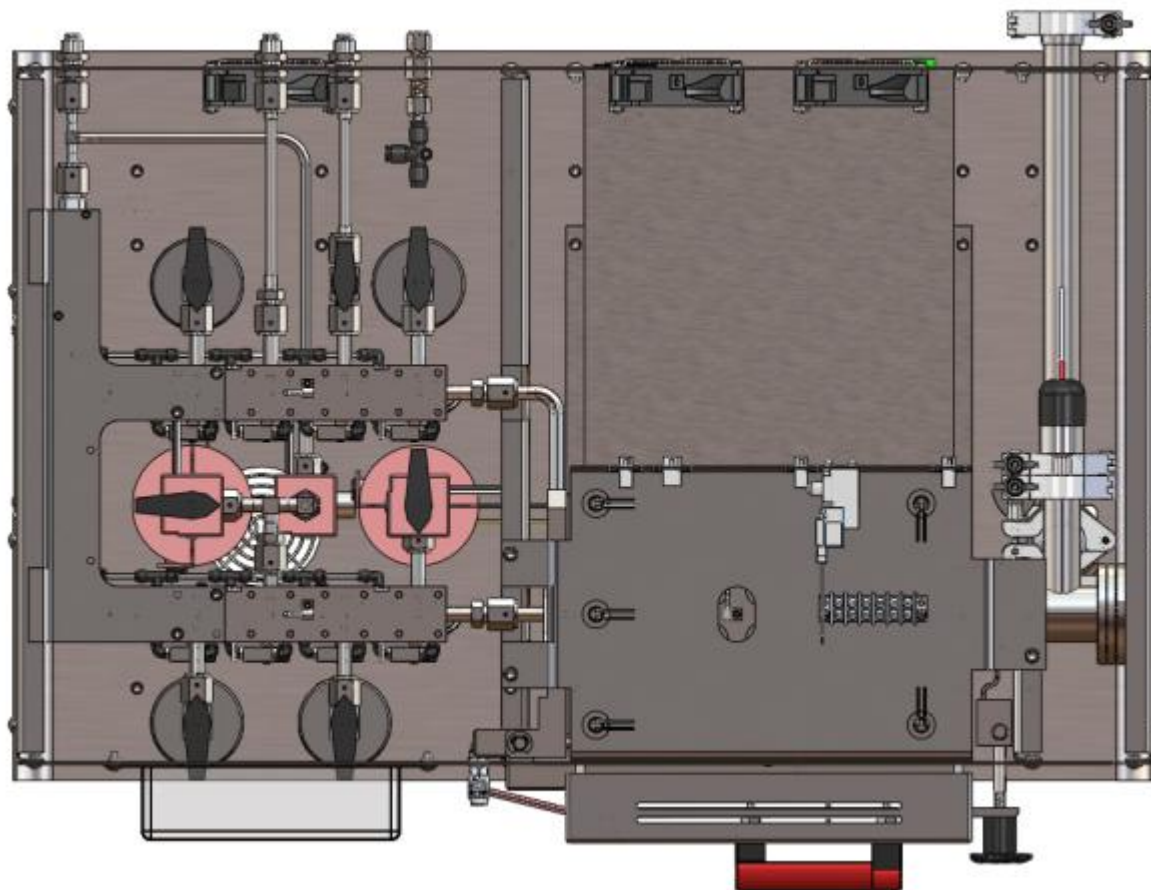
- ◀ **GEMSTAR-8™** solution: differentially pumped system seals
 - ◀ No decrease in O-ring cross section with temperature at any interface.
 - ◀ Two O-rings are used, with a vacuum-side groove to vector permeation out prior to the O-ring at the process reactor chamber, at gas inlet & chamber door



