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First LAPD Collaboration Meeting
Argonne National Lab
October 15-16, 2009

STATUS OF AAO SUBSTRATES AT SYNKERA

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OUTLINE

MCP DEVELOPMENT PROGRAM AT SYNKERA

- AAO/ALD AS A MATERIALS PLATFORM
- CERAMIC MCP WITH INTRINSIC AND MICROMACHINED CHANNELS
- **LAPD SUBCONTRACT - LARGE AREA MCP SUBSTRATES, INTRINSIC CHANNELS**

PROGRESS ON AAO SUBSTRATE / "INTRINSIC" CHANNELS

- CHANNEL DIAMETER AND SPACING
- CHANNEL ENTRANCE AND CHANNEL SHAPE
- SCALE-UP

STATUS OF RELATED WORK

- ALD RESISTANCE MODIFICATION
- MICROMACHINED SUBSTRATES

SUMMARY

DEVELOPMENT OF AAO MCP AT SYNKERA

SYNKERA OVERVIEW

Core strengths

- Nanotechnology, materials science and engineering, chemical sensing and separation, microfabrication

Current Products/Services

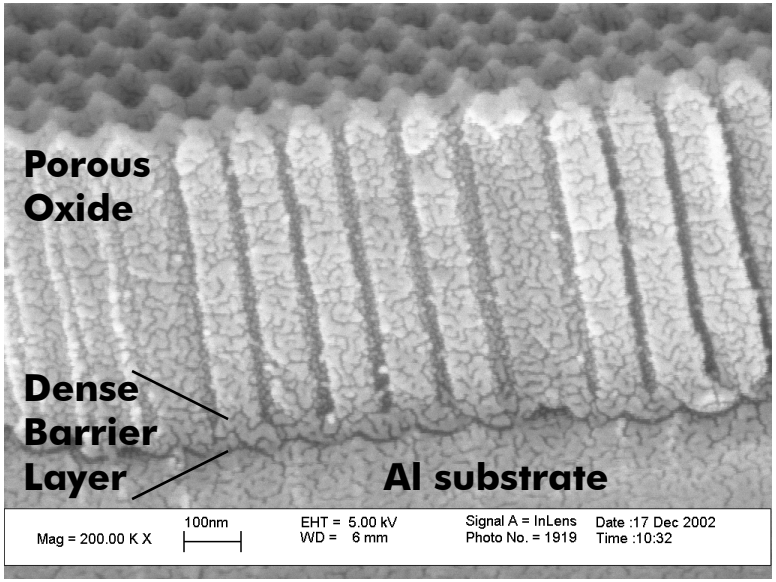
- Gas sensors and modules
- Ceramic membranes
- Contract research

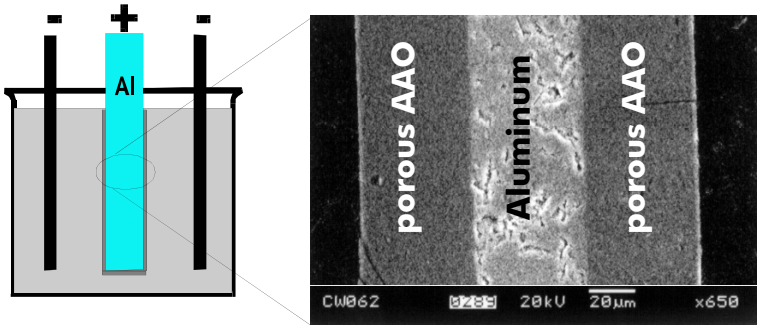
Markets

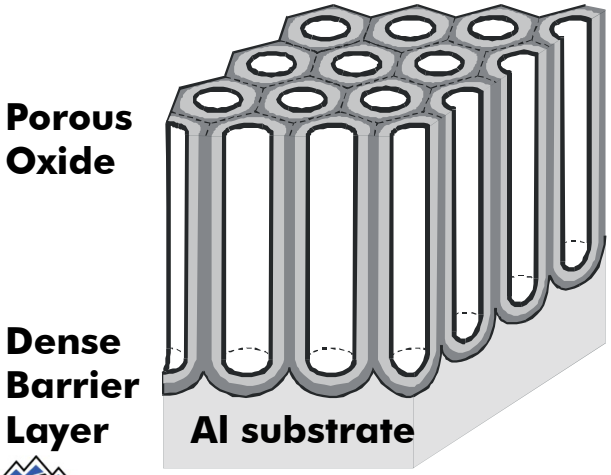
- Industrial health and safety, air quality
- Gas separation, hydrogen generation, R&D
- Emerging markets: clean and alternative energy, bio- and life sciences, filtration

Mission - bring practical products to the market through advanced knowledge and precision engineering of materials.

ANODIC ALUMINUM OXIDE (AAO) INTRODUCTION







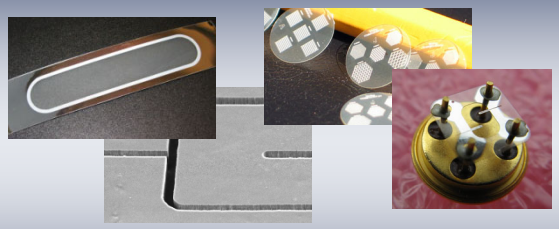
- High quality self-organized material with regular nanoporous lattice
- Uniform & aligned arrays of cylindrical nanopores
- Formed by anodic oxidation of Al in certain electrolytes
- Tunable parameters
  - Pore diameter: 5 - 300 nm
  - Pore density: 10^{12} -10^{8} cm^{-2}
  - Thickness: 0.1 - 300 µm
- Scalable, manufacturing-friendly
- Platform for nano/microfabrication

AAO/ALD-BASED PRODUCTS AT SYNKERA

Hydrogen Generation
- Separation membranes
- Catalytic membranes
- Adsorbents & Reactors
- Fuel cells, distributed
- Oil and gas, petrochemical

Gas Sensors
- Industrial health & safety
- Air quality control
- Environmental monitoring
- Homeland security

PRODUCT PLATFORM



- **Nano / microscale engineering**
- **Scalable manufacturing**

Liquid Filtration Membranes
- Life Sciences
- Bioanalysis
- Ultra/nanofiltration
- Water purification

Energy Conversion
- Gen-3 PV for solar cells
- Photocatalysts
- Radiation Detection
- Photonics

Ceramic Microdevices
- **Ceramic MCPs**
- Microcomponents for harsh environments
- Chips for cell culturing, bioanalysis & bioimaging
- Bio- and life sciences

SYNKERA MCP DEVELOPMENT PROGRAM

AAO as Ceramic MCP Substrate

- Opportunity: low cost, high resolution replacement of glass MCPs
- Started in 1999 with intermittent funding; sustained program since 2005
- **MCPs with Micromachined Channels (channel size 3-10 μm)**
  - Status: functional MCPs with channel diameter < 4 μm produced and tested
  - Current effort: 25 mm diameter prototypes
- **MCPs with Intrinsic Channels (sub-μm channel size)**
  - Current NSF Phase II SBIR project – development of ***functional MCPs***
  - **LAPD subcontract** – development of ***large MCP substrates***

