## PILOT PRODUCTION OF LARGE AREA MICROCHANNEL PLATES AND PICOSECOND PHOTODETECTORS

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Pilot production performance is reported for large area atomic layer deposition (ALD) coated microchannel plates (ALD-GCA-MCPs) and for Large Area Picosecond Photodetectors (LAPPD<sup>™</sup>) which incorporate them. "Hollowcore" glass capillary array (GCA) substrates are coated with ALD resistive and emissive layers to form the ALD-GCA-MCPs, an approach that facilitates independent selection of glass substrates that are mechanically stronger and that have lower levels of radioactive alkali elements compared to conventional MCP lead glass, reducing background noise[1,2,3,4]. ALD-GCA-MCPs have competitive gain (~10<sup>4</sup> each or ~10<sup>7</sup> for a chevron pair ), enhanced lifetime and gain stability (7 C cm<sup>-2</sup> of charge extraction), reduced background levels (0.028 events cm<sup>-2</sup> sec<sup>-1</sup>) and low gamma-ray detection efficiency. They can be fabricated in large area (20cm X 20 cm) planar and curved formats suitable for use in high radiation environment applications, including astronomy, space instrumentation, and remote night time sensing. The LAPPD photodetector incorporates these ALD-GCA-MCPs in an all-glass hermetic package with top and bottom plates and sidewalls made of borosilicate float glass. Signals are generated by a bi-alkali Na<sub>2</sub>KSb photocathode, amplified with a stacked chevron pair of ALD-GCA-MCPs. Signals are collected on RF strip-line anodes integrated into to the bottom plates which exit the detector via pin-free hermetic seals under the side walls [5]. Tests show that LAPPD<sup>TM</sup>s have electron gains greater than 10<sup>7</sup>, submillimeter spatial resolution for large (multiphoton) pulses and several mm for single photons, time resolution less than 50 picoseconds for single photons, predicted resolution less than 5 picoseconds for large pulses, high stability versus charge extraction[6], and good uniformity for applications including astrophysics, neutron detection, high energy physics Cherenkov light detection, and quantum-optical photon-correlation experiments.

**Introduction:** The Large Area Picosecond Photodetector Collaboration was established in 2009 with the goal of developing an MCP based photodetector, capable of imaging, with high single-photon sensitivity, high spatial and temporal resolution, in a hermetic package with a minimum 400 cm<sup>2</sup> active area. The motivation for this development was a recognition that MCP detectors, despite their high speed, were not considered for applications that require large-area coverage due to the small size, high-cost / area, and the poor lifetime [7] of those that were commercially available. It is now acknowledged that large area, affordable, very fast photodetectors with sub-mm space resolution and time resolutions below 10 picoseconds (psec) would be a disruptive technology across all three of the physics frontiers [8]. While the initial target applications for this development were in the field of high energy physics (HEP), applications in many other areas that will benefit from this development are evolving, including: homeland security, astronomy, space instrumentation, remote night time sensing, TOF mass spectrometry, plenoptic and medical imaging (PET scanning) and others.

Early on, the performance of the LAPPD<sup>TM</sup> was established using an experimental test stand that allowed standard LAPPD components to be assembled with an O-ring seal between the top window and the body of the photodetector, and tested while the tile was being dynamically pumped. Since a bi-alkali photocathode cannot tolerate ambient exposure, an aluminum photocathode was used. While a thin metal film of aluminum can serve as a photocathode, it typically has a low quantum efficiency (QE), however when used with a laser source, the light intensity is sufficient to allow reliable testing. This test apparatus to evaluate LAPPD<sup>TM</sup> performance was referred to as the "demountable". Performance results [6] were as follows: absolute time resolutions for single-photoelectrons were consistently < 100 psec, and typically < 60 psec; for large pulses projected time resolutions were < 5 psec. Spatial resolution determined for single photoelectrons was several millimeters, while for large pulses a spatial resolution of 700-750 microns was demonstrated. Fig. 1 shows the measured 50psec transit time variation for a single photoelectron determined on the "demountable".

