SYNKERA TECHNOLOGIES, INC.
2021 Miller Drive, Suite B
Longmont, CO 80501
www.synkera.com

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Argonne National Lab
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AAO/ALD AS A PLATFORM FOR LARGE AREA MCP's - PROSPECTS AND LIMITATIONS FROM THE MATERIALS PERSPECTIVE

Dmitri Routkevitch, Ph.D.
Product Manager and Principal Scientist

OUTLINE

INTRODUCTION

- AAO AS A NANO/MICROFABRICATION PLATFORM
- ORIGINS OF THE AAO-BASED MCPs

AAO AS MICROCHANNEL PLATE SUBSTRATE

- MICROMACHINED CHANNELS vs. “NATIVE” NANOPORES
- AAO vs. GLASS vs. SILICON
- SCALABILITY (CHANNEL DIAMETER vs. ASPECT RATIO vs. SIZE)

SYNKERA AAO-MCP DEVELOPMENT

- MICROMACHINED CERAMIC MICROCHANNEL PLATES
- “NATIVE” PORES - SUBMICROCHANNEL PLATES
- ALD FOR CHANNEL MODIFICATION TOOL
- SCALE-UP

HOW CAN SYNKERA PARTICIPATE?

SYNKERA TECHNOLOGIES, INC.

COMPANY OVERVIEW

Core strengths

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

Current Products/Services

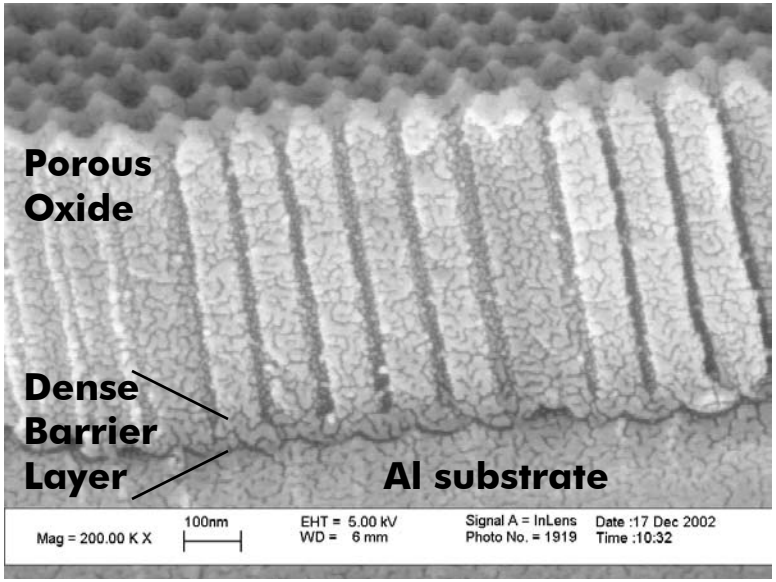
- Gas sensors and modules
- Ceramic membranes
- Contract research

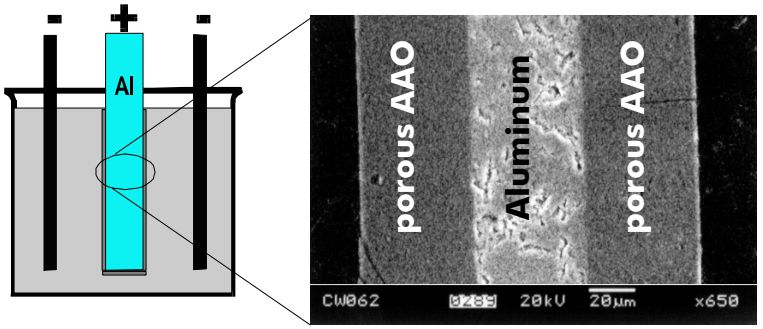
Markets

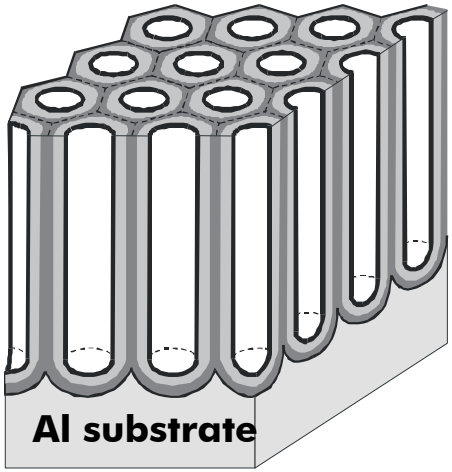
- Industrial health and safety, air quality
- Gas separation, hydrogen generation, R&D
- Emerging markets: clean and alternative energy, bio- and life sciences, filtration

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

ANODIC ALUMINUM OXIDE (AAO) INTRODUCTION



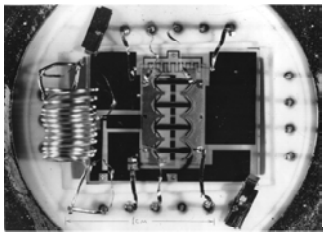


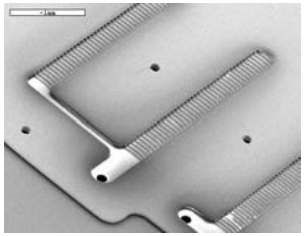


- High quality self-organized material with regular nanoporous lattice
- Uniform & aligned arrays of cylindrical nanopores
- Formed by anodic oxidation of Al in certain electrolytes
- Tunable parameters
  - Pore diameter: 5 - 300 nm
  - Pore density: 10^{12} -10^{8} cm^{-2}
  - Thickness: 0.1 - 300 µm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

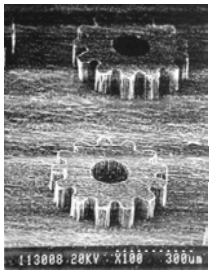
HISTORY OF AAO IN MICRODEVICES

- 1970-80 - pioneering work on microdevices from AAO at the Institute of Electronics, National Acad. Sci., Belarus, Minsk.