ALD as a Tool for MCP Development

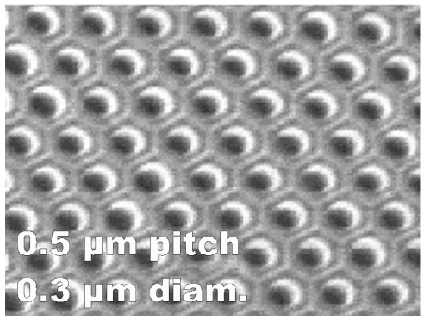
- Synkera used ALD for development of AAO-based products since 2000
  - Modification of AAO geometry and properties
  - Nanocomposites, membranes & reactors, gas sensors, energy conversion materials
- First ALD-modified MCP prototypes produced in 1999-2000
- In-house ALD facility in 2005

Partners

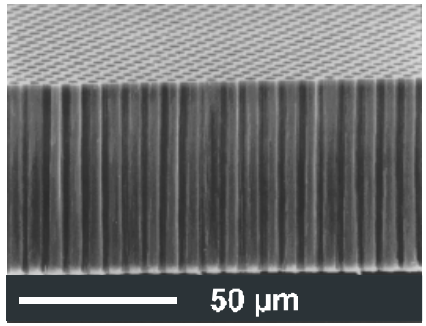
- Steven George (University of Colorado) - ALD process development
- Ossy Siegmund, Anton Tremshin (Space Science Lab, UC Berkeley) - testing

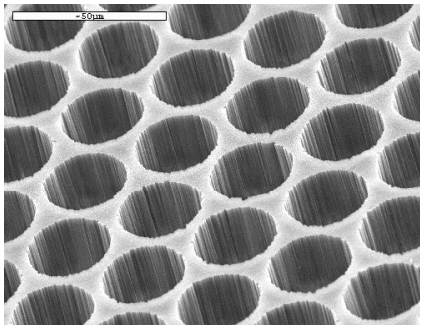
AAO MCP SUBSTRATE
- STATUS OF INTRINSIC CHANNELS -

AAO CHANNEL DIAMETER

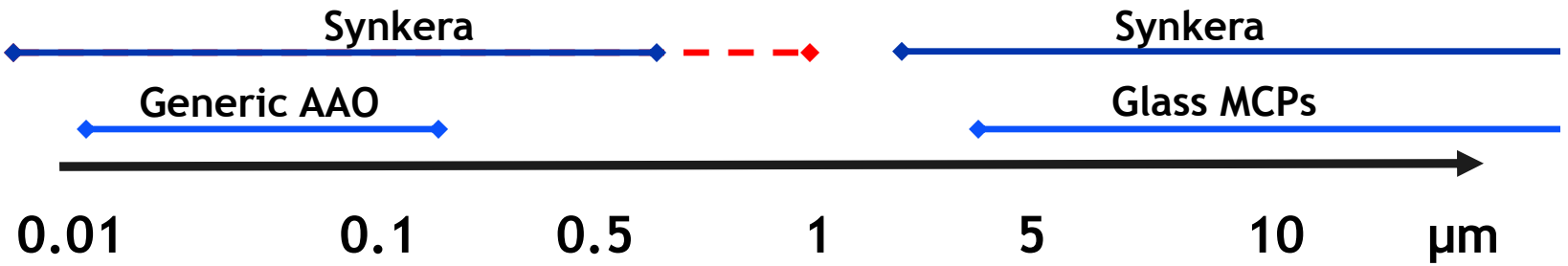


Intrinsic Pores





Micromachined Channels



Main challenges:

- Implement 0.5 – 1 µm diameter channels
- Scale to dimensions and formats of detector targeted by LAPD
- Produce MCP substrates for fabrication of functional MCP prototypes
- Validate performance and cost reduction potential
- Define the path for high volume low-cost manufacturing.

SYNKERA SUBCONTRACT

Status: contract negotiation wrapped up, proposal submitted to ANL

Overall goal: develop ceramic MCPs substrates targeting the need for next generation low-cost large-area detectors.

Year 1 – Development of Required Channel Structure

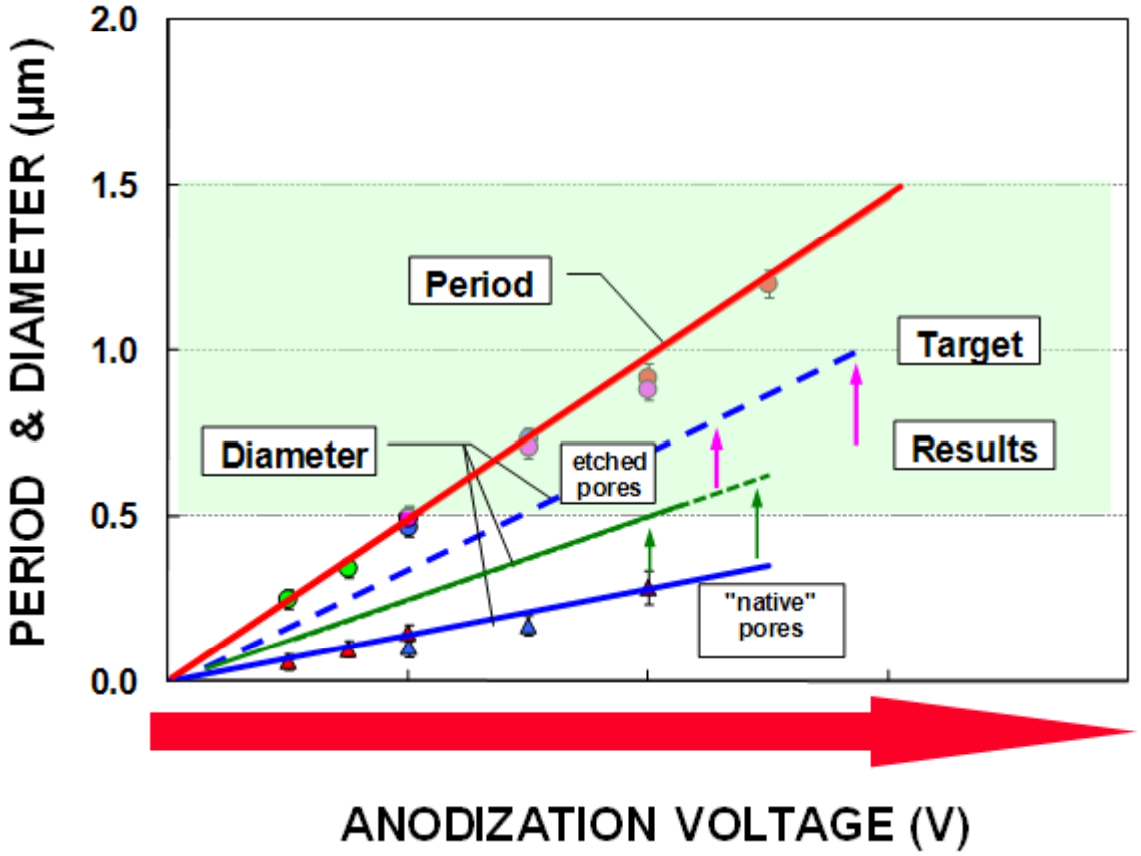
- Objective: maximize the channel diameter and enable a funnel-shaped opening, while maintaining well-aligned channels.
- Deliverables:
  - Demonstrate AAO with channel diameter ≥0.5 μm, OAR ≥60%, L/D of 50-100
  - MCP substrates for the LAPD project team (32.8 mm, qty ≥ 15) targeting above specs
  - Initial cost projections for 8”x8” AAO substrates