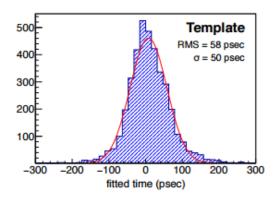


Fig. 1, Measured 50 psec Transit Time Variation for Single Photoelectron

These results made it apparent that the next challenge was to establish a pilot production facility, establish routine fabrication of the large area microchannel plates that were the enabling technology for the LAPPD<sup>TM</sup>, and to replicate the performance results demonstrated with the demountable, with a fully sealed photodetector incorporating a bi-alkali photodector. In April 2014, the Department of Energy awarded Incom Inc. a two year contract to establish that facility and to demonstrate a pathway toward pilot production [9].

**ALD-GCA-MPC Manufacture -** One of the enabling technologies for the LAPPD<sup>TM</sup> is the large area microchannel plate which provides signal amplification. This MCP, the world's largest, is made by a novel technique that uses atomic layer deposition (ALD) to apply resistive and emissive layers to a GCA to produce the "ALD-GCA-MCP". The ALD-GCA-MCP is so named to differentiate it from conventional lead oxide MCPs, and conventional lead oxide MCPs that have been enhanced with an ALD applied secondary emissive layer. Fig. 2 depicts the process for fabricating large blocks of glass capillary array material using Incom's hollow core technique. These blocks, approximately 229 mm X 229 mm X 406 mm can produce ~140-150 203mm square glass capillary array (GCA) wafers that serve as substrates for application of resistive and emissive coatings applied by ALD, as depicted in the sketch [10], lower left. Once coated, the microchannel plate (MCP) amplifies signals as shown in the sketch [11], lower right.

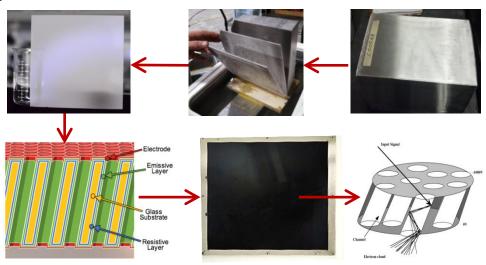


Fig. 2, right to left: Incom uses a "Hollow Core Process" to produce large blocks of capillary array material that can be sliced, producing GCAs. Bottom, left to right: Resistive and emissive coatings are applied to these GCA wafers by atomic layer deposition to convert the GCA to an ALD-GCA-MCP. Signal amplification is achieved as shown in the sketch, lower right

The performance achieved with ALD-GCA-MCPs is shown in Fig. 3, top left; Conventional MCPs require an extensive "burn-in" and the gain often drops significantly (factor of 5) after burn in before stabilizing. Little burn-in is required for ALD-GCA-MCPs indicated by the violet colored line in the plot [12]. Top right chart shows MCP gain vs. voltage, showing that the gain for a pair of 33 mm, 60:1 L/D, 20 µm pore ALD-GCA-MCPs remains high

and stable even after extracting 7 C/cm² charge at  $\sim$ 3  $\mu$ A [13]. The plot shown in Fig. 3 bottom left shows the secondary electron yield as a function of thickness, of the secondary electron emissive layer, which is typically either MgO or Al<sub>2</sub>O<sub>3</sub>, both of which have high secondary emissive yield [14]. As shown in Fig. 3 bottom right, gain uniformity across full area of ALD-coated 203 mm square MCP is typically within  $\sim$ 15%. The gain map image shown is for a pair of 20  $\mu$ m pore, 60:1 L/D, ALD borosilicate MCPs, measured at 950 volts per MCP, with 184 nm UV exposure. Table 1 provides a summary of ALD-GCA-MCP attributes and competitive advantages.