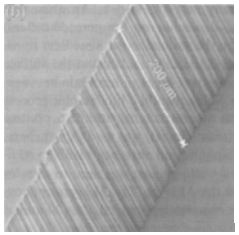


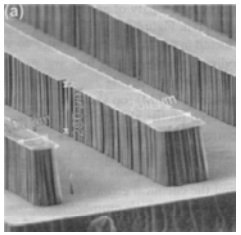


- 1989 - first disclosure, 1994 - first publication after projects were declassified
  - Grigorishin et al., Revue “Le Ville, les Couches Minces”, Grenoble, France, Suppl. N271, 304 (1994)
  - Grigorishin, Mukhurov, et al., Proc. 7th Conf. of ITG Committee “Vacuum Electronics and Displays", VDE, Verlag, 132, 155 (1995)
  - Govyadinov, Mardilovich et al., *ibid.*,132, 161 (1995)



- 1995 - first publications by others:
  - Reed et al., Proc. of IEEE MEMS’95, 267 (1995)
  - Gösele et al., Adv. Mater. 11(6), 483 (1999)





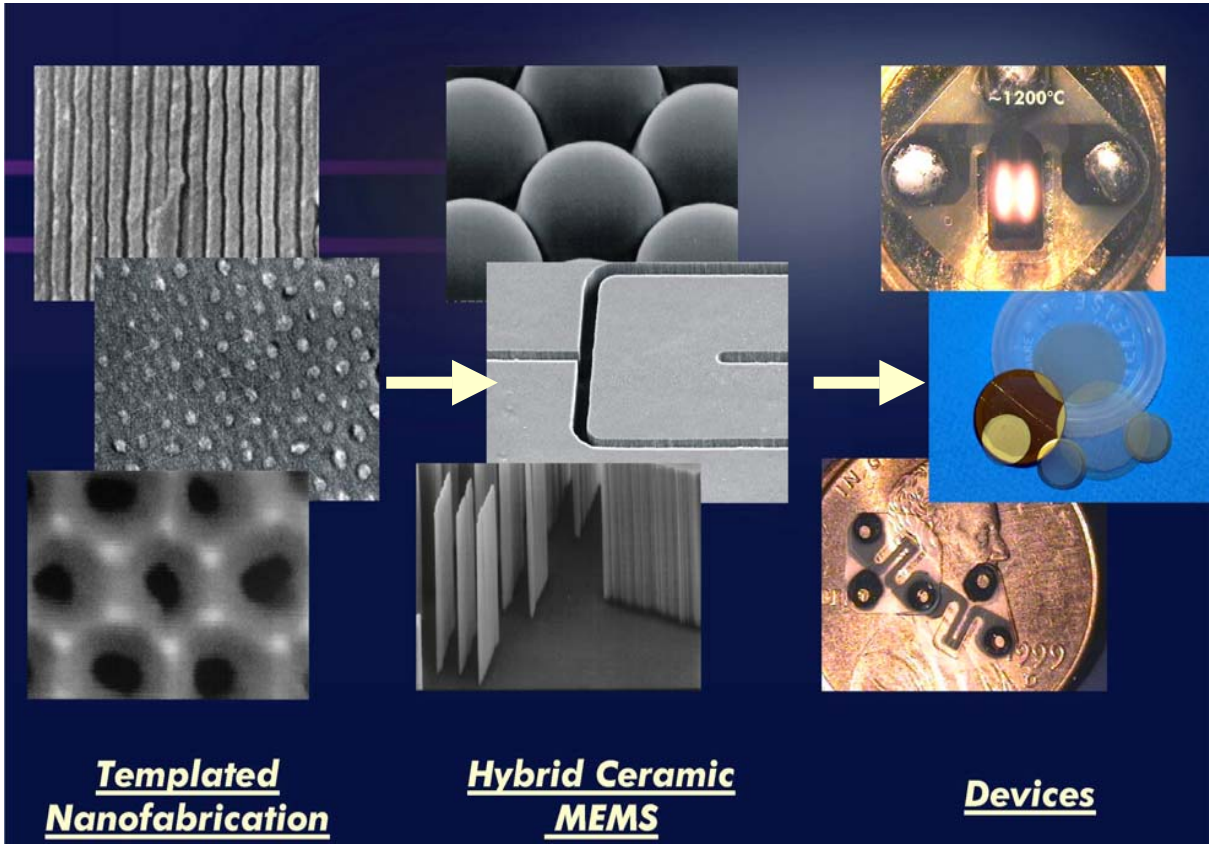
- **1997, 98 - ceramic MCP and s-MCPs**
  - Govyadinov, Emeliantchik, Kurilin, Anodic Alumina Microchannel Plates, Nucl. Instr. Methods Phys. Res., A 419 , 667-675 (1998)
  - Raspereza, Govyadinov, Kurilin, Kukhnovetc, Emel'yanchik, et al., Submicrochannel plate multipliers, Appl. Surf. Sci. 111, 295-301 (1997)