Year 2 Option – Support of MCP Development and Scale-Up

- Objective: enable targeted MCP performance via development of AAO substrates; limited scale-up to validate the size (8”x8”) and cost reduction potential.
- Deliverables:
  - Demonstrate channel diameter ≥0.7 μm, funnel-shaped opening, OAR≥65%, and L/D 40-100.
  - MCP substrates for the LAPD project team (32.8 mm, qty ≥ 40) targeting above specs
  - Optional: scaled 8”x8” “demo” substrates
  - Validated cost projections for 8”x8” AAO substrates.

This project will benefit from prior and related IP, established facilities and on-going R&D and scale-up efforts at Synkera.

ACHIEVING TARGET PITCH & DIAMETER



Pore Period:

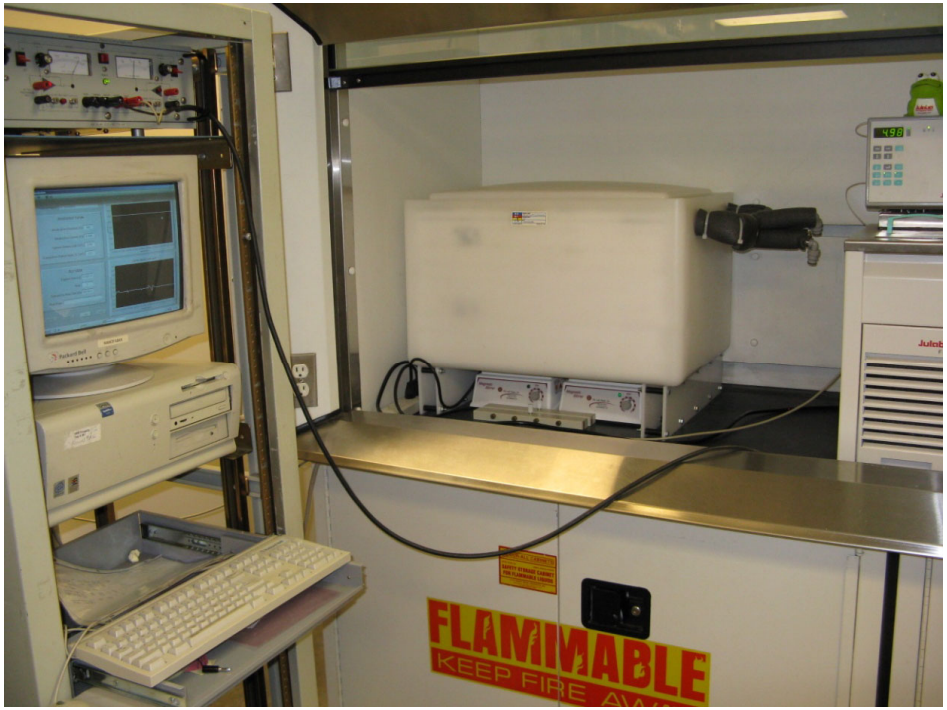
- Proportional to anodization voltage
- Does not depend on electrolyte, temp.

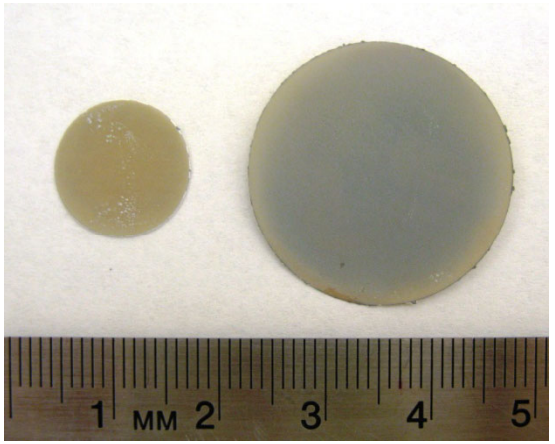
Pore Diameter:

- Increases with voltage, [H+] and temp.

Required new electrolytes & procedures - developed in '06-09 under NSF funding

ANODIZATION FACILITY





13 and 25 mm AAO with larger pores

Computer controlled process

- Voltage, current, charge, temperature
- Feedback on process parameters
- Bath maintenance
- Complex process algorithms