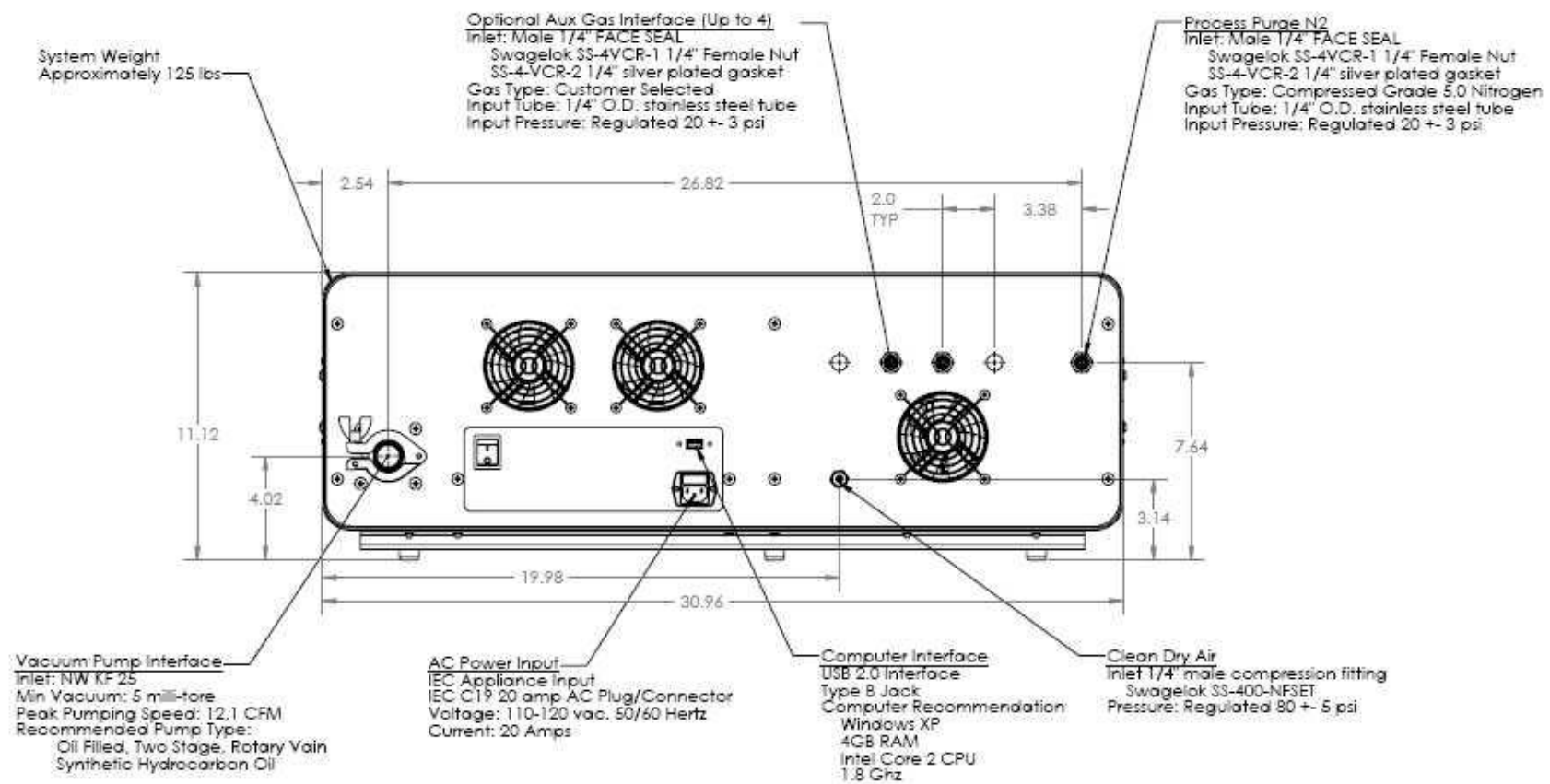
GEMSTAR-8™ Standard Features



- ◀ Control Module
- ◀ Heated FASTFlow Manifolds
- ◀ Metrology Interface
- ◀ Inboard electronics
- ◀ Ease of Reactor Removal
- ◀ Pressure Gauge
- ◀ Split Manifold (no cross-contamination)
 - ◀ Metal-organic
 - ◀ Oxidizer Reducer
- ◀ 2 precursor valves standard. Room for up to six high capacity bottles and 2 independent gas lines



