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Physics with and Physics of Atomic Layer Deposited Nanofilms

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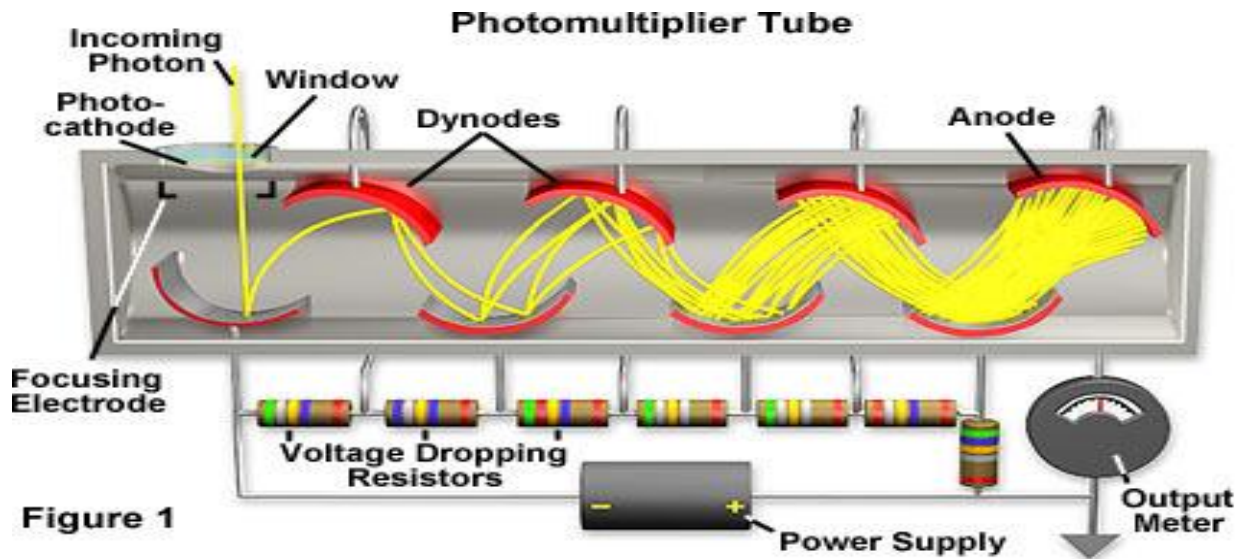


Outline -

- ◆ Background
 - ◆ Photomultiplier (PMT) Photo Detectors (c1930)
 - ◆ Microchannel Plate Photomultiplier (MCP-PMT)
 - ◆ Microchannel Plate (MCP) manufacturing (c1960)
- ◆ The MCP-PMT in HEP
- ◆ Why does the MCP-PMT fail?
- ◆ Failure mitigation strategies
- ◆ Atomic Layer Deposited (c1970) nanofilm technology for MCPs
- ◆ The role of emissive layer charging
- ◆ Analytical results
- ◆ MCP-PMT lifetime test results
- ◆ What does the ALD nanofilm do?
- ◆ Conclusions and future work

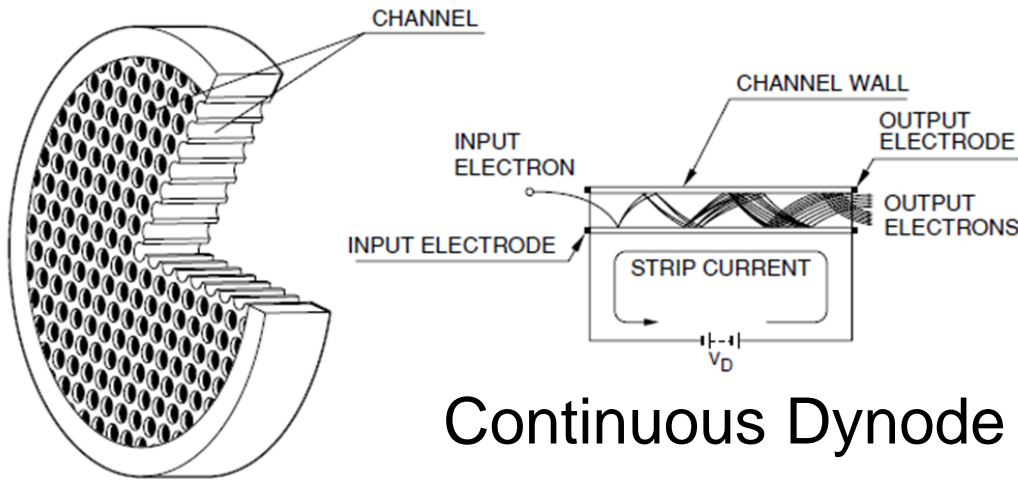
PMT principles (1937)

- Capable of single photon detection.
- Quantum Efficiency (QE) of 20-50%
- Low noise amplification of electrons
- Robust, long life
- BUT:**
 - Insufficient spatial & temporal resolution (>1ns)**



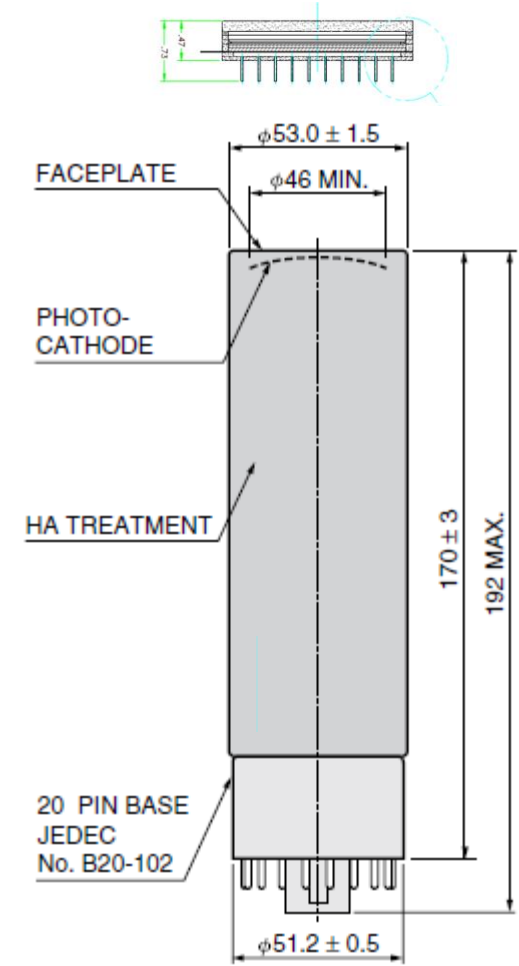
Discrete
Dynode

MCP (1960s) and MCP-PMT (2000)



Continuous Dynode

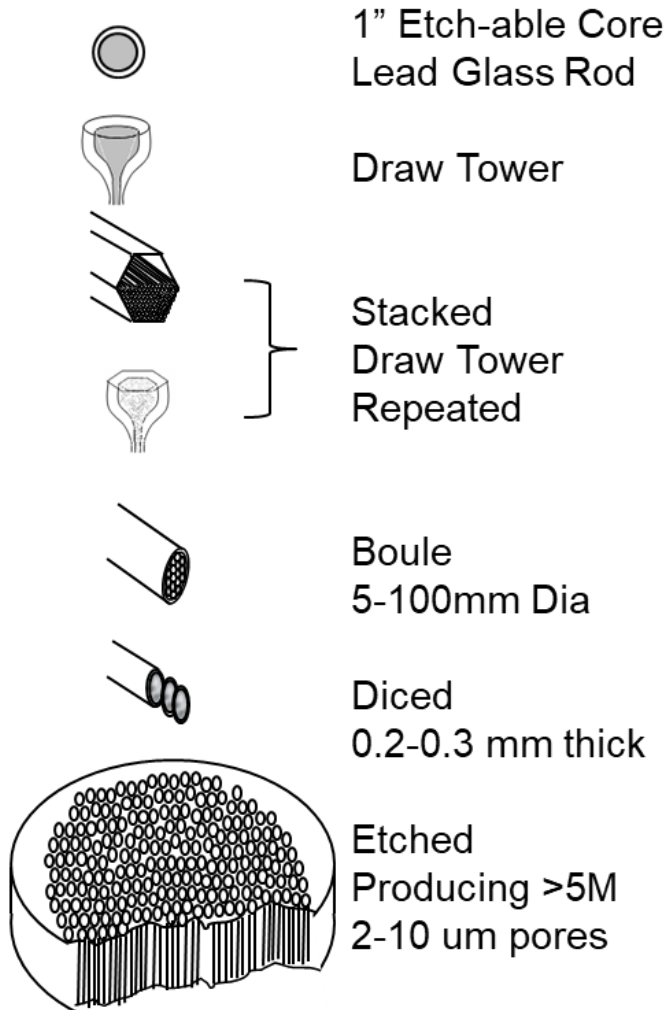
- MCP-PMT vs. PMT
 - Temporal resolution 10-100x BETTER
 - Spatial resolution 10-100x BETTER
- **BUT:**
 - **Poor Lifetime: Photocathode degraded by impact with Positive ions**