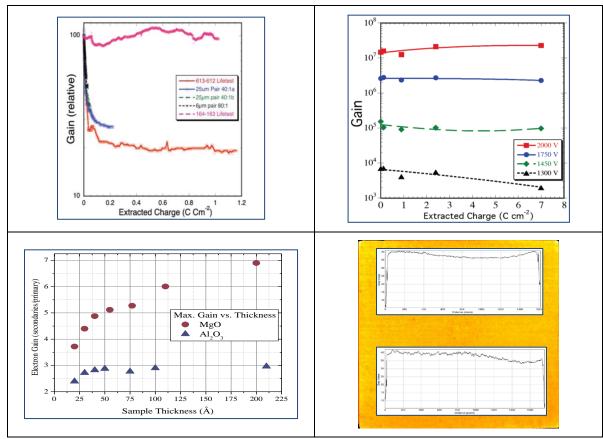


Fig. 3, Performance achieved with ALD-GCA-MCPs

Table 1 - Summarizes ALD-GCA-MCP attributes and competitive advantage

MCP Attribute	ALD-GCA-MCP Competitive Advantage
Tunable	Independent selection of glass substrate and tuning of resistive and emissive properties.
Gain Stability,	High (10 <sup>5</sup> -10 <sup>7</sup> ) overall gain, long-term temporal stability, minimal burn-in Vs. conventional MCPs that experience 5-
Burn-in	10X gain drop and require extended (200 hrs) charge extraction. Hydrogen free process, negligible ion feedback.
Low Dark Count	10-25 X Lower dark count (0.025-0.040 vs. 0.25-1.0) since for ALD-GCA-MCPs contain little or no radioactive
(cm <sup>-2</sup> s <sup>-1</sup> )	isotopes, Enhanced S/N.
High SEY	~2.5 – 3.5 for conventional vs. ~2.5 to 3.0 and ~4 to 7 for Al <sub>2</sub> O <sub>3</sub> and MgO SEE Layers. <b>Higher Gain Sensitivity</b>
Low X-ray Cross- Section	No lead, for application in an X-ray background.
TCR (K <sup>-1</sup> )	Semiconductor like behavior with TCR = ~-0.01 to -0.03 for conventional, and ~ -0.02 to -0.04 for ALD-GCA-MCPs
Pore Size	10 µm pores, with OARs up to 74% in large plate sizes for enhanced detection efficiency; and spatial and temporal
& OAR	resolution.
Large Size	203mm X 203mm MCPs wafers, the world's largest
Gain uniformity	Within 15% across 203mm x 203mm plates with 20 μm pores
Robust MCPs	No H <sub>2</sub> firing or acid etching, that make conventional lead silicate MCPs fragile, moisture sensitive and prone to shape
	distortions. Chemically, thermally and mechanically stable.
Curved Shape	Enhanced resolution for space and terrestrial TOF instrumentation, simplified design, reduced instrument volume, cost
	and mass.
Lower cost per area	Large area MCPs, diced to smaller sizes, low cost glass substrates with independently optimized resistive and emissive
	coatings results in enhanced performance with significant cost and design flexibility.

## **LAPPD Design** - The LAPPD follows a simple design, depicted in Fig. 4,

- a) Left showing a chevron pair of ALD-GCA-MCPs separated with X-spacers, sitting in a pre-fabricated lower tile assembly (LTA) the bottom of which is the detector anode. X-spacers restrain window deflection under atmospheric pressure, control and provide critical spacing between components, and support for getters. A borofloat or optional fused silica glass window sits on top and is hermetically sealed to the sidewalls with a low melting indium alloy.
- b) Middle Lower Tile Assembly (LTA), consisting of borofloat glass sidewalls and bottom anode plate, hermetically sealed together. Thin film power & signal anode strips pass under the hermetic seal providing a "penetration free" connection into and out of the tile. Internal corner pins hold components and deliver voltage to the top and bottom of each MCP.
- c) Right a side view showing the stack-up of components, and the groove in the sidewall that retains the molten indium alloy used for top window sealing.