NANO- and MICROFABRICATION PLATFORM

Synkera's product development is supported by a unique technology platform based on nanoporous anodic alumina

Core capabilities enabled by anodic alumina:

- Nanoscale engineering of materials architecture and performance
- Ceramic micromachining
- Integration of materials into functional devices
- Scaleable processes amenable to high volume manufacturing

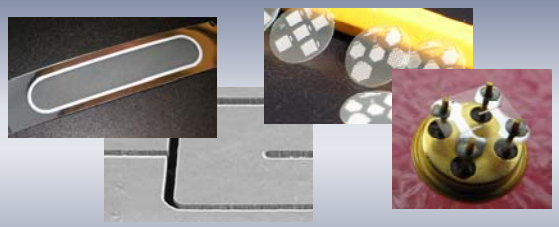


PRODUCT GROUPS

Gas Processing
- Membranes, reactors & adsorbents
- Fuel cell systems (H_2)
- Oil and gas (H_2, He)
- Petrochemical (H_2)
- Distributed (H_2)

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

PRODUCT PLATFORM



- Nano / microscale engineering
- Scalable manufacturing

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

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

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

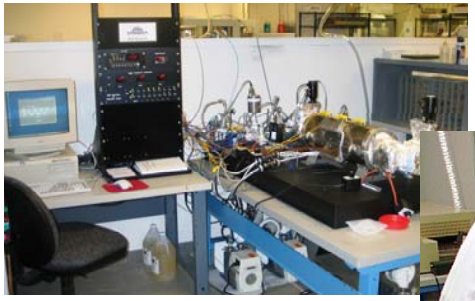
SYNKERA FACILITIES

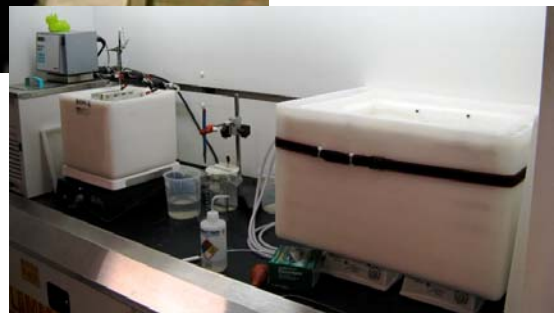
Over 5,000 sq. ft. RD&E and production facility:

- materials processing
- wet laboratory
- analytical equipment
- pilot-scale manufacturing
- testing & QC

Capacity

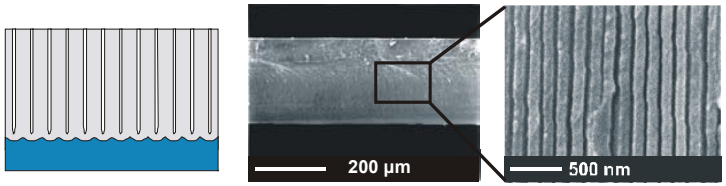
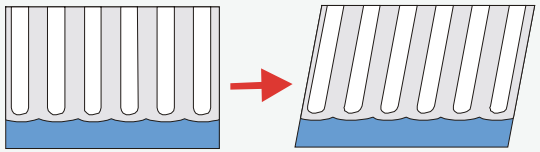
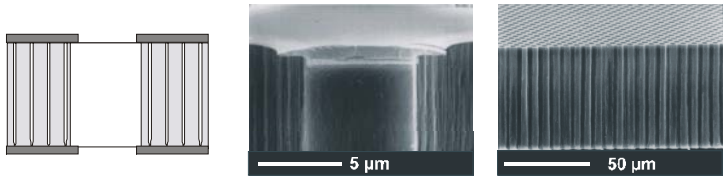
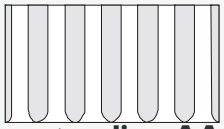
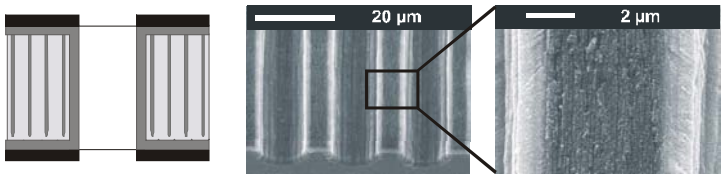
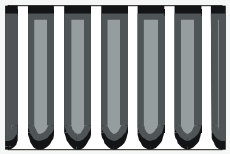
- thousands to tens of thousands of components per year
- plans in place for increasing to 100's of thousands of devices per year





AAO AS MICROCHANNEL PLATE SUBSTRATE

TWO-TIER APPROACH

	Micromachined Ceramic MCPs	Sub-Microchannel Plates
1. Synthesis of nanoporous anodic alumina	 pore diameter 20-100 nm	 pore diameter 0.2 - 0.8 µm
2. Separation, patterning and micromachining		 free-standing AAO, funnel-shape entrance channels
3. Modification, deposition of electrodes		
	"Conventional" micromachining of AAO, minimal channel diameter ~2 µm	*Large "Intrinsic"* pores, channel diameter 0.2 - 0.8 µm