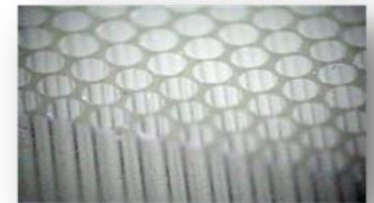
MCP Manufacture: Composition determines manufacturing **AND** structure **AND** function

| Material | Wt % |
|--------------------------------|------|
| 8161 | 8161 |
| PbO | 50.5 |
| SiO ₂ | 38 |
| K ₂ O | 5.44 |
| Rb ₂ O | 3.7 |
| BaO | 2.05 |
| Na ₂ O | 0.34 |
| Cs ₂ O | 0.29 |
| Al ₂ O ₃ | 0.24 |
| Bi ₂ O ₃ | 0.04 |
| Fe ₂ O ₃ | 0.02 |
| B ₂ O ₃ | 0 |
| MgO | 0 |
| CaO | 0 |
| As ₂ O ₃ | 0 |
| Sb ₂ O ₃ | 0 |

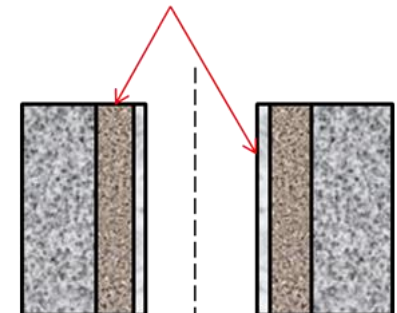
Substrate Fabrication



Substrate Functionalize



H₂ Firing
 conduction & emission layer produced simultaneously



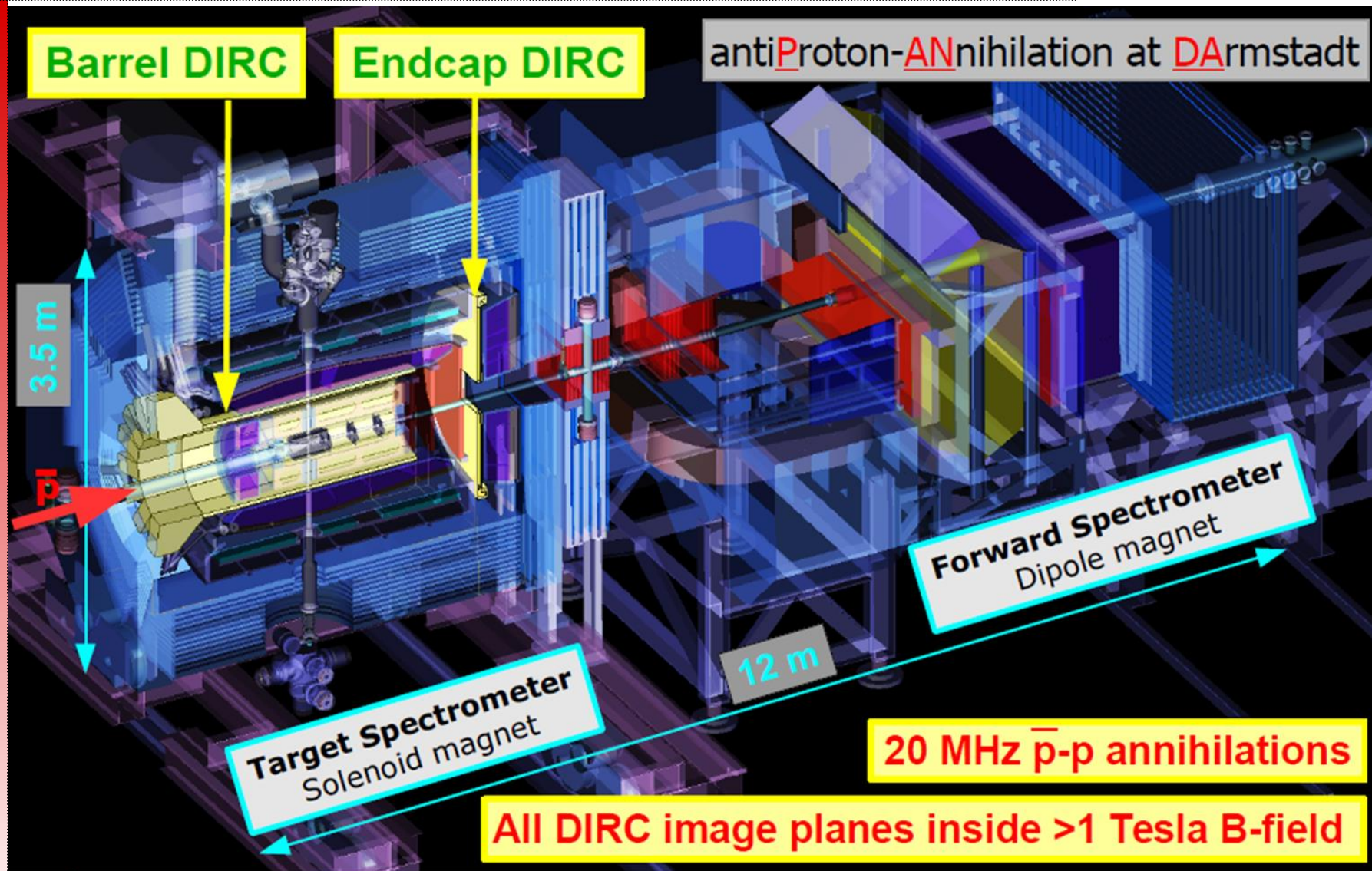


Applications in Particle Physics

- ◆ Latest generation particle physics experiments require
 - ◆ Efficient single photon detection in high magnetic fields (>1 Tesla)
 - ◆ Capability of dealing with very high photon rates (MHz/cm²)
 - ◆ Picosecond time resolution and \sim mm position resolution
 - ◆ Compact form factor
- ◆ MCP-PMT ideal sensor for use in particle identification
 - ◆ Cherenkov detectors (RICH, DIRC)
 - ◆ Time-of-Flight detectors (TOF)
- ◆ Examples of physics goals addressed
 - ◆ Understanding dynamics of heavy quarks (b-physics in Belle & LHCb)
 - ◆ Search for new states of matter (hybrid & glueball resonances in PANDA)
 - ◆ Spin structure of the nucleon (addressed with planned electron-ion-collider)



\bar{P} ANDA Detector at FAIR



MCP-PMTs for PANDA DIRCs: Lifetime is the issue

❖ MCP-PMTs for PANDA DIRCs

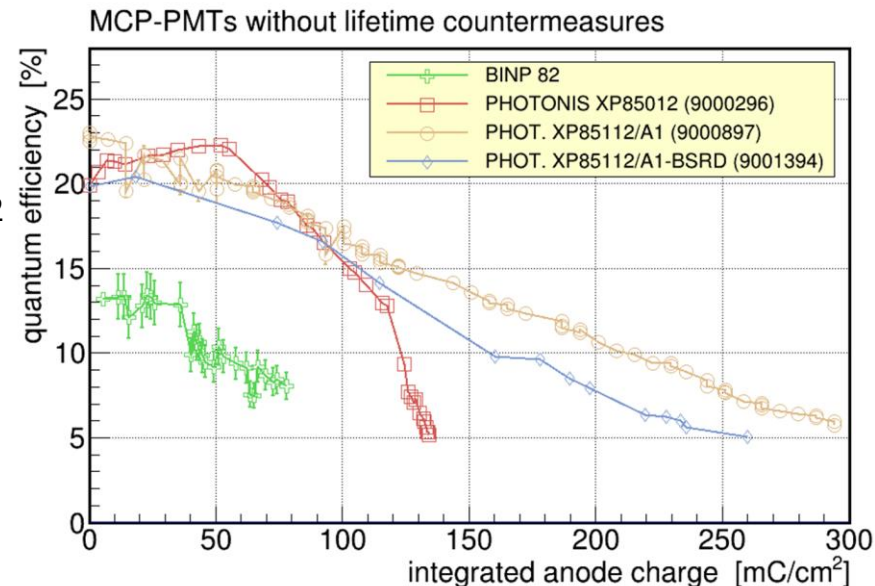
- ❖ MCP-PMTs are the only suitable sensors for PANDA
- ❖ Compact and available as multi-anode devices
- ❖ Single photon detection in B-fields of 1 – 2 Tesla
- ❖ Time resolution <50 ps
- ❖ Low dark count rates