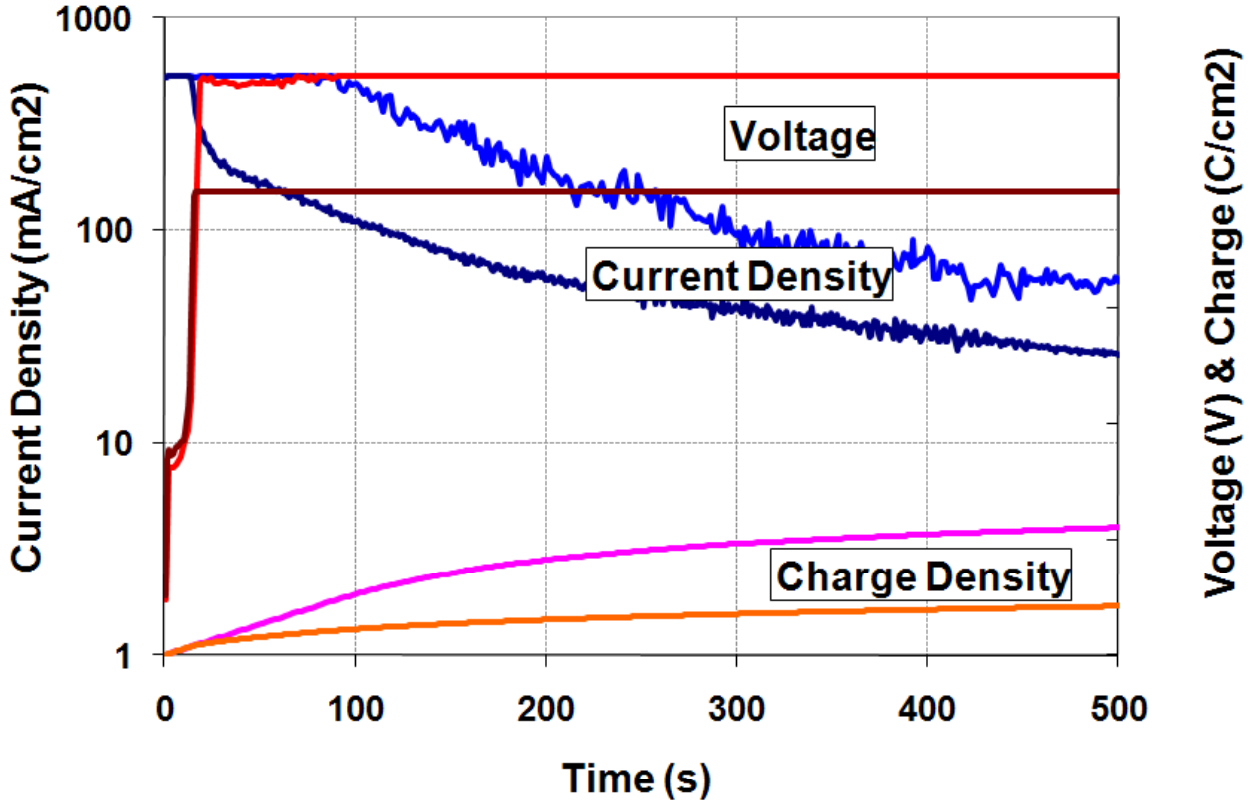
Development Bath

- 1-2 of 25 mm MCPs in one run

Prototyping bath

- 4-16 of 25 mm MCPs in one run

ANODIZATION AT HIGH VOLTAGE



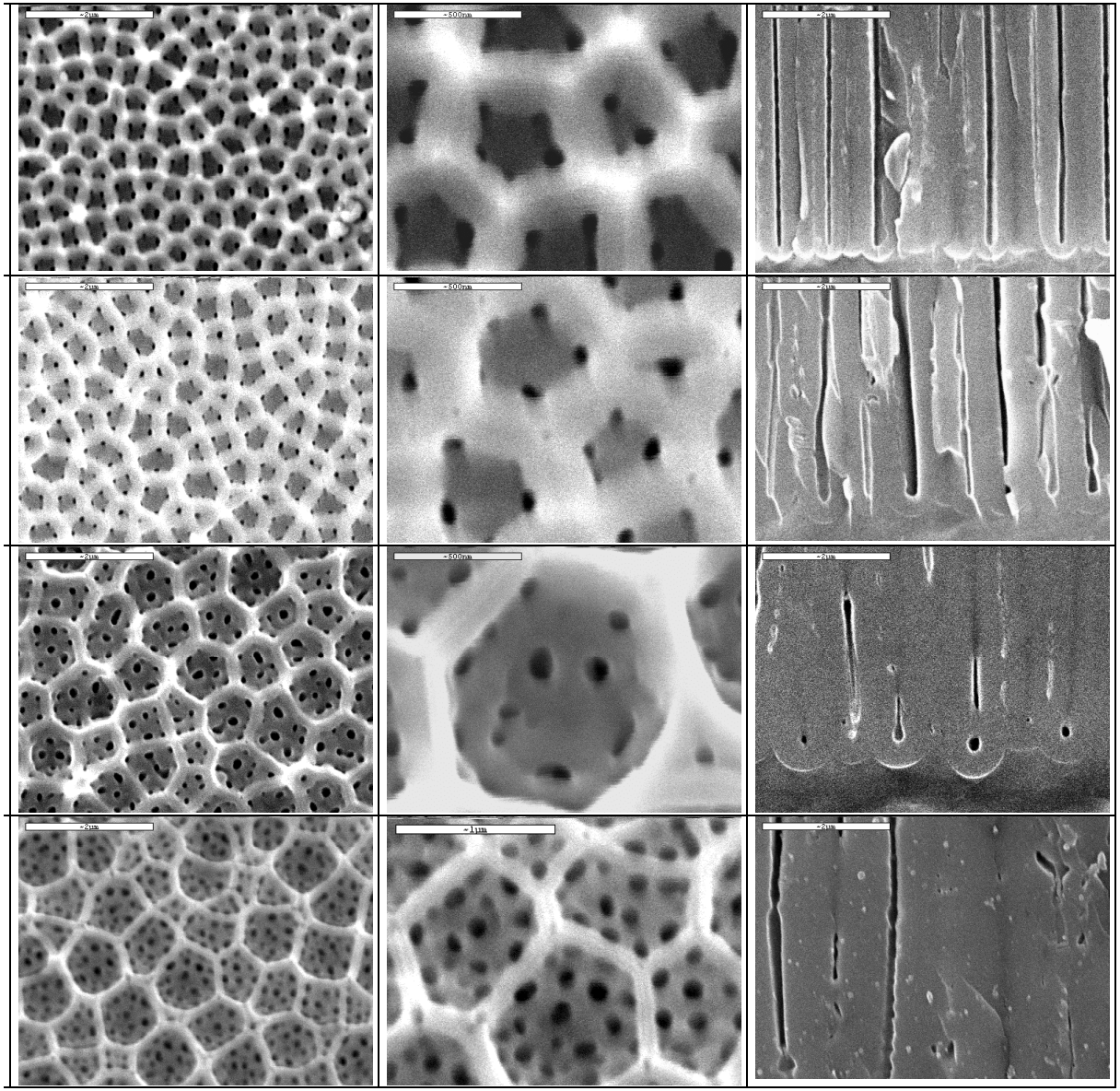
- Need to avoid voltage ramp (thus achieve final pore structure)
  - Challenges - high current and rapidly changing load
- Solutions (NSF project) : new electrolytes, two-stage anodization