LARGE AREA MCP - MATERIALS PERSPECTIVE

	GLASS	SILICON	AAO micromachined	AAO native pores
Status / Availability	mature & improving products	on hold	prototype development	early feasibility
Projected Price (for large scale)	$$$	$$	$$	$
Max Size	1 - 20 cm	up to 30 cm	up to 30 cm	up to few m
Channel Size, µm	2 - 50	2 - 50	3 - 50	too small / 0.01 - 0.2 / generic better / up to 0.8 / Synkera
L / D	satisfactiry	satisfactiry	low / generic up to 80 / Synkera	too high / generic better / Synkera
Channel Uniformity	artifacts / improving	excellent	potentially excellent (TBC)	
Channel Bias	satisfactiry	satisfactiry	n/a (generic) 0 - 15° (Synkera)	
Max Temperature	250	500	1100	
Mechanical Durability	low	poor	robust	very robust
Environmental Sensitivity	poor (moisture)	good	excellent	
TCR	negative	negative	positive	
Lifetime at high rate	months	months?	potentially years (TBC)	
Count Rate	low ?	med	potentially high (TBC)	
Dynamic Range, S/N	good	good	potentially high (TBC)	
Manufacturability of large ps MCPs	poor	?	medium	excellent

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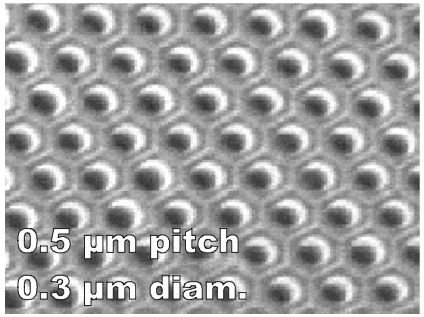
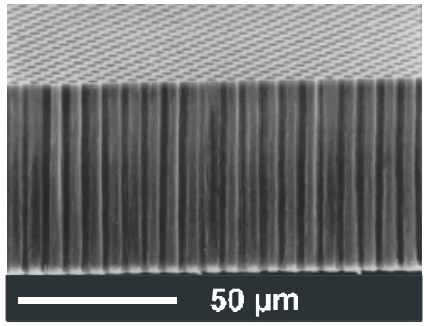
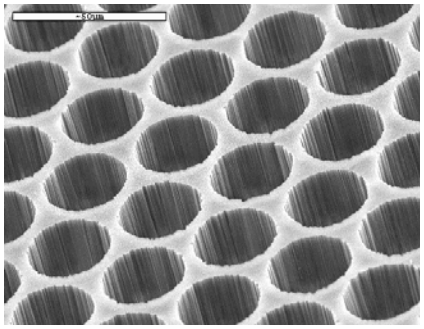
CHANNEL DIAMETER







Synkera AAO, intrinsic pores — Micromachined AAO

Generic AAO, intrinsic pores — Glass MCPs

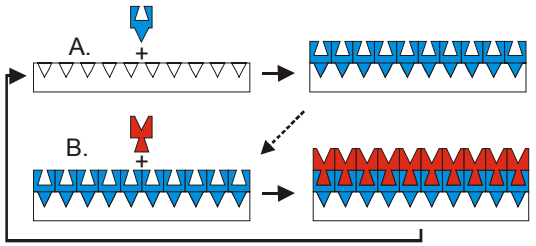
0.01 0.1 0.5 1 5 10 µm

Challenges	Generic AAO	Synkera AAO
• diameter - native pores	< 0.2 µm	< 0.8 µm
- microfab. channels	> 3 µm	> 2 µm
• thickness	0.2 mm	up to 1.5 mm
• channel bias	0	0 - 15° feasible

- L/D, OAR, channel roughness, realizing functional prototypes ...

ALD for MCP CHANNEL MODIFICATION

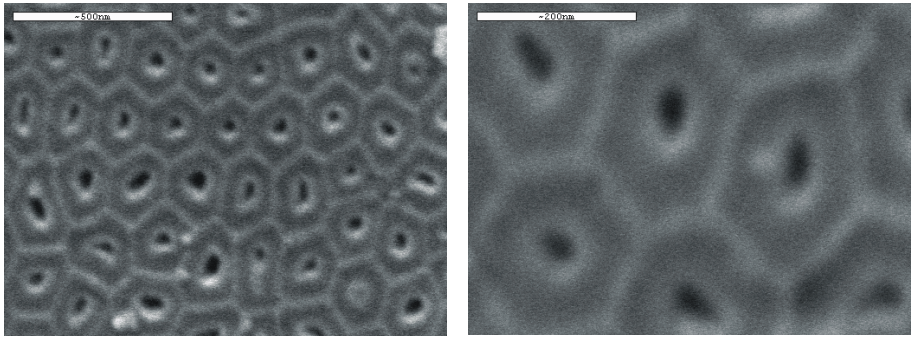
ALD CHANNEL FUNCTIONALIZATION



A. $ZnOH^* + Zn(CH_2CH_3)_2 \rightarrow ZnOZn\ CH_3CH_3^* + CH_3CH_3$

B. $\underline{ZnCH_2CH_3^* + H_2O \rightarrow ZnOH^* + CH_3CH_3}$

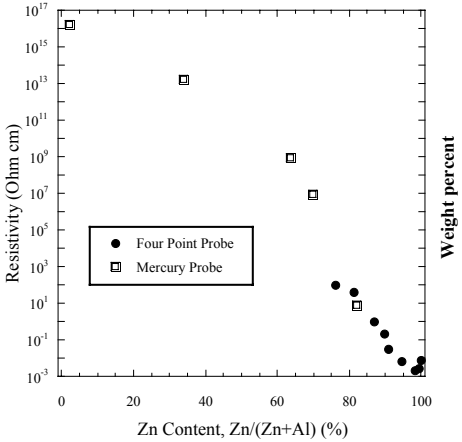
$Zn(CH_2CH_3)_2 + 2H_2O \rightarrow Zn(OH)_2 + CH_3CH_3$