Facilities Interface



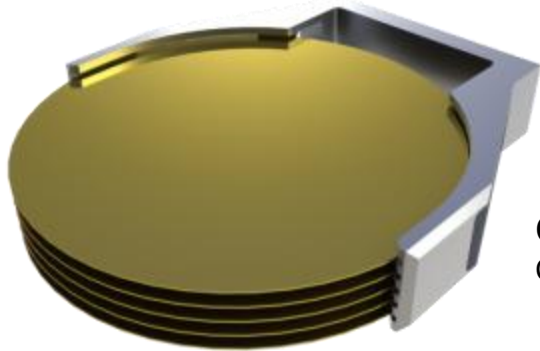


GEMSTAR-8™

Optional Substrate End Effectors and Cassettes

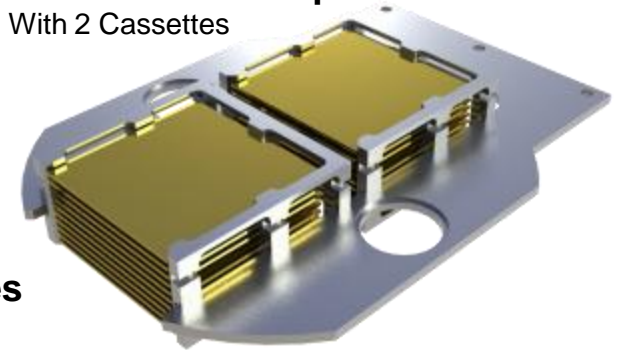
QTY 5 150 mm Dia Substrates

Cassette Mounts To Door



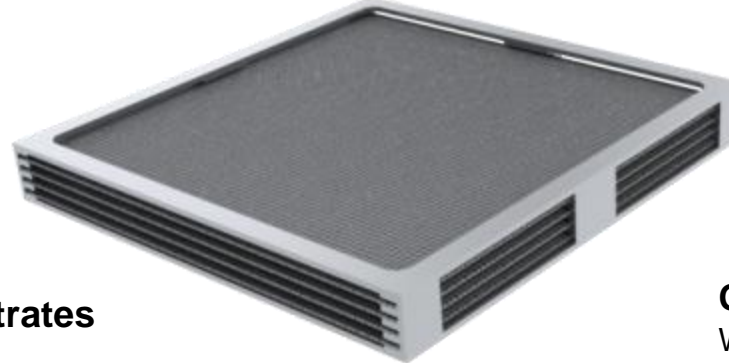
QTY 20 54 mm Square Substrates

With 2 Cassettes



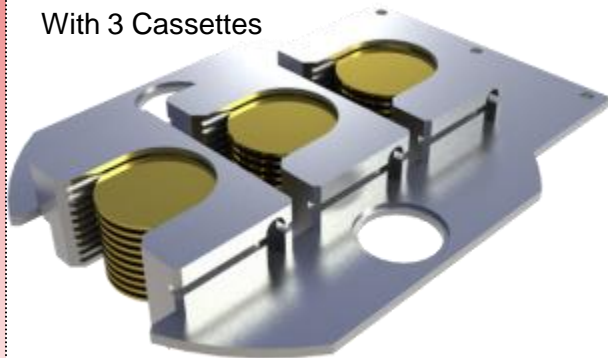
QTY 5 200 mm Square Substrates

Cassette slides into chamber



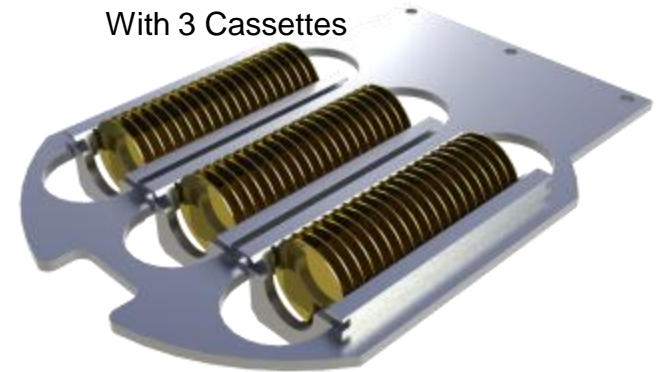
QTY 30 33 mm Dia Substrates

With 3 Cassettes



QTY 60 25mm Dia Substrates

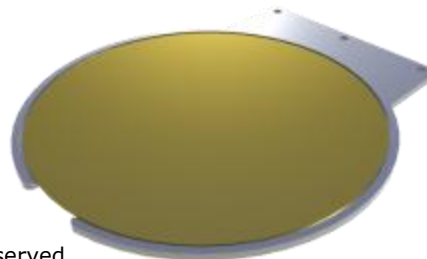
With 3 Cassettes



GEMSTAR

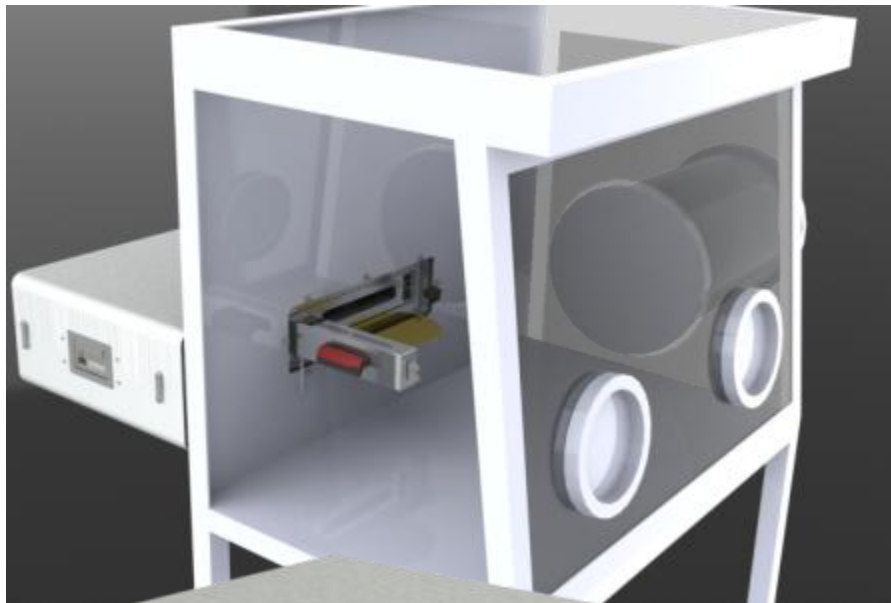
STD End Effector

Single/Flexible Substrates





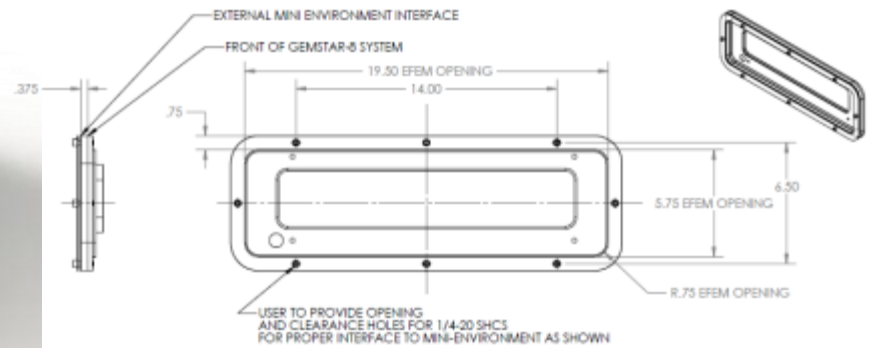
GEMSTAR-8™: Sealed Mini-Environment Interface



Processing Environmentally Sensitive Material With ALD

GEMSTAR-8 provides an optional sealed glove box / mini environment interface with defined hard points making it user friendly