U

1.5xU

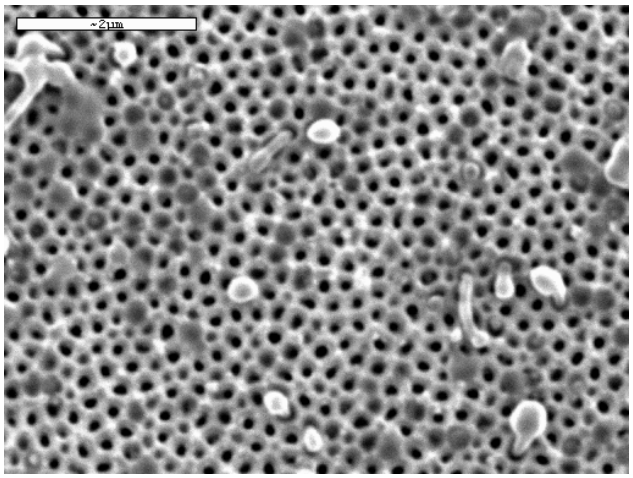
2xU

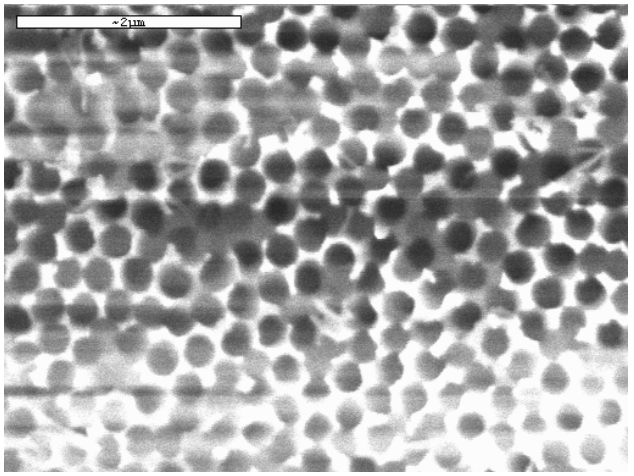
2.5xU



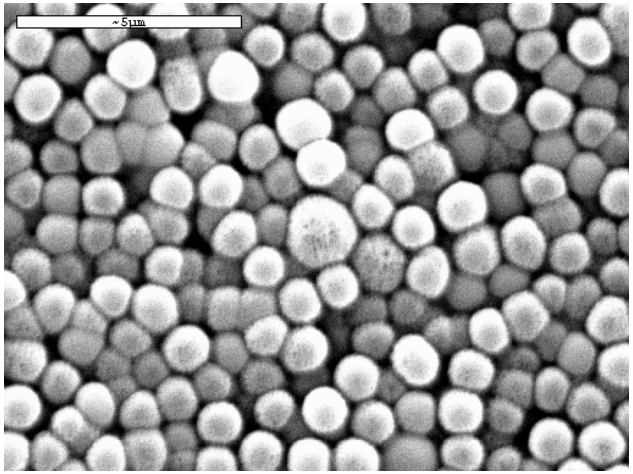
Before channel opening

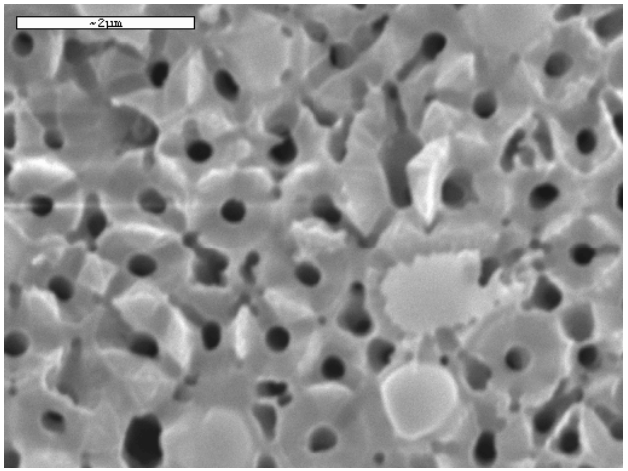
After channel opening





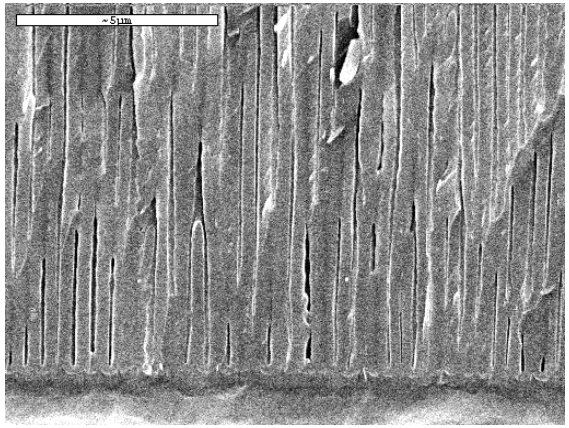
Top Surface



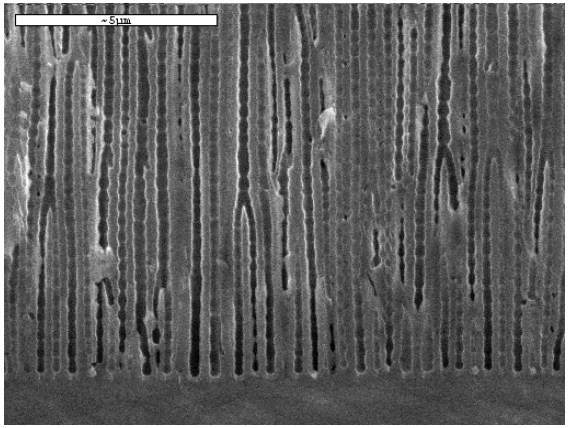


Bottom Surface

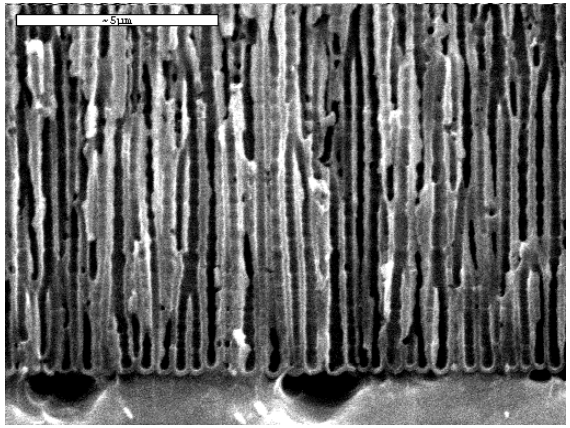
CONFORMAL CHANNEL ETCHING



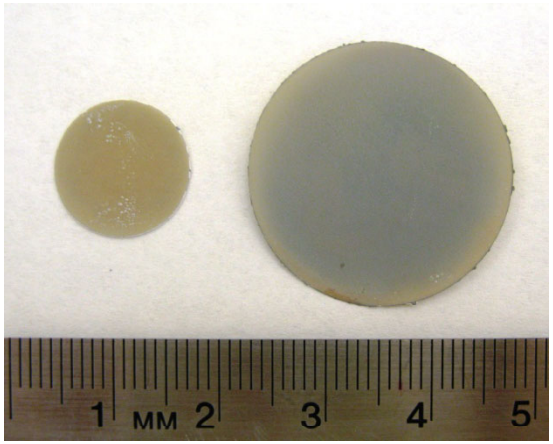
As anodized

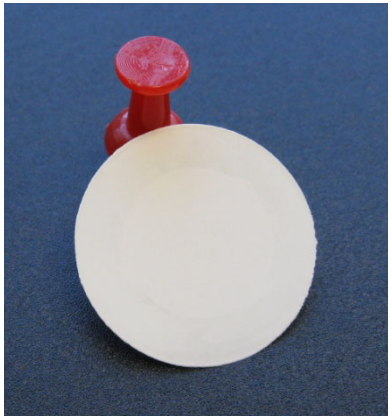


90 min

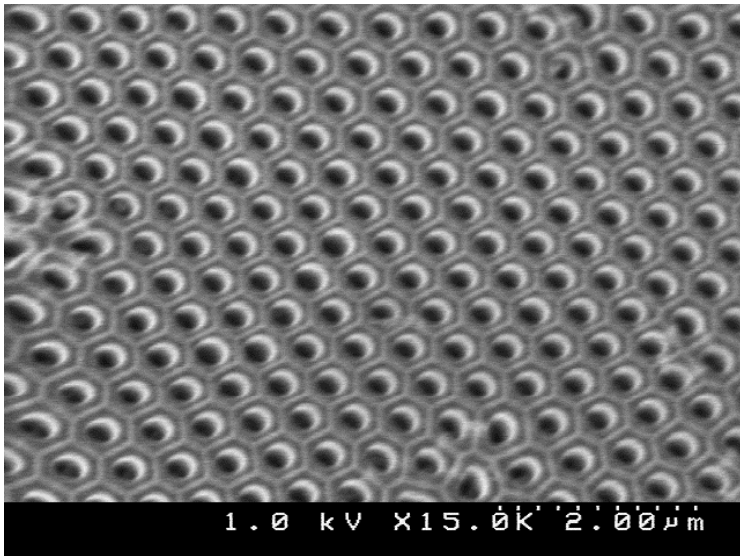


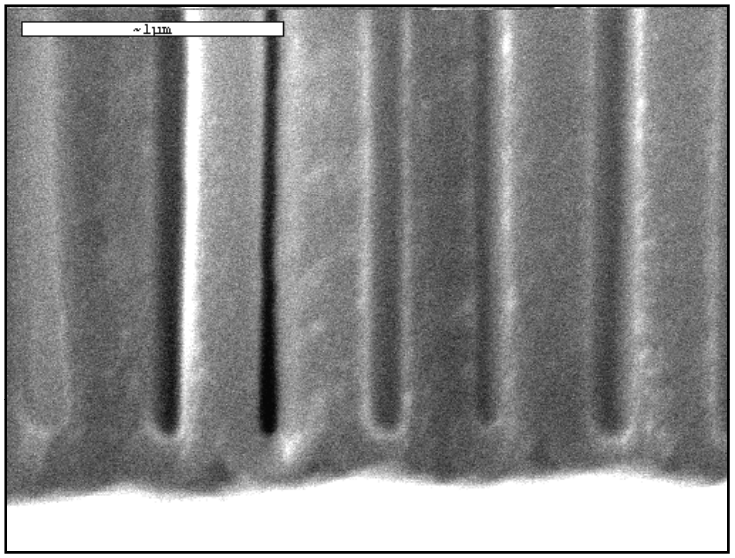
135 min





PORE ORDERING METHODS

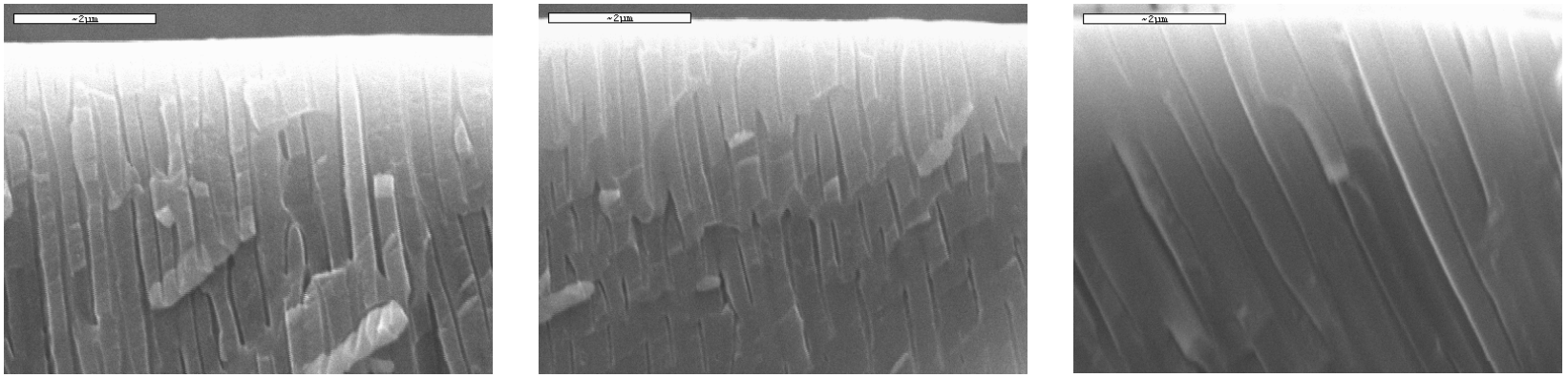




pore diameter ~0.3 µm, pore period ~0.55 µm