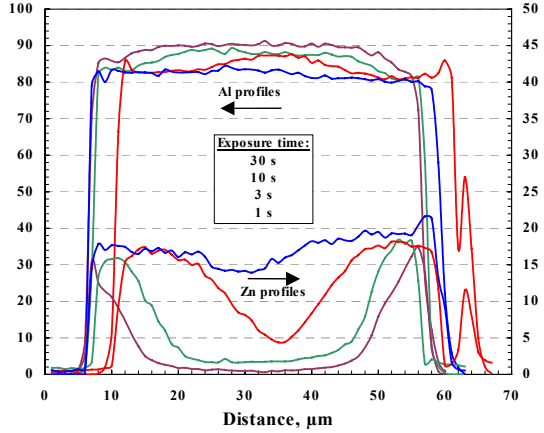


near the surface 25 µm deep

65 nm pores, ~10 nm ALD

- ALD: self-limiting binary reactions
- Thickness = f(number of cycles)
- Precise control of layer composition and properties - critical for MCPs
  - resistance layer
  - electron emission layer
  - channel diameter, roughness
  - gradients of composition and properties to make complex layers
- Mixed Al_2O_3/ZnO provide 14 orders of magnitude resistance variations





J. Electrochem. Soc. 2002, 150, G339-G347.
Chem. Materials, 15, 3507-3517 (2003).

ALD FACILITY AND CAPABILITIES

SYNKERA ALD FACILITY

- Fully automated ALD reactor; 6 precursor channels
- Operating modes: low for flats, static for AAO
- Batch processing up to 20 of 2” MCP membranes
- Processes validated for a number of materials (oxides, nitrides, metals)
- Collaboration with S. George, CU

CHALLENGES for AAO

- High surface area
- High aspect ratio
- Large doses required, thus high rate of precursor consumption and maintenance

MCP-RELEVANT COATINGS

- Resistive coatings developed
- Emissive coatings developed (Al_2O_3, MgO)
- Semiconductor coatings developed (GaN, InN)

MCP PROTOTYPE FABRICATION

- Prototypes with micromachined channels fabricated
- Prototypes with intrinsic pores in development

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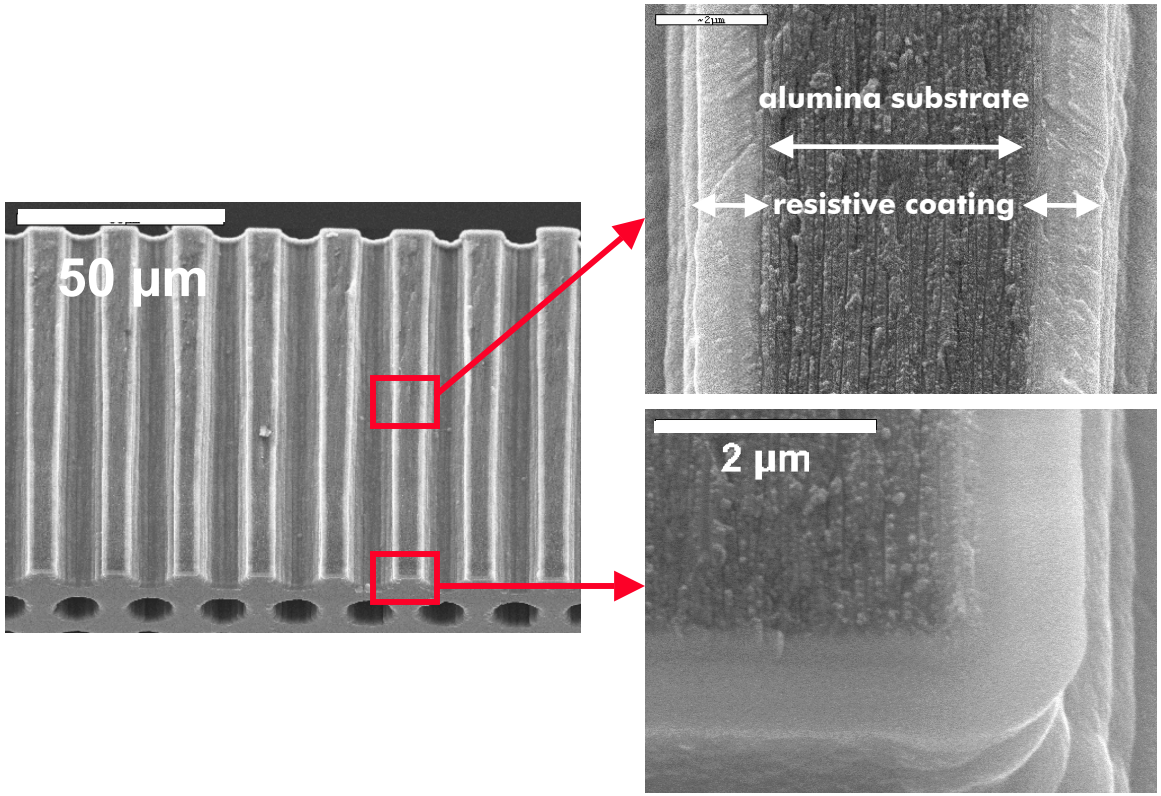
ALD COATING ON MICROCHANNEL PLATE