❖ Barrel DIRC Lifetime

- ❖ Photon rate: ~ 200 kHz/cm²
- ❖ **10yr anode: 5 C/cm²**
- ❖ Pixel size: $\sim 6 \times 6$ mm²

❖ Endcap DIRC Lifetime

- ❖ Photon rate: ~ 1 MHz/cm²
- ❖ **10yr anode: >5 C/cm²**
- ❖ **Pixel size: $\sim 0.5 \times 16$ mm²**



Lifetime status 2011 – < 200mC/cm²

Barrel DIRC: 3-6 months

Endcap DIRC: < 3 months

Why do MCP-PMTs fail?

- ⚡ Positive ions desorb during operation¹
 - ⚡ Alkali from bulk migrates to the pore surface^{1,2}
 - ⚡ Residual gases (H, H₂O, CO, CO₂) from MCP fabrication process
- ⚡ **MCP** permanent gain degradation due to desorption of alkalis
- ⚡ **Photocathode** - Cs:O layer sensitive to damage
 - ⚡ QE drops below useful value (~50% of original)

1. A.M. THEN & C.G. PANTANO, "FORMATION AND BEHAVIOR OF SURFACE LAYERS ON ELECTRON EMISSION GLASSES," Journal of Non-Crystalline Solids 120 (1990) 178-181
 2. B. PraEek and M. Kern, AES studies of active intrachannel surface in microchannel plates Applied Surface Science 70/71(1993) 169-171

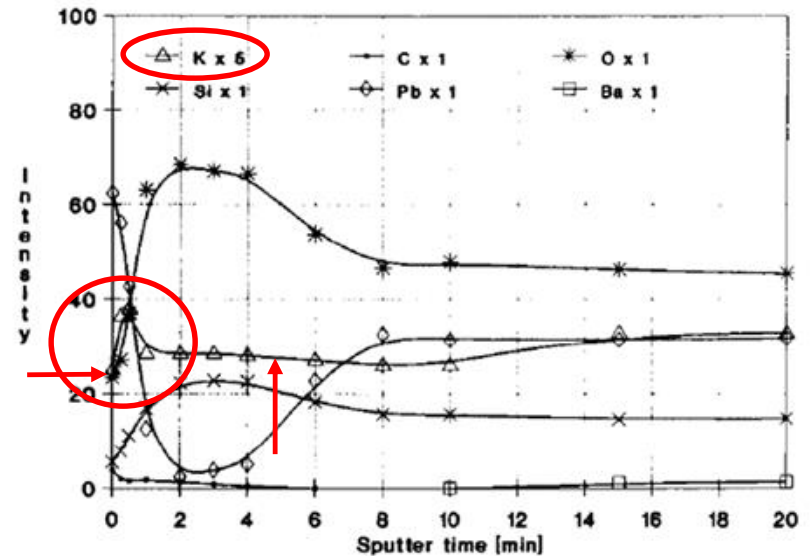


Fig. 2. AES sputter depth profiles of the important elements in the active surface of the channel before ageing of the MCP. The measured peak-to-peak intensity is normalized to the corresponding pure elemental target value which is set at 100.

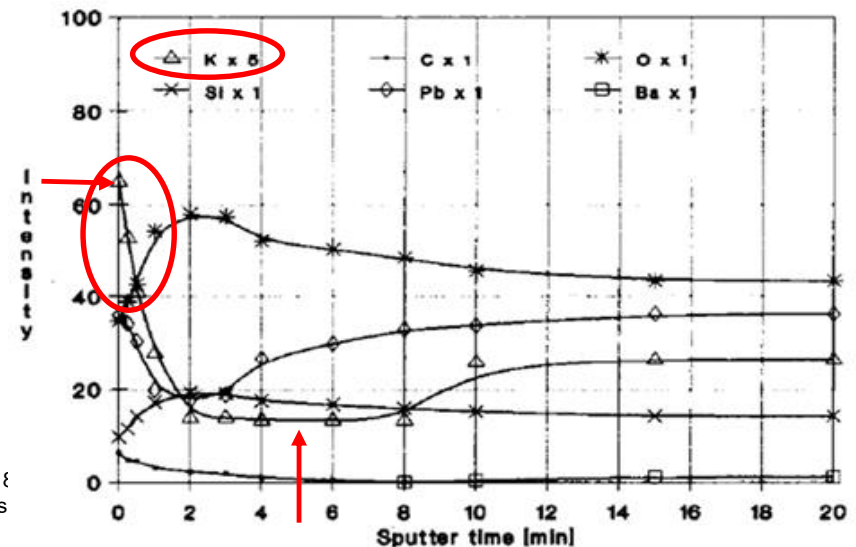


Fig. 3. As fig. 2, but now after ageing of the MCP.

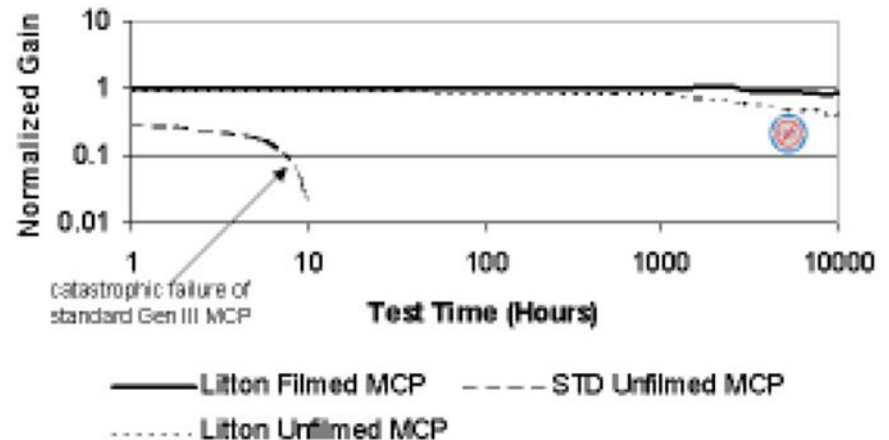
What has been done?

- ⚡ Bake 350°C for > 8hrs
- ⚡ Electron scrub of MCP
 - ⚡ < 1C/cm² stabilize gain
- ⚡ Gen III Nightvision
 - ⚡ Al₂O₃ ion barrier
 - ⚡ "Long Life" Glass
 - ⚡ Electronic gating
 - ⚡ Bulk conductive glass
- ⚡ High Energy Physics
 - ⚡ Al₂O₃ ion barrier
 - ⚡ Package re-design
 - ⚡ PC robustness
 - ⚡ Grid field filter
- ⚡ Can ALD help? Yes!
 - ⚡ HAR – 60:1
 - ⚡ High surface areas (m²)

| | Wt % | |
|--------------------------------|------|-------|
| Material | 8161 | L3 NG |
| PbO | 50.5 | 26.6 |
| SiO ₂ | 38 | 37 |
| K ₂ O | 5.44 | 0 |
| Rb ₂ O | 3.7 | 0.85 |
| BaO | 2.05 | 19.7 |
| Na ₂ O | 0.34 | 0 |
| Cs ₂ O | 0.29 | 4.12 |
| Al ₂ O ₃ | 0.24 | 1.35 |
| Bi ₂ O ₃ | 0.04 | 2.48 |
| Fe ₂ O ₃ | 0.02 | 0 |
| B ₂ O ₃ | 0 | 2.8 |
| MgO | 0 | 0.85 |
| CaO | 0 | 2.25 |
| As ₂ O ₃ | 0 | 0.65 |
| Sb ₂ O ₃ | 0 | 0.28 |