Maintaining ease of service, maintenance and precursor modification



GEMFlow™ Control & Analysis Software



GemStar ALD -- ARR-PC4

Main Chamber

Main Temp. (°C): 146.6

Door Temp. (°C): 146.3

Expo Valve: Heater On, Actuator Open

Vent / Pump Seq.: Vent, Pump

Set: 150.0

PID Output %: 36.3

Bottle Heater Temp. (°C)

1: 99.9

2: 0.0

Set: 100.0

PID Output %: 37.6

Manifold Temp. (°C)

Metal/Organic: 115.0

Oxidizer/Reducer: 115.0

Set: 115.0

PID Output %: 37.9, 36.4

System

Status: OK

Standby, Heat Off

Advanced... (Password Required)

Event Log

System Temperature

Current: Measured Max

0 °C, 100 °C, Reset

Metal/Organic ALD Valve Control

Valve 1	Valve 2	Valve 3	Valve 4
Time (mS): 1000	Time (mS): 20	Time (mS): 1000	Time (mS): 1000
Fill Estimate: 0.0 g	Fill Estimate: 4.1 g	Fill Estimate: 5.2 g	Fill Estimate: 8.3 g
0.0 %	40.2 %	49.7 %	78.0 %

Mass Flow Controller

SCCM: 93.28

Set: 99.9

Nitrogen Injection

20 millSec.