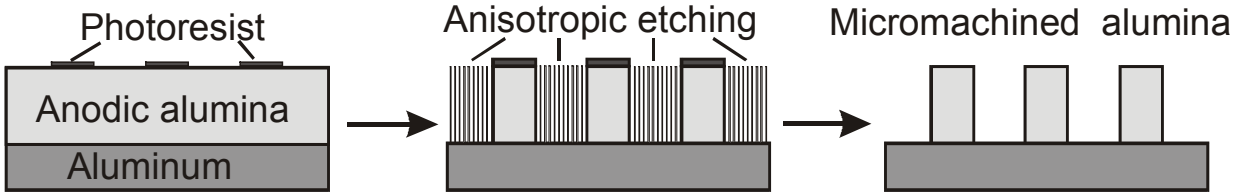


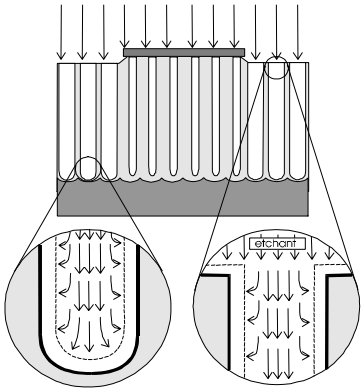
AAO-based MCP with ALD coating inside micromachined channels.

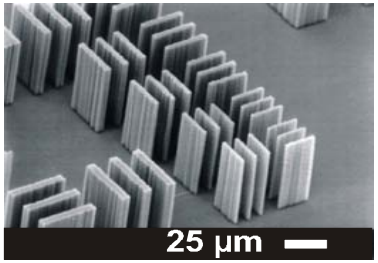
- Note uniform & conformal layer with excellent adhesion
- ALD coating survived annealing to 900°C without failure
- Leveling surface roughness
- Diameter reduction is possible by depositing thick layers