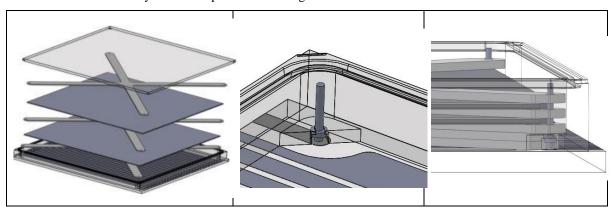


Fig. 4 – Showing the construction of the LAPPD

Final assembly of the LAPPD is done in an ultra-high vacuum (UHV) tank depicted in Fig. 5, equipped with conflat seals, scroll, turbo and ion pumps. Tile kit components are pre-assembled & loaded in place, and then baked @ 350C to a low 10<sup>-10</sup> torr range. After in-tank scrubbing of the ALD-GCA-MCPs, the Na<sub>2</sub>KSb photocathode is applied at an elevated temperature using SAES alkali-metal dispensers. Development of the photocathode can be monitored by measuring photocurrent. The absolute quantum efficiency of the photocathode can be measured using NIST calibrated photodiodes. Once the photocathode is deposited, the window is moved back using an in-vacuum window transfer process and lowered onto the detector sidewalls where it forms a hermetic seal as the molten indium alloy cools. The current set-up provides for limited measurement of the detector performance while it is still in the tank.



Fig. 5, Final assembly of the LAPPD<sup>TM</sup> is done in an ultra-high vacuum (UHV) tank.

Fig. 6 (top) shows the photocathode QE for LAPPD<sup>TM</sup> #7. QE measurements are done during deposition, while the PC is still hot. Prior experience shows that the bi-alkali photocathode QE will increase upon cooling, as shown

(bottom left) for PC Shoot #6 which increases as the film cools (red, as-deposited at 190C on 4/7/2016, and at room temperature on 4/8 dark blue, on 4/11 light blue, and on 4/20 black). QE uniformity had been previously shown to be uniform over a 203mm square area as shown in the lower right figure [15].

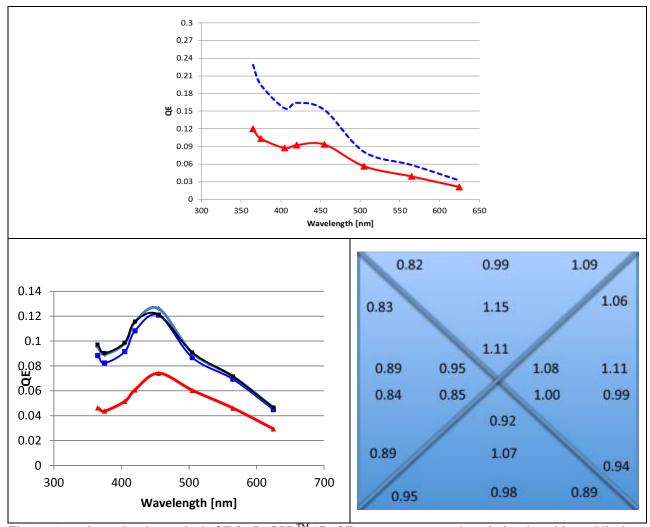


Fig. 6 – (top) shows the photocathode QE for LAPPD<sup>TM</sup> #7. QE measurements are done during deposition, while the PC is still hot. Prior experience shows that the bi-alkali photocathode QE will increase upon cooling, as shown (bottom left) for PC Shoot #6 which increases as the film cools (red, as-deposited at 190C on 4/7/2016, and at room temperature on 4/8 dark blue, on 4/11 light blue, and on 4/20 black). QE uniformity had been previously shown to be uniform over a 203mm square area as shown lower right.

Pilot production commissioning trials - Pilot production commissioning trials were begun in January 2016, following a two year construction period in which pilot production infrastructure including equipment and clean room facilities were completed. The goal of these trials was to identify and resolve technical issues ultimately leading to a routine process for fabrication of fully integrated, free standing LAPPD<sup>TM</sup>. Multiple leak tight indium top window seals were made demonstrating that sealing along the 812mm perimeter of the LAPPD<sup>TM</sup> was not a barrier to success, while also achieving a major interim program goal. Process variables affecting the ability to produce effective, high QE photocathodes were understood and optimized achieving QEs that varied from 1% to 12% @ 365 nm and 190C. A "manufacturing pipeline" for tile kit components, including large area ALD-GCA-MCPs was created at Incom to insure that the pace of future manufacturing would not be limited by supply of these critical path components. In addition, LAPPD<sup>TM</sup> tiles were assembled, sealed and tested, demonstrating a pace of 2 tiles / month with the current UHV integration & sealing unit, with ample opportunity for cycle time reduction and scale-up to higher volumes. Detector tile design was optimized to eliminate stack-height variability issues which

were a major cause of failure for a number of the early tile trials. Another area of concern was whether specially prepared sealing surface along the edge of the top window were being contaminated with alkali metals during photocathode deposition, preventing a good seal.