- Pre-anodization - well-known Masuda papers; Synkera proprietary procedures
- Nanoimprint lithography - DO NOT HAVE TO PATTERN EVERY PORE
  - conventional nanoimprint methods and tools
  - nanosphere imprint
    (Fournier-Bidoz, Routkevitch, et al, Adv. Mater., 16 (23-24), 2193-2196 (2004)).
- Serial methods (FIB; e-beam, conventional &interference lithography)
  - Expensive, slow and impractical for large area MCPs

BIAS ANGLE or EQUIVALENT



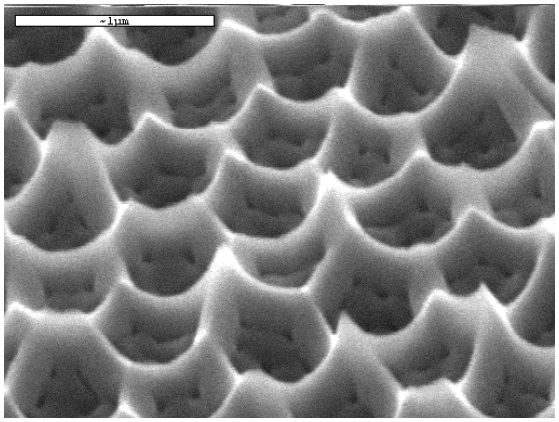
Approach #1: bias the nanopores, then translate into the microchannel bias

- feasibility demonstrated.

Alternative - funnel-shaped pore entrance (intrinsic pores)

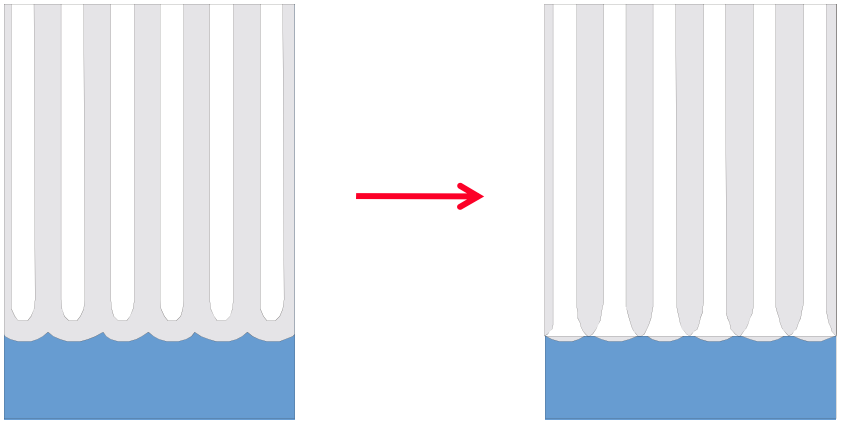
- inherent feature of Synkera free-standing AAO