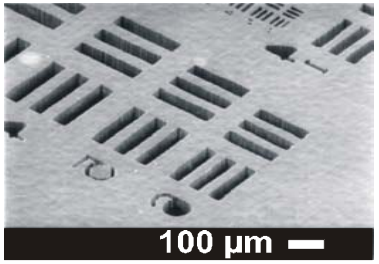
MICROMACHINED CHANNELS

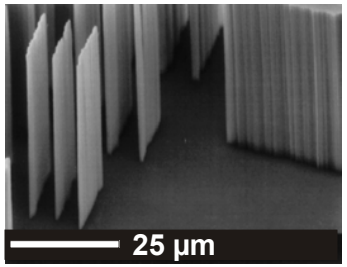
MICROMACHINING OF ANODIC ALUMINA

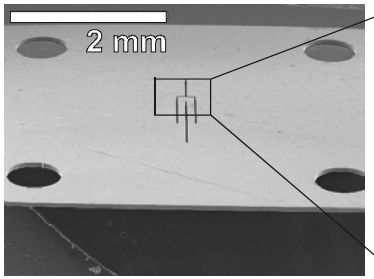


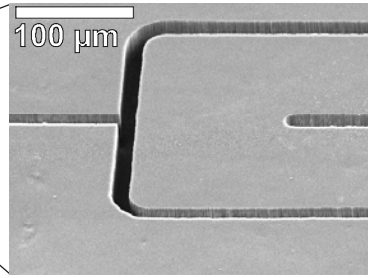


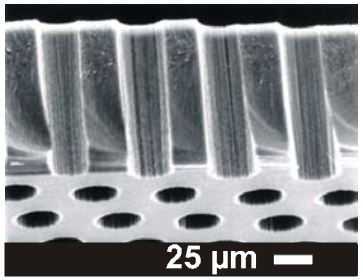






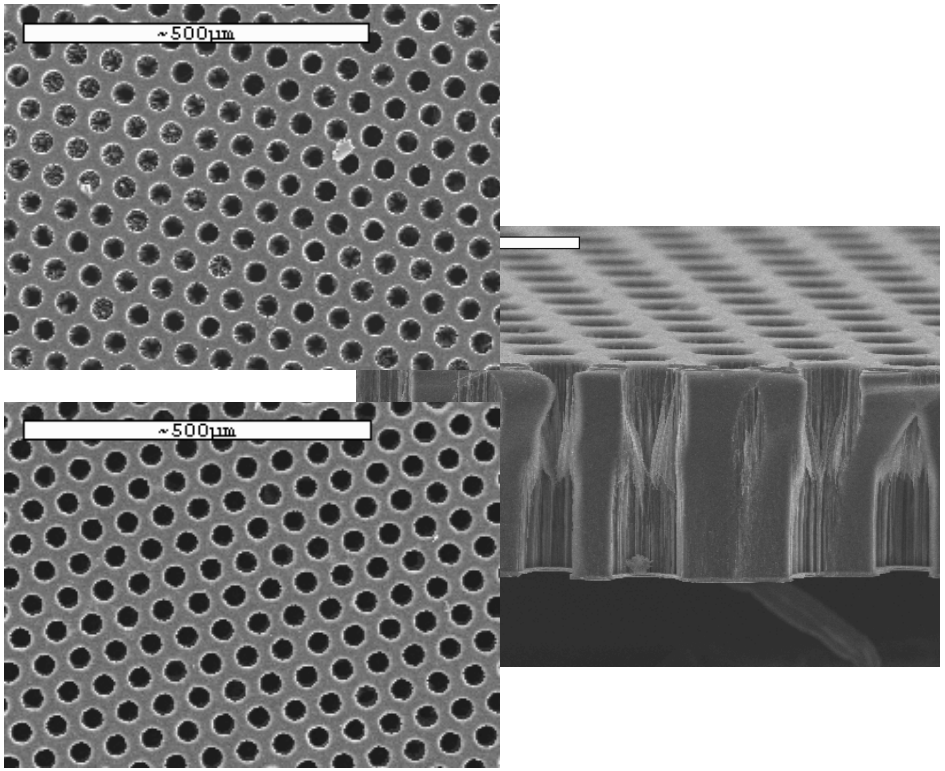


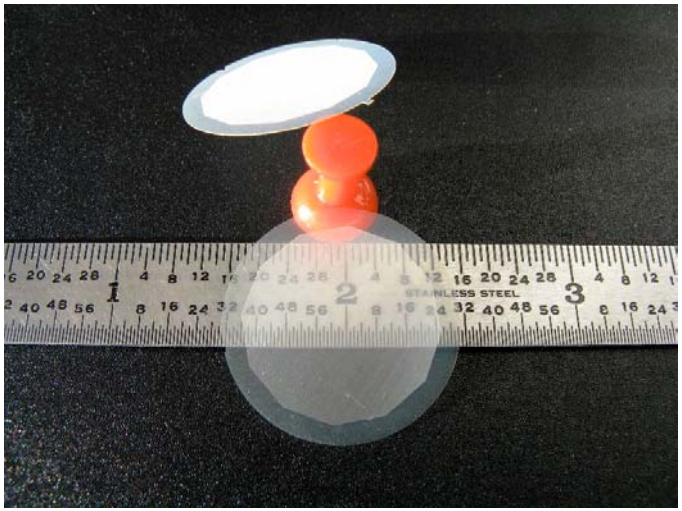




- High resolution, high aspect ratio ceramic micromachining
- Inexpensive, large-scale substrates, conventional exposure sources
- An enabling technology for a number of ceramic MEMS products