US Pat 6271511

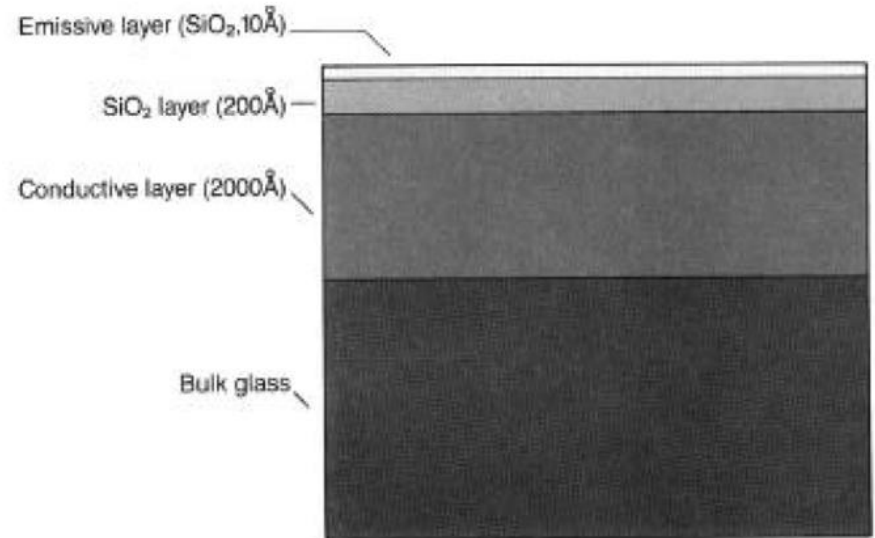
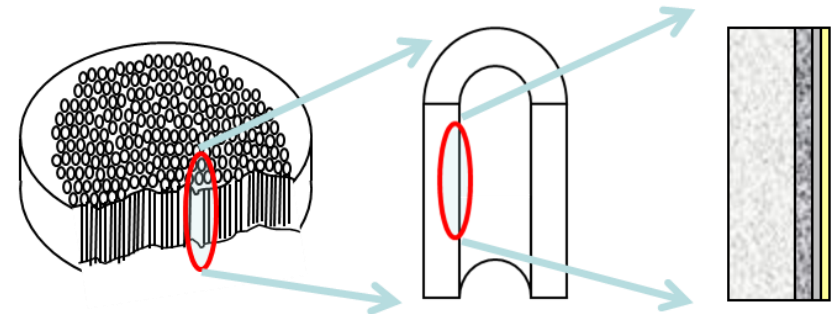
Reliability Test Data (Gain)



J. P. Estrera et al, "Long Lifetime Generation IV Image Intensifiers with Unfilmed Microchannel Plate," Proceedings of SPIE Vol. 4128 (2000)

Experimental: MCP ALD nanofilm technology on 4 substrates (different compositions and vendors)

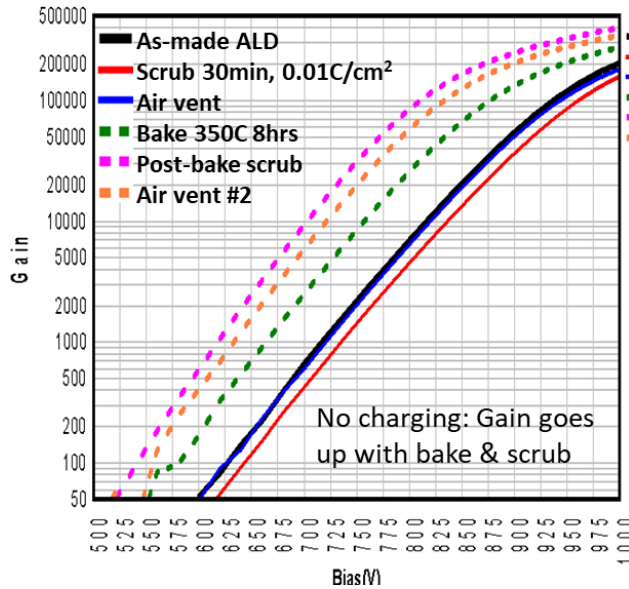
- Al₂O₃ Emissive on fired glass MCP substrates (Samples A-D)
 - TMA, H₂O, 150C, 38Å
 - Gain performance correlates with fired MCP performance
- Charging behavior - insulator
 - Charging** - permanent gain reduction in vacuum – recovers only with forced dissipation (e.g. exposure to atmosphere)
 - Charging** observed as a function of film thickness
 - Charging** observed as a function of glass composition



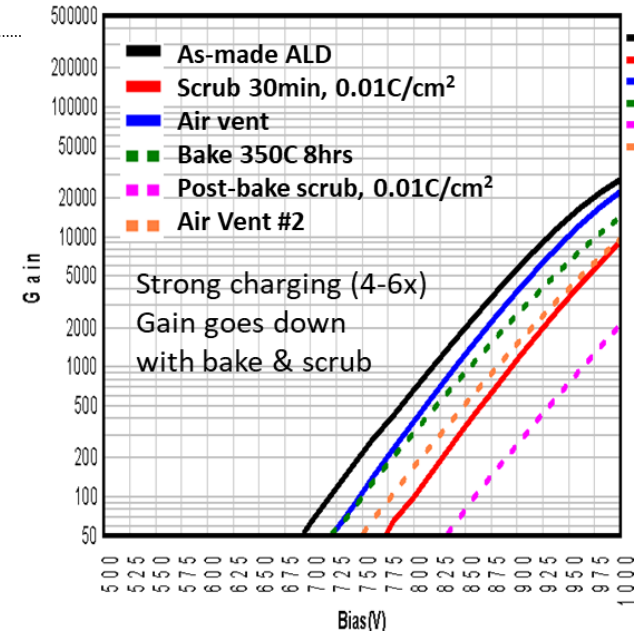
Channeltron electron multiplier handbook (Burle)