Pulse

Oxidizer/Reducer ALD Valve Control

Valve 5	Valve 6	Valve 7	Valve 8
Time (mS): 20	Time (mS): 1000	Time (mS): 1000	Time (mS): 1000
Fill Estimate: 9.4 g	Fill Estimate: 10.0 g	Fill Estimate: 9.0 g	Fill Estimate: 9.3 g
86.7 %	90.7 %	80.6 %	81.2 %

Chamber Status

Chamber Vented, Vacuum Ready

*** Simulator Mode ***

ARRADIANCE v1.0.8

Exit

Recipe Setup -- ARR-PE-ALD2

Label	Device	Action	Value	Branch
1	EXPO Heater	Set to On	0.0	
2	ALD Valve 1 Heat	Set to Value	110.0	
3	ALD Line 1 Heat	Set to Value	35.0	
4	Precursor 1 Heat	Set to Value	32.0	
5	ALD Valve 2 Heat	Set to Value	110.0	
6	ALD Line 2 Heat	Set to Value	35.0	
7	Precursor 2 Heat	Set to Value	32.0	
8	ALD Valve 3 Heat	Set to Value	100.0	
9	ALD Valve 4 Heat	Set to Value	100.0	
10	Chamber Heat 1	Set to Value	175.0	
11	Chamber Door Heat	Set to Value	175.0	
12	ALD Valve 1 Heat	Set to Value	0.0	
13	MFC Iso. Valve	Set to Open	0.0	
14	MFC Flow	Set to Value	200.0	
15	Chamber Heat Avg.	Wait Until Set Point +/-	2.0	
16	MFC Flow	Set to Value	10.0	
17	Delay (Sec.)		2000.0	
18	ALD Valve 1 Heat	Set to Value	0.0	
19	APImer	ALD 2 Actuator	Pulse (mSec.)	20
20	Delay (Sec.)		1.0	
21	Branch n Times	Number	2	APImer
22	AZ03	MFC Flow	Set to Value	5.0
23	Delay (Sec.)		1.0	
24	EXPO Actuator	Set to Closed	0.0	
25	Delay (Sec.)		0.4	
26	TMA	ALD 2 Actuator	Pulse (mSec.)	20
27	Delay (Sec.)		0.5	
28	EXPO Actuator	Set to Open	0.0	
29	MFC Flow	Set to Value	60.0	

Recipe Control

Insert Before, Insert After, Delete

Save, Load

Run Control

Run Once, Single Step, Stop

Cycle Count

1, 1

Run Cycles

Cycle Time

Elapsed: 09:01:54, Last: 09:01:54

Sequence Time

Elapsed: 09:01:54, Est. Remaining: Don't know

File Logging

Logging: \\var-srv1\process equipment\ALD\ARR-PE-ALD2\Log\AZ03\07-19-2009 500x AZ03.txt

Interval Log: 0.10 Sec.

Comment: Post

Chamber Temp. PID Loop -- ARR-PE-ALD2

Scale: 180

Plot Span (S): 0.00

135.00 Setpoint

135.66 Temp. C

0.00 Output %

PID Parameters

Kc: 400.000, Ti: 0.000

Td: 0.400, Sample Rate: 1.255

Loop Jitter (mS): 0.0

Autotune

Kc: 0.00, Ti: 0.00

Td: 0.00, Noise: 0.00

Status: Start, Apply, Quit

Note: Wait for process variable to converge before autotuning.

GEMSTAR-8™: Summary



- ◀ Precursor temperature precisely controlled from bottle to chamber.
- ◀ Metallorganics and oxidants/reductants are mounted on separate manifolds, **increasing the lifetime of valves** and eliminating film growth on manifold walls.
- ◀ **Exposure control** critical to grow films with the desired conformality.
 - ◀ Partial pressure & residence time precisely controlled with downstream valve.
 - ◀ Precursor usage (\$\$) can be precisely controlled
- ◀ Controlled laminar flow throughout:
 - ◀ Improves ALD performance on complicated film stacks
 - ◀ Provides exceptional conformality on high aspect ratio structures
 - ◀ Quick flow manifold & high conductance result in fast purge times & minimal ALD **cycle time**.
- ◀ Zero permeation seals facilitates **deposition of metals**
- ◀ **8 different precursors** can be installed and run concurrently
- ◀ Chamber design allows for quick attachment of optional QCM, **motorized fixtures (e.g. powder)** or mass spectrometer