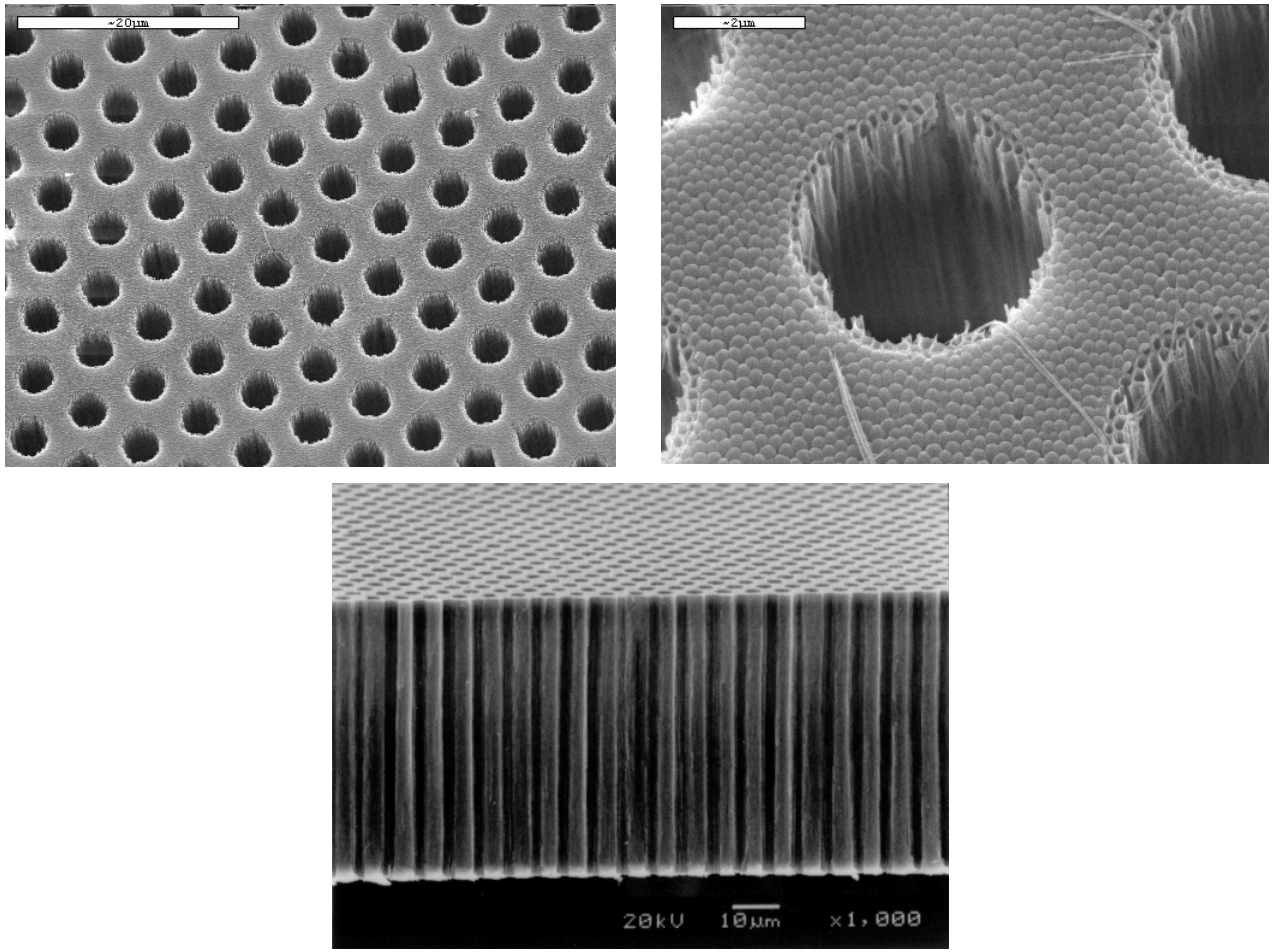
LOW RESOLUTION MCP SUBSTRATES





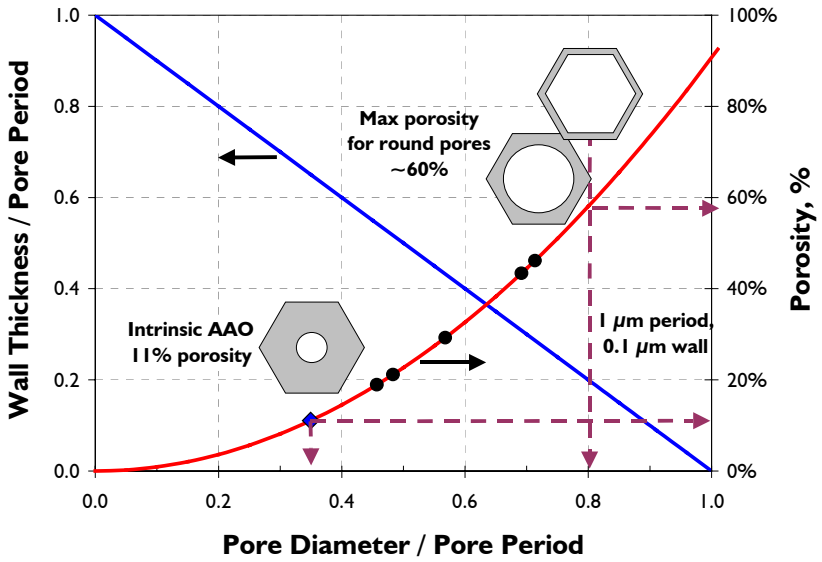
- Channels >10 µm; thickness 50-300 µm; can be annealed to gamma/theta-alumina
- Prototype MCPs with low resolution channels produced and tested,
- Blank 25 mm substrates available (current work at SSL on III-V photocathodes)

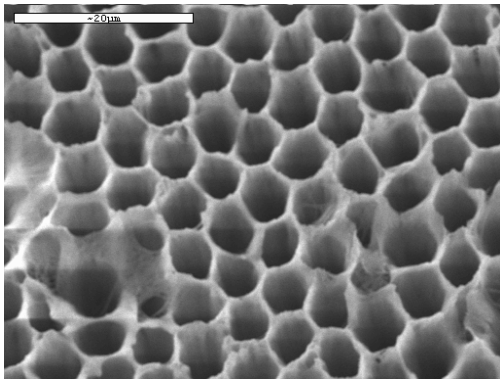
HIGH RESOLUTION CHANNELS

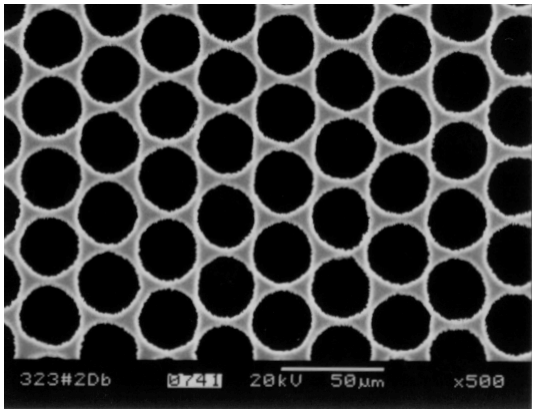


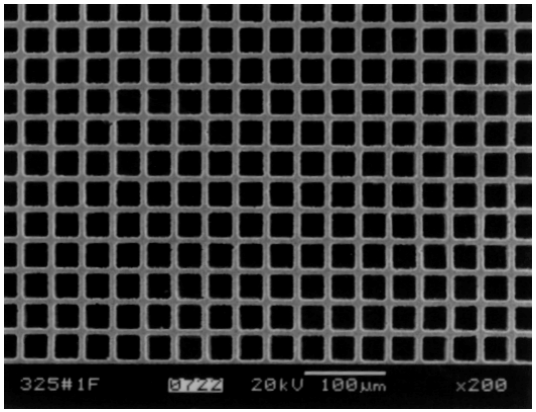
- Ceramic MCP substrate with ~3 µm channels, 60-100 µm thick
- Under development

MAXIMIZING OPEN AREA