Experimental: Test sample charging results

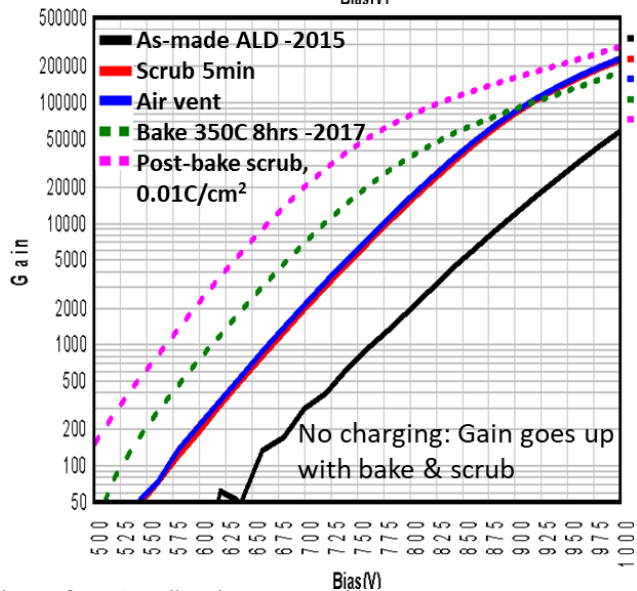
A



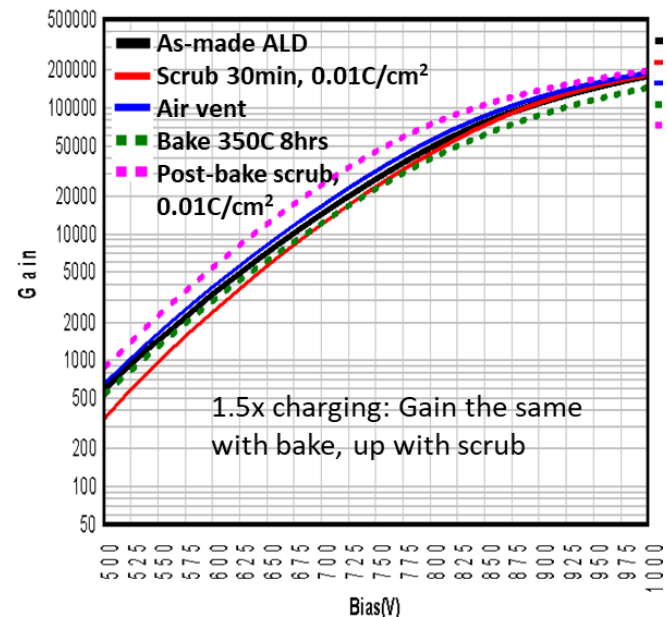
B



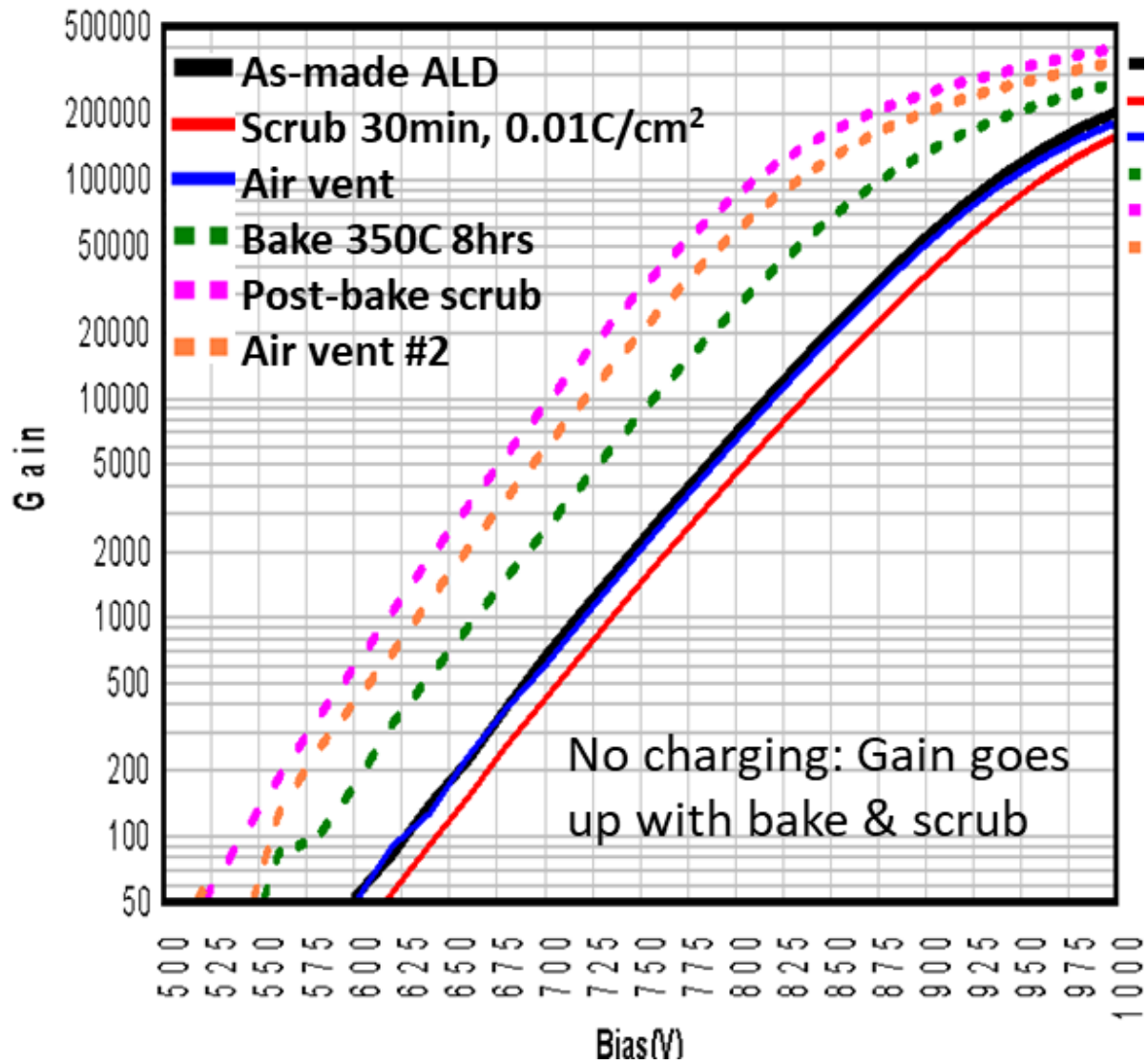
C



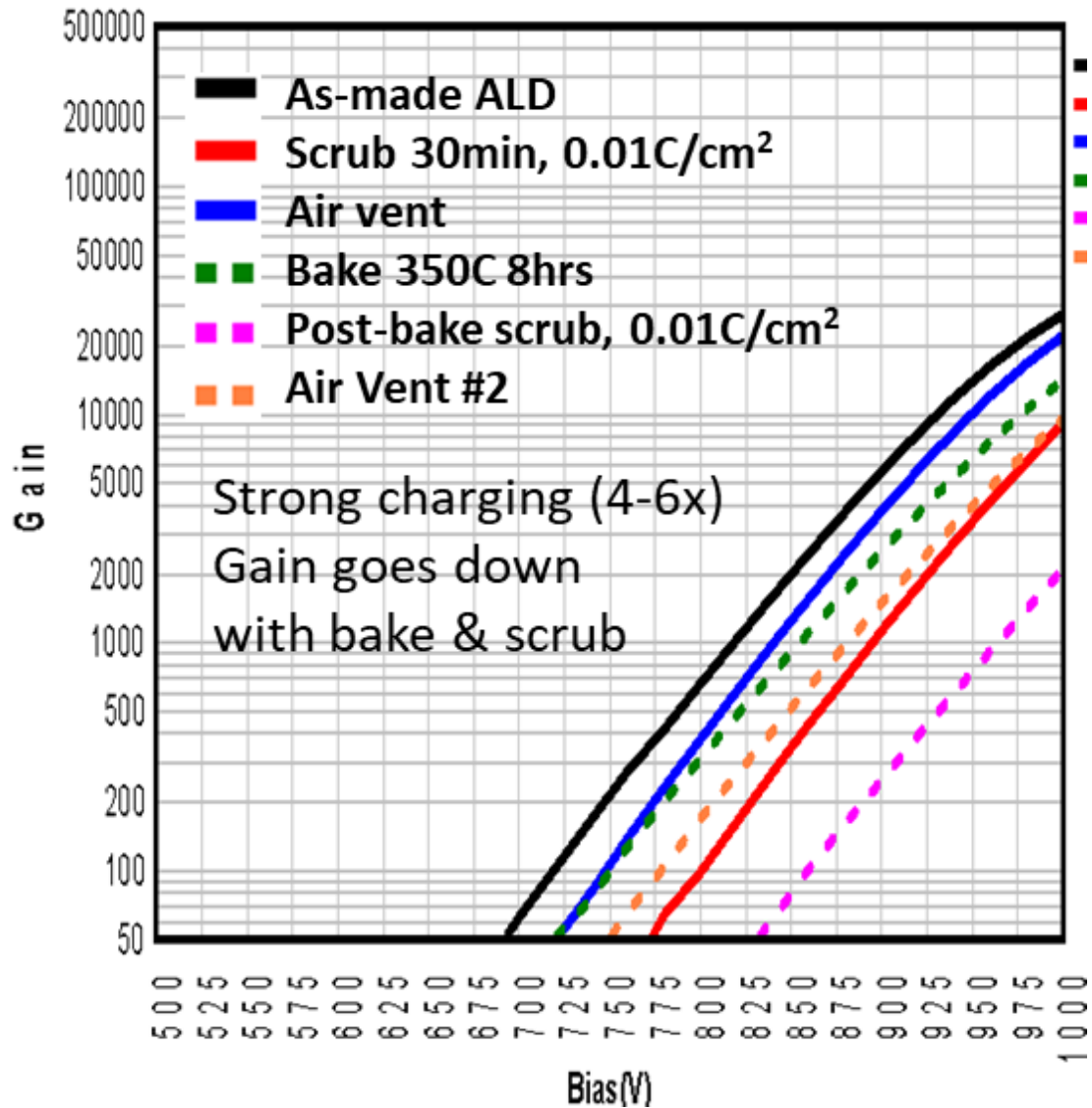