CHANNEL ENTRANCE SHAPE



Intrinsic funnel-like channel entrance

- Hemi-spherical AAO / Al interface (can be reused to make AAO)
- Localized dissolution of the pore bottom during separation from Al



AAO on Al substrate

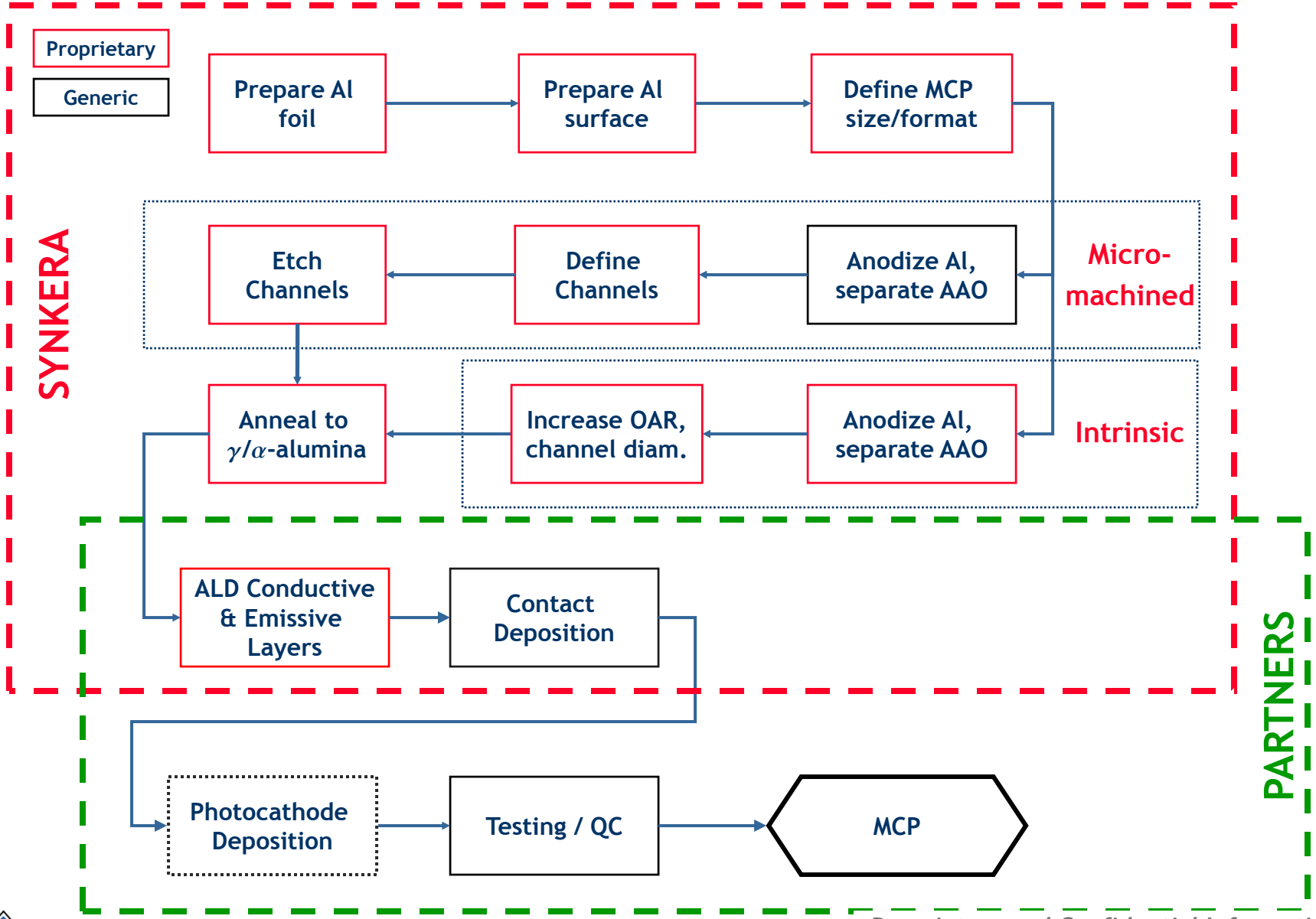
Separating AAO from Al

Free-standing AAO

funnel-shape entrance

Pre-patterned Al (precursor for new AAO)

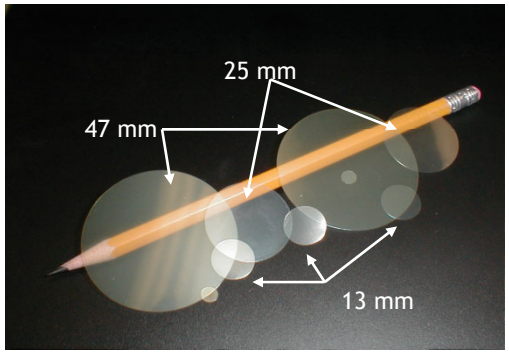
MANUFACTURING OF AAO MCP & s-MCP



AAO SCALE-UP

Free-standing AAO ("membranes")

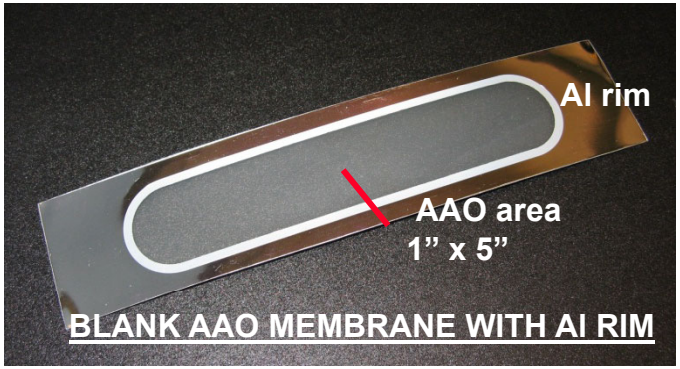
- Commercial production, open sales
- Volume: 100's per week
- Size: from 3 to 150 mm (1/4 to 6")
- Different formats and specifications



Current AAO Membrane Products

AAO membranes supported by Al rim

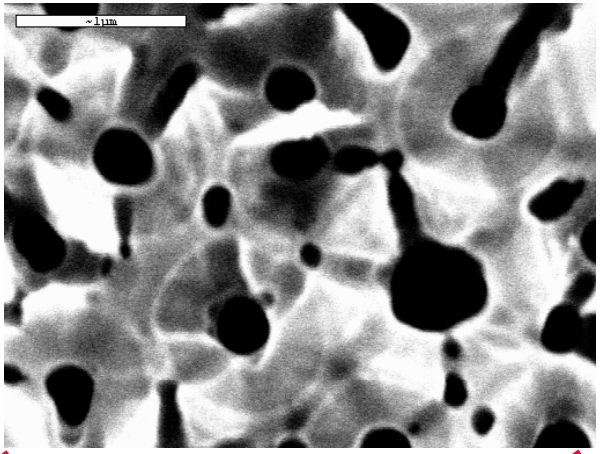
- Facilitates integration into modules
- Membranes up to 11x18" were produced
- Scale-up in progress for several applications, leveraged by related products