Tile trial #9 was designed to use a thin film aluminum metal photocathode, as a test to confirm whether alkali photocathode spill-over onto the critical indium sealing surfaces was contributing to earlier sealing problems. LAPPD<sup>TM</sup> #9 was completed and removed from the UHV tank on September 14th, 2016. The limited testing done to date convincingly demonstrated that the tile is functional, at a level consistent with our expectations for an aluminum photocathode [16]. Fig. 7 shows the gain vs. voltage for the MCP pair used in LAPPD #9. Fig. 8 shows pulses recorded under ambient full atmosphere conditions, when exposed to a Hg(Ar) lamp. The pulse rate doubles when the lamp is turned on. The threshold trigger rate for the oscilloscope was set high due to periodic noise from the lamp. Further ambient testing of the tile was performed employing various illumination scenarios as shown in Fig. 9. The voltages applied to the stack was -2,600 to the photocathode, -2,400 and -1,400 respectively to the top and bottom of the entry ALD-GCA-MCP, -1,200 and -200 respectively to the exit ALD-GCA-MCP. That corresponds to a gain of 3 X 10<sup>6</sup> based upon measurements done earlier on the MCP pair used in LAPPD #9 as shown in Fig. 7.

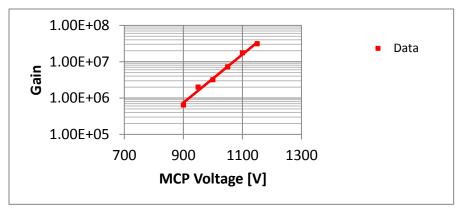


Fig. 7 – Gain vs. voltage for the MCP pair used in LAPPD #9

Fig. 9 shows the anode current over time, as the Hg(Ar) lamp was turned on, then the photocathode turned off, then the Hg(Ar) lamp turned off, and finally with a 360nm LED turned on. The photocathode was "turned off" by reversing the electric field in the gap between the entry window and the top face of the entry ALD-GCA-MCP thus blocking photoelectrons from entering the ALD-GCA-MCP. In this "photocathode off" mode anode current was still detected, because the UV light was able to stimulate emission of electrons from the top of the entry ALD-GCA-MCP, which were amplified by the ALD-GCA-MCPs and extracted from the anode. With the lamp turned off the dark current, read at the anode strips, was about 13nA.

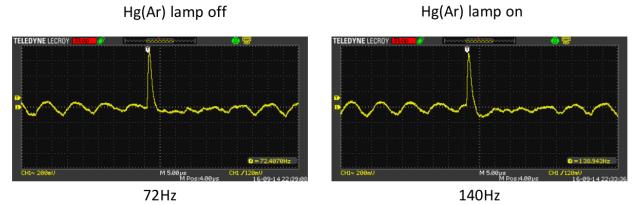


Fig. 8, Oscilloscope pulses recorded for sealed tile #9 under ambient full atmosphere conditions, when exposed to a Hg(Ar) lamp. 1,000V was applied to each ALD-GCA-MCP corresponding to a gain of 3X10<sup>6</sup>. The pulse rate doubles when the lamp is turned on. The threshold trigger rate for the oscilloscope was set high due to periodic noise from the lamp.

## **Anode current vs Time** 2.50E-06 2.00E-06 360nm LED on 1.50E-06 1.00E-06 5.00E-07 0.00E+00 0.5 1 1.5 2 2.5 e [Hours] Photocathode off Hg(Ar) lamp off Hg(Ar) lamp on

Fig. 9, Ambient testing of Tile #9 under various illumination scenarios confirmed that the detector was operational.