D



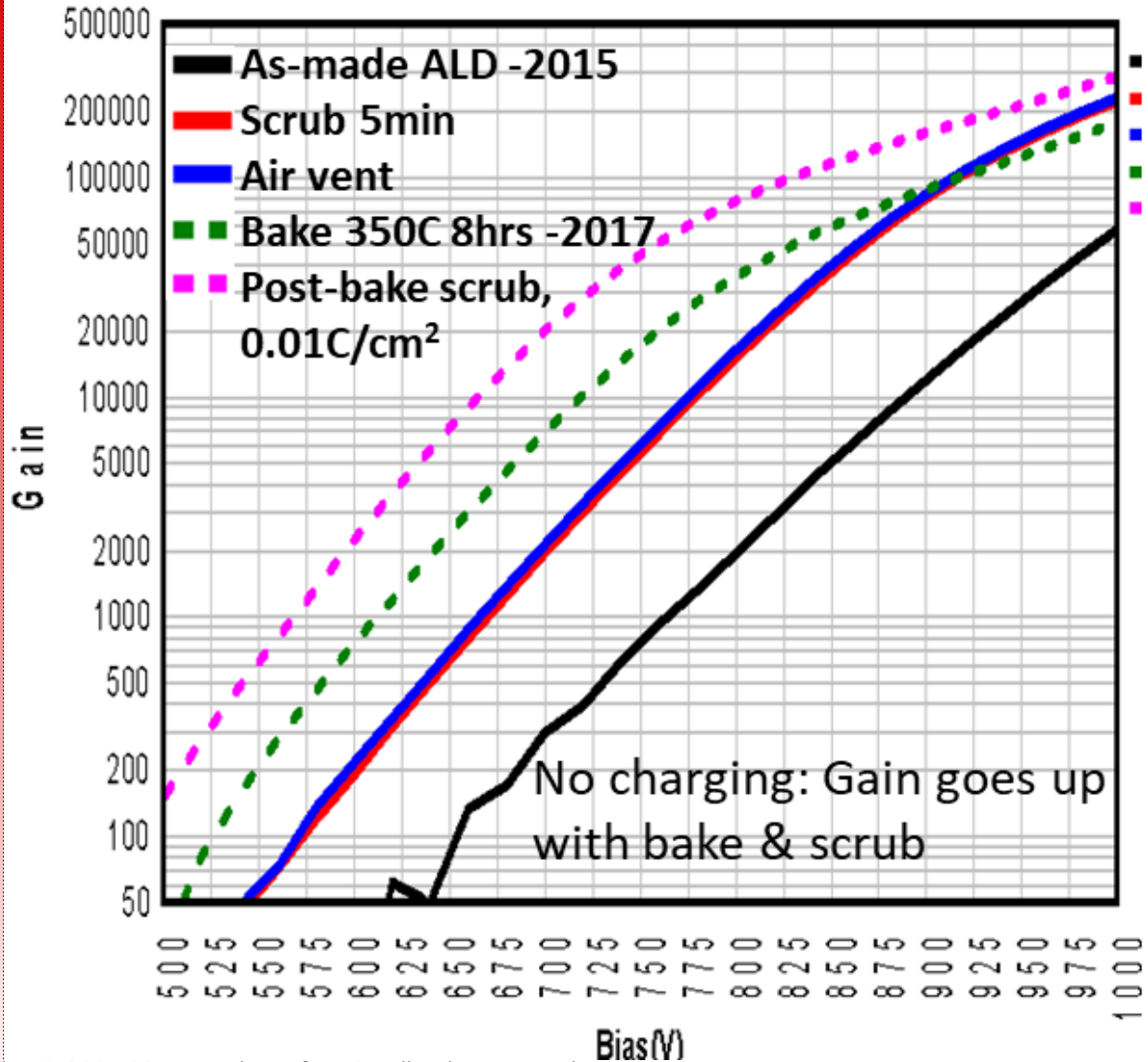
Experimental: Test Sample Charging Results – Sample A



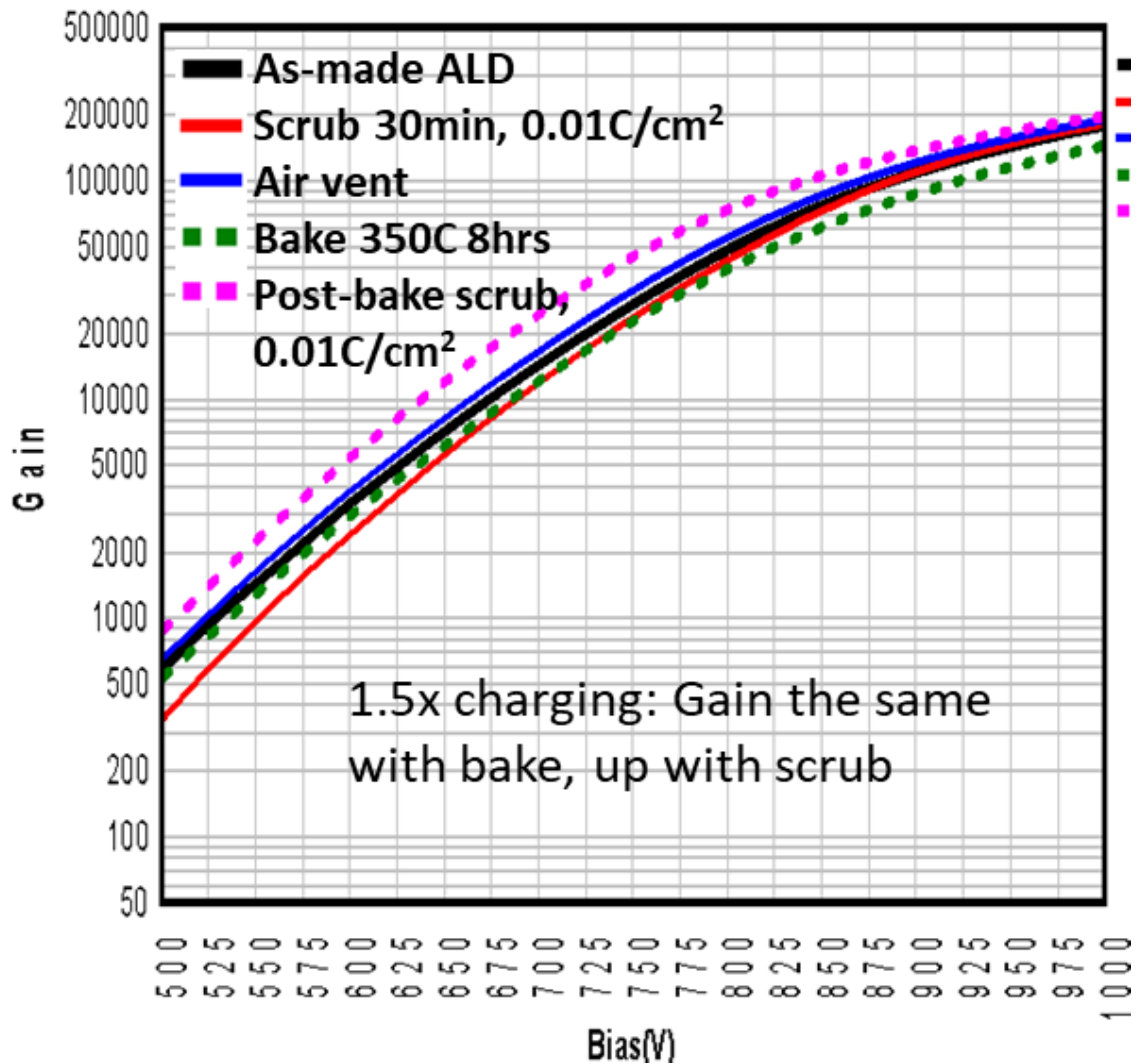
Experimental: Test Sample Charging Results – Sample B