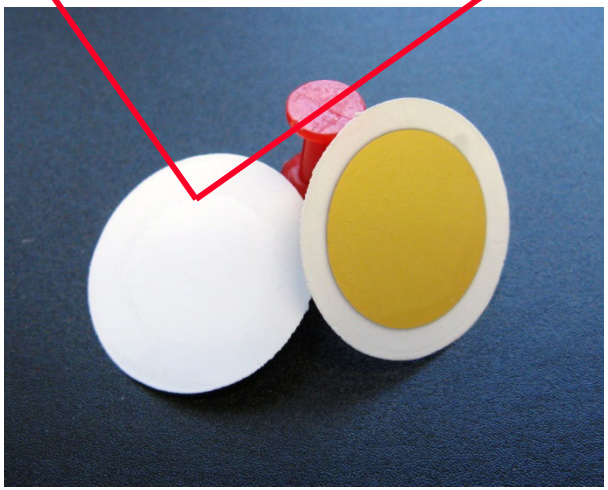


Scale-up emphasis

- achieving target performance in large format
- achieving required durability in a module
- scaling cost- and productivity-limiting steps

RESULTS TO DATE (Synkera & NSF funding)





AAO Substrates / Intrinsic Channels

- Channel geometry
  - Max. diam. 0.3 - 0.6 μm; pitch up to 1.2 μm
  - Open area ratio 30-50%
- Funnel-like entrance on one face
- Irregular channel shape imperfect
- Feasibility of controllable bias (0 to 15°) confirmed
- Size / Format:
  - Current - 25 mm under NSF project
  - Target for LAPD - 32.8 mm
- Withstand temperatures up to 1100°C

ALD

- modification of resistance with Zn-Al oxide
- 200-500 MOhm over 23 mm contact area achieved
- Non-linear I-Vs (resistance levels with voltage)
- Stable current established after a short period.
- Negative TCR (-0.006 to -0.01/°C)
- Resistant to voltage breakdown up to 2500V

Evaluation of electron amplification in progress.

UPDATE ON RELATED WORK

UPDATE ON ALD

SYNKERA ALD FACILITY

- ALD reactor with 6 precursor channels
- Designed to handle substrates with high surface area, high aspect ratio pores (AAO)
- Operating modes: flow for flats, static for AAO
- Batch processing up to 20 of 2” MCP membranes
- Processes validated for a number of materials
- Collaboration with S. George, CU

PRODUCTS UNDER DEVELOPMENT

- Membranes and reactors
- Gas sensors
- Optical Nanocompoistes
- Energy conversion materials
- Microchannel Plates (current NSF funding)

MCP-RELEVANT COATINGS

- Resistive (ZnO, ITO)
- Emissive (Al2O3, MgO)
- Semiconductor (GaN, InN)

UPDATE ON MICROMACHINED CERAMIC MCPs

Targeted Benefits and Differentiation

- Micromachined channels in refractory ceramic
- Improved image quality
- High temperature capability (integration of photocathodes)
- Improved performance (S/N, dynamic range, local and global count rate), stability and lifetime
- Platform for large area MCPs
- Lower cost, scalable to mass-production

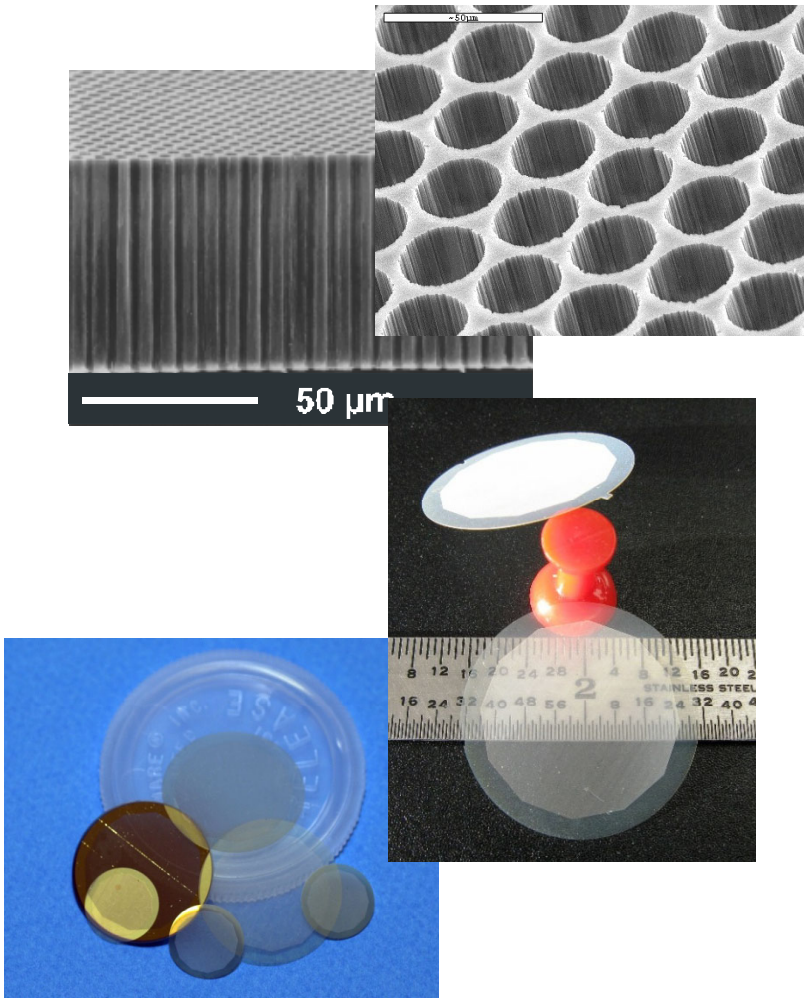
Status

- Channels 3.2 - 10 µm; thickness 50-300 µm;
- Functional prototypes demonstrated
- Excellent breakdown voltage (up to 10V/µm)
- Self-healing on breakdown

Availability

- 25 mm MCP substrates with 10 µm channels available (current work with SSL)
- High resolution (3-5 µm) under development

Synkera Ceramic MCP (micromachined)



SUMMARY

- Contract documents submitted, waiting to be signed
- Synkera is ready to start work
- Targeted intrinsic AAO structure is proven feasible
  - Channel diameter up to 0.4-0.6 μm, period up to 1.2 μm feasible
  - Channel entrance diameter and channel shape need to be improved
- Processes need to be transferred to 33 mm and 8”x8” size
- 25 mm MCP prototypes are being developed under NSF funding
- Other related work (AAO, ALD) will be leveraged into LAPD effort