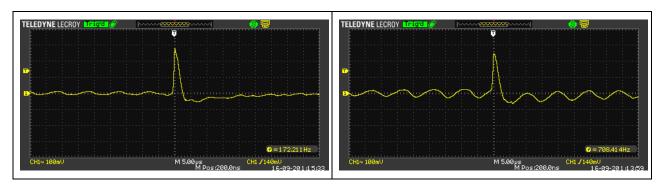


Fig. 10 - Hg(Ar) Lamp-off (left), lamp-on (right) testing was repeated six days later under ambient pressure conditions with the output waveforms averaged over 32 pulses. 1,100 volts was applied to each of the MCPs, corresponding to a gain of  $2X10^7$ .

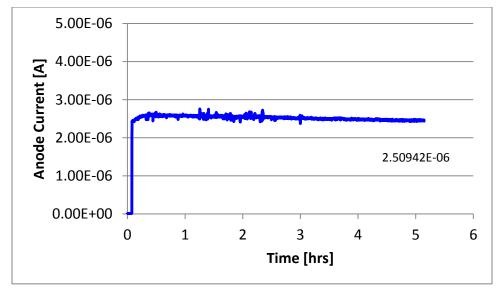


Fig. 11 – Anode current vs. time measurement was repeated for LAPPD #9 on 9/20/2016 after 7 days, with the LED off, the dark current ( $I_{DARK}$ ) was ~20nA, increasing to 2.5 micro-amps when exposed to 360nm with LED on.

"Lamp-off, lamp-on" testing was repeated six days after the tile was sealed, under ambient pressure conditions with the output waveforms averaged over 32 pulses, as shown in Fig. 10. 1,100 volts was applied to each of the MCPs, corresponding to a gain of  $2X10^7$ . In these trials pulse rate increased by more than a factor of 4X from 172Hz to 708Hz. Anode current vs. time measurement was repeated after 7 days, with the LED off, the dark current ( $I_{DARK}$ ) was ~20nA, increasing to 2.5 micro-amps when exposed to 360nm with LED on, as shown in Fig. 11.

Comprehensive testing of the tile continues, with plans to characterize timing and operational stability vs. time. Production of additional tiles remains on pace, which will utilize the more efficient Na<sub>2</sub>KSb bi-alkali photocathode.

Conclusions and future plans - Progress on infrastructure and process development has been steady. In the 2½ years since beginning construction in April 2014, a pilot production infrastructure has been created, starting with nothing. An experienced team of scientists, engineers, and technicians was assembled enabling ALD-GCA-MCP and LAPPD<sup>TM</sup> process development commissioning trials to begin in December 2015.

No technical roadblocks or insurmountable barriers have been encountered since commissioning trials began in December 2015. Development trials progressed as follows: a) Routine operation of Incom integration & sealing tool operating at 10<sup>-10</sup> Torr, b) demonstration of a repeatable 203mm X 203mm ALD-GCA-MCP fabrication process, c) multiple sidewall to top window sealing trials, d) photocathode deposition trials, e) mock LAPPD<sup>TM</sup> assembly and sealing trials, followed by f) nine fully integrated LAPPD<sup>TM</sup> fabrication trials. Eight leak tight indium top window seals were made achieving a major program goal. Eleven K<sub>2</sub>NaSb PCs were deposited on 8"X8" widows with QEs that varied from 1% to 12% @ 365 nm and 190C, estimated at 1.4- 22% of QE at room temperature. Evolutionary optimization, as practiced in pilot operations, has proven to be an effective strategy for identifying & resolving technical issues, including the following: ALD-GCA-MCP functionality, photocathode QE, HV stability, HV connectivity, optimized stack height, improved leak integrity, and use of fused silica windows.

A major program milestone was achieved on Sept 14 with the production of a fully functional LAPPD #9. The tile has a 10nm thick Aluminum photocathode that has a rather low QE of  $1X10^{-3}$  to  $1X10^{-5}$  in the UV spectral range. At the time of this manuscript, LAPPD #9 had maintained vacuum integrity for a period of 1 week as evidenced by test measurements. The detector demonstrated a stable spark free operation at a gain of  $2X10^7$  providing sensitivity to single photoelectrons in the UV spectral range. More detailed evaluation tests are on-going including measurements of time and spatial resolution, gain uniformity across the area and long term gain stability.