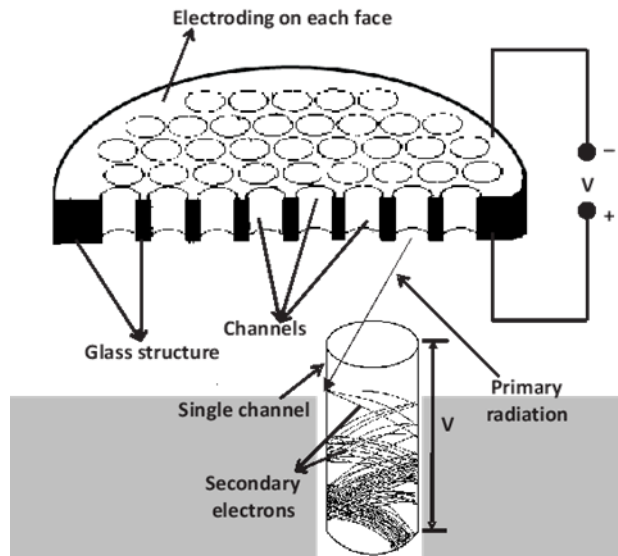
Experimental: Test Sample Charging Results – Sample C



Experimental: Test Sample Charging Results – Sample D



Experimental: FESAM Analytical methodology



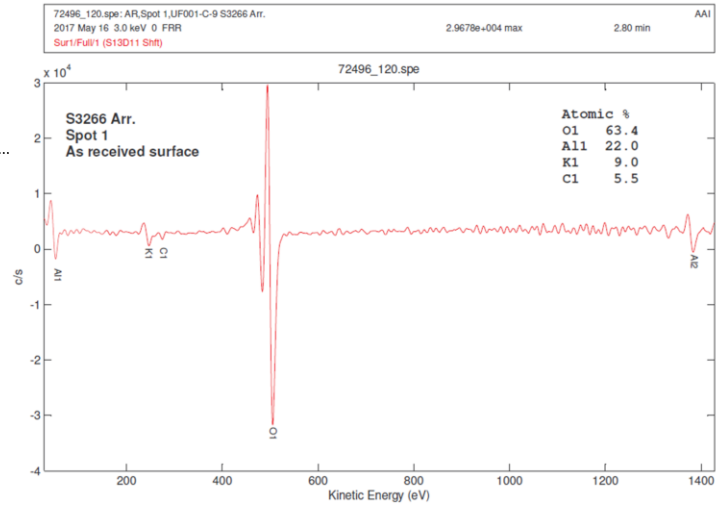
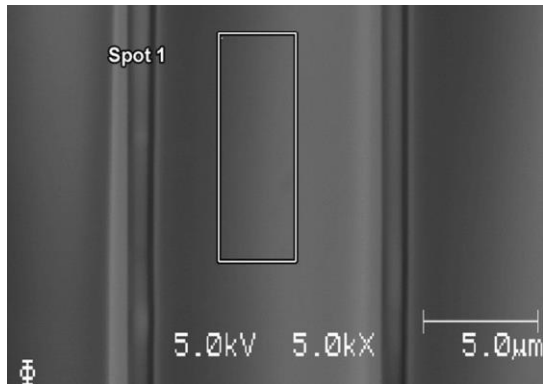
Elemental composition of MCP glass^a.

| Z | Element | Weight percent |
|----|---------|----------------|
| 82 | Pb | 47.8 |
| 8 | O | 25.8 |
| 14 | Si | 18.2 |
| 19 | K | 4.2 |
| 37 | Rb | 1.8 |
| 56 | Ba | 1.3 |
| 33 | As | 0.4 |
| 55 | Cs | 0.2 |
| 11 | Na | 0.1 |

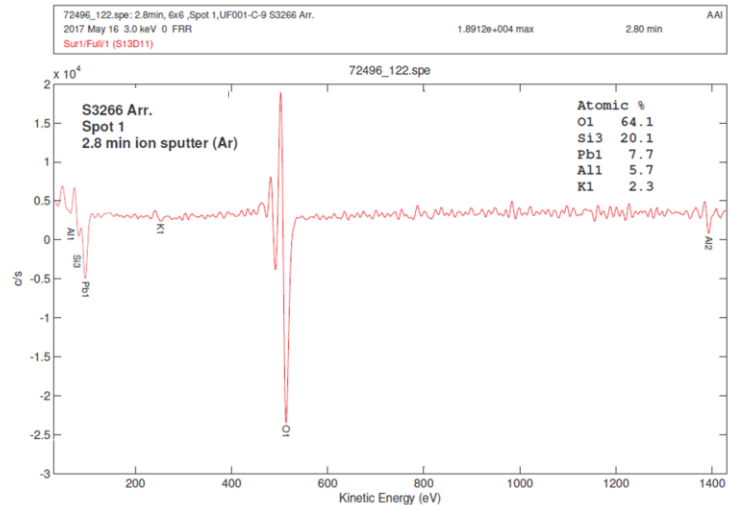
^aDensity - 4.0 g./cm³.

- Available literature analyses performed on processed, flat, Pb-glass test coupons
- This study on ALD nanofilmed and tested MCP device pore.
- MCP critical dimensions
 - Pore diameter (d) = 10 μ m
 - Pore pitch = 12 μ m
 - Pore Aspect Ratio = 60:1
 - NiCr Electrode 2d into pore
- PHI 670 Auger Nanoprobe
 - Multipak and CASAXPS software
 - Conditions - 3kV, 10nA tilt $\sim 30^\circ$
- Ar⁺ ion gun sputter at 4kV and a raster area of 6x6mm.

Experimental: FESAM Results



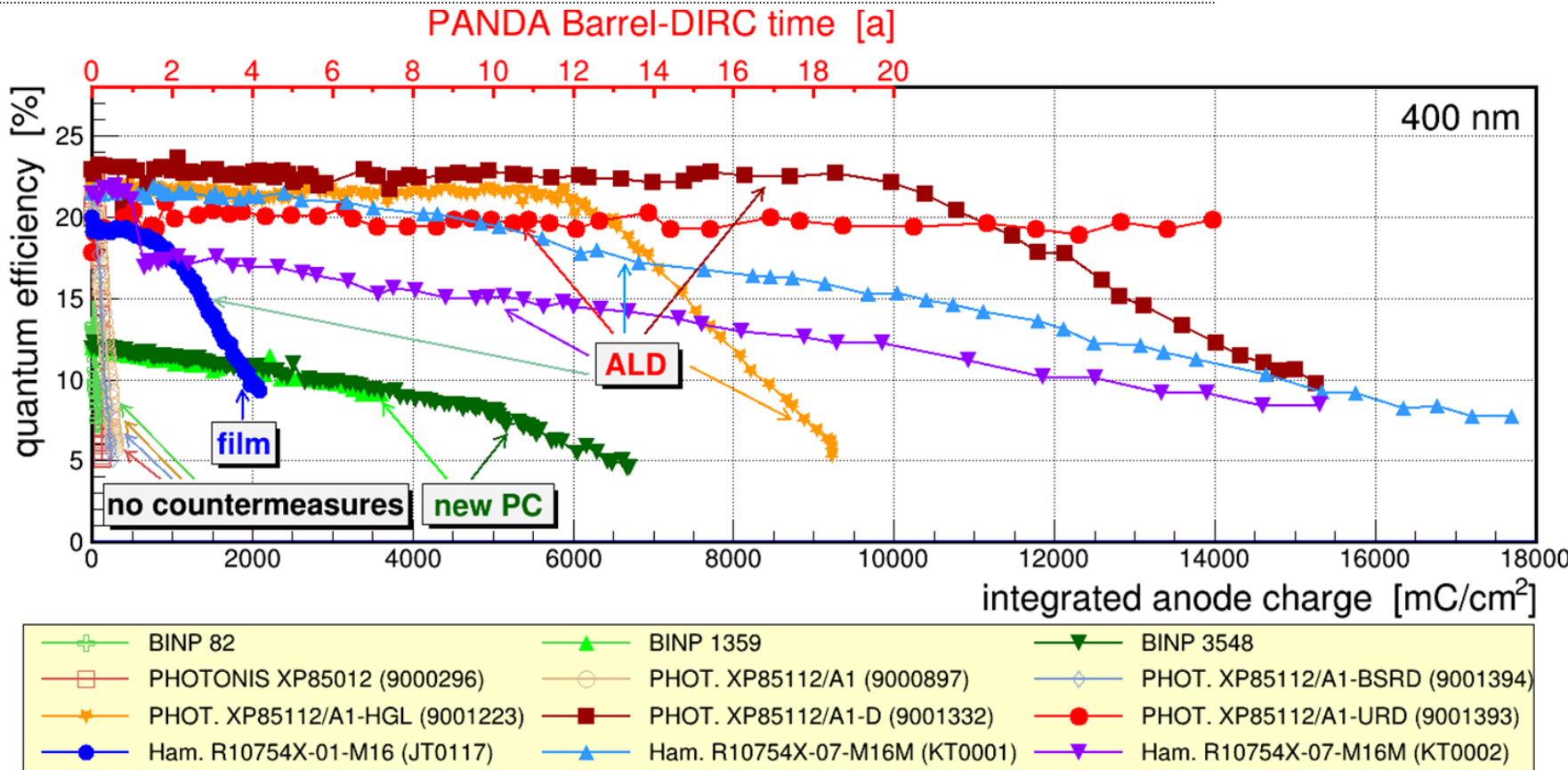
- ◀ Data from Sample **A**
 - ◀ Survey area within MCP pore indicated by rectangular box
 - ◀ Profile Data 12s ($\sim 24 \text{ \AA}/\text{min}$)
 - ◀ Information depth few nanometers at 3kV



Summary Table: % Potassium vs Charging

| SN | A | B | C | D |
|-----------------|-----|-----|-----|-----|
| Gain multiplier | 10 | 0.2 | 5 | 1.5 |
| Potassium % | 4.5 | 0 | 4.1 | 0* |

Experimental: Lifetime of MCP-PMTs (06/2017)



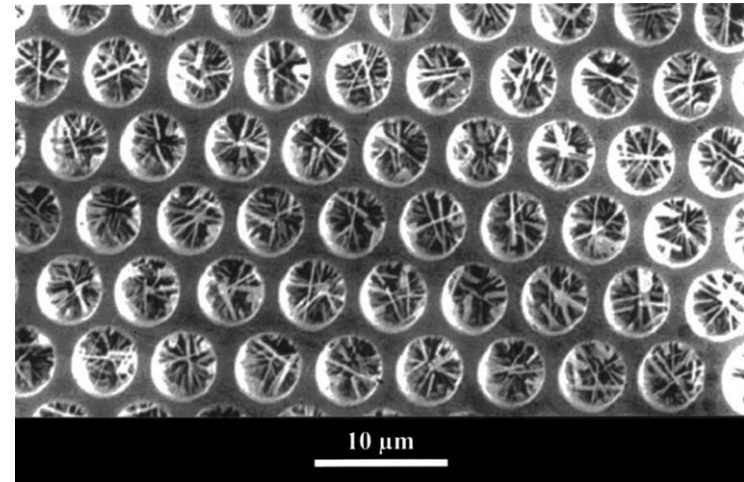
- ⚡ No countermeasures: unusable after <200 mC/cm²
- ⚡ Modest lifetime improvements with film and new PC
- ⚡ “Quantum leap” in lifetime (x70) with ALD:
 - ⚡ **best performing device at 14 C/cm²** – No QE Loss!

Discussion: 38Å of Al₂O₃ = No photocathode degradation! SPECIAL K

- ◀ MCP-PMT test results
 - ◀ Sample **D** formulation >10C/cm²
 - ◀ Sample **C** up to 20C/cm² in other testing¹

- ◀ Literature findings:
 - ◀ Monolayer of K on pore surface²
 - ◀ ALD of KCl in MCP devices⁴
 - ◀ ALD of potassium aluminate³

- ◀ Characterization & Test results
 - ◀ Substrate (**B**) cannot be optimized by Al₂O₃ layer properties alone
 - ◀ Al₂O₃ Conductivity improved with presence of K
 - ◀ Final gain is correlated to initial fired substrate gain



¹ https://indico.cern.ch/event/393078/contributions/2195231/attachments/1332045/2002282/RICH2016_matsuoka.pdf

²Then, A.M., Pantano, C.G., "Formation and behavior of surface layers on electron emission glasses", J. of Non-Cryst. Solids Vol. 120(1-3), 178-187(1990)

³ E. Østreg et al, "Atomic layer deposition of sodium and potassium oxides: evaluation of precursors and deposition of thin films," Dalton Trans., 2014, 43

⁴ Zhang et al, "Potassium chloride nanowire formation inside a microchannel glass array." Appl. Phys. Lett. 86, 263110 2005



Summary and Future Work

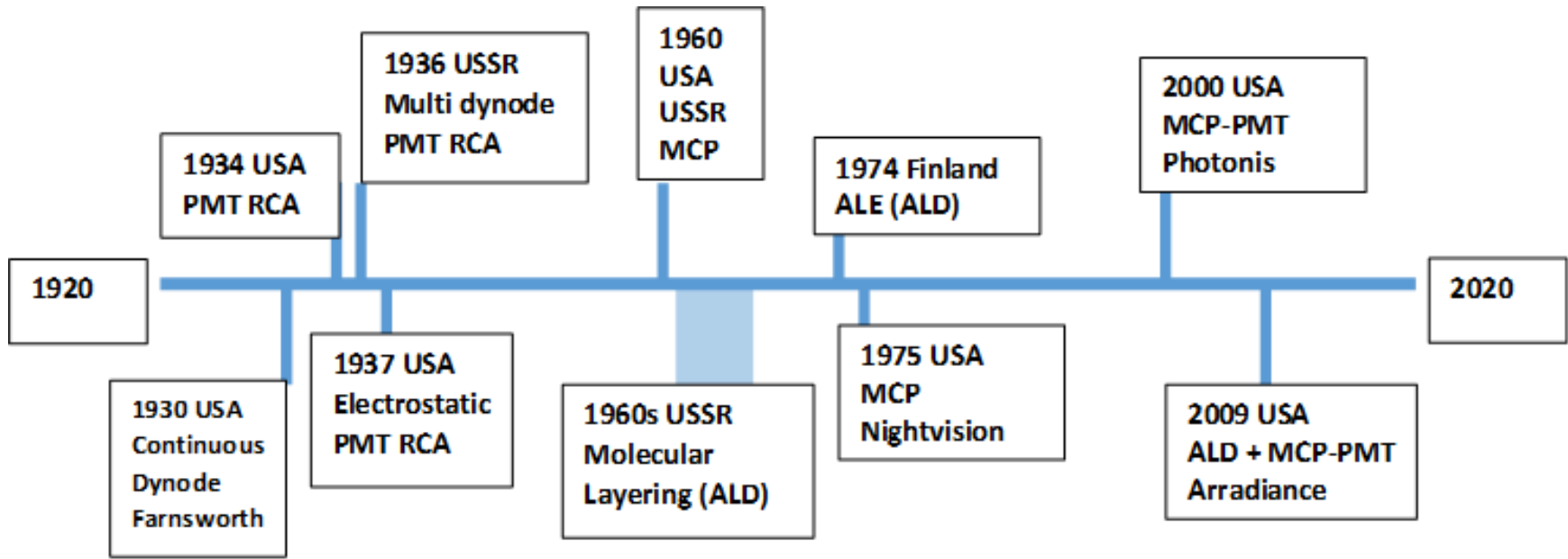
Summary

- ◀ FESAM results, in pore, demonstrate viability of method and correlate well with literature from flat test samples
- ◀ Presence of Alkali, specifically K, in Al_2O_3 film correlates with best operational MCP-PMT performance
- ◀ TMA + H_2O reacts with K
 - ◀ To lock down K & produce conducting "aluminate"
- ◀ Resistive & Emissive ALD layers best lifetime 14 C/cm² (24yrs Barrel DIRC)
 - ◀ remove other residual species (H, H_2O , CO, CO_2) due to the eliminating of the firing step and the thicker encapsulation

Next Steps – Still many Questions...

- ◀ Perform analytical testing "aluminate" composition
 - ◀ SEM evidence of additional "reactions" on surface
 - ◀ Role of KOH in sustaining the reaction
- ◀ Study aluminate formation through deposition temperature
- ◀ Investigate methods to optimize "long Life" glasses

Acknowledgements – Thank You for Your Attention!



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 - ◀ Photonis Inc.
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 - ◀ University of Chicago HEP
- ◀ Darlene Reid
 - ◀ Analytical Answers