Continued development of LAPPD<sup>TM</sup>s and ALD-GCA-MCPs is being supported by grants from DOE, NASA and the National Geospatial Intelligence Agency (NGA) targeting specific requirements for high energy physics (HEP), space applications, and reduced cost. Areas of research include the development of alternate glasses for reduced noise, large-area ALD-GCA-MCPs with smaller pores for improved timing resolution, curved ALD-GCA-MCPs for compact detector devices for terrestrial and space applications, ceramic body LAPPD<sup>TM</sup>s for improved durability and expanded design options for LAPPD anodes, and the development of ALD coatings that reduce thermal coefficient of resistance (TCR).

A transition from "commissioning stage" to "exploitation" and routine pilot production is expected after identified component and hardware improvements are implemented and process experience is gained. Improvements currently being implemented include: improved UHV window transfer hardware, enabling down pressure application during sealing, and improved window cleanliness & masking for optimal PC QE and preservation of seal surfaces. ALD-GCA-MCPs (203mm X 203mm) are being fabricated routinely and are available for LAPPD<sup>TM</sup> and other applications. Incom remains on-plan to deliver prototype "all glass" LAPPD<sup>TM</sup>s to early adopters.

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A.U. Mane et el., "A novel atomic layer deposition method to fabricate economical and robust large area microchannel plates for photodetectors", Physics Procedia, 37, 722-732 (2012).

J. W. Elam, A. U. Mane, J. A. Libera, J. N. Hryn, O. H. W. Siegmund, J. McPhate, M. J. Wetstein, A. Elagin, M. J. Minot, A. O'Mahony, R. G. Wagner, H. J. Frisch, W. M. Tong, A. D. Brodie, "Synthesis, Characterization,"

- and Application of Tunable Resistance Coatings Prepared by Atomic Layer Deposition", ECS Trans. 2013 volume **58**, issue 10, 249-261, DOI: 10.1149/05810.0249ecst
- A. U. Mane and J. W. Elam, "Atomic Layer Deposition of W:Al<sub>2</sub>O<sub>3</sub> Nanocomposite Films with Tunable Resistivity", Chem. Vap. Deposition, **19**, 186–193, (2013). doi: 10.1002/cvde.201307054
- <sup>4</sup> A. O'Mahony et al., "Atomic layer deposition of alternative glass microchannel plates", J. Vac. Sci. Techn. A, 34, 01A128 (2016); doi: 10.1116/1.4936231
- Frisch, H.J., et al "RF strip-line anodes for Psec large-area MCP-based photodetectorsH Nuclear Instruments and Methods A71 (2013) 124.
- M. Wetstein, B.W. Adams, A. Elagin, H.J. Frisch, R. Obaid, E. Oberla, A. Vostrikov, R.G. Wagner, J. Wang, Timing characteristics of Large Area Picosecond Photodetectors Nuclear Instruments and Methods in Physics Research A 795 (2015) 1–11
- Henry J. Frisch, et. al., A Brief Technical History of the Large-Area Picosecond Photodetector (LAPPD) Collaboration, http://arxiv.org/abs/1603.01843 March 2016
- <sup>8</sup> Report of the Particle Physics Project Prioritization Panel, http://lbne-old.fnal.gov/pdfs/Threefrontiers.pdf
- DOE, DE-SC0009717 "LAPPD Commercialization Fully Integrated Sealed Detector Devices"
- <sup>10</sup> Attribution for sketch: Ertley MCP Imaging Detectors for High Dynamic Range 2016\_SORMA\_
- Attribution for sketch: O.H.W. Siegmund
- <sup>12</sup> O.H.W. Siegmund et al., NSS/MIC, IEEE.N45-1, pp.2063-2070 (2011)
- <sup>13</sup> O.H.W. Siegmund, Advanced Maui Optical and Space Surveillance Technologies Conference (2012).
- <sup>14</sup> S.J. Jokela et al., Physics Procedia, 37, 740 747 (2012)
- <sup>15</sup> Siegmund LAPPD2 Hermetic Package Godparent Review, April 03, 2013
- <sup>16</sup> A. Lyashenko, "Incom LAPPD", Presented at the Pico-Second Workshop, Kansas City, Sept 15-18 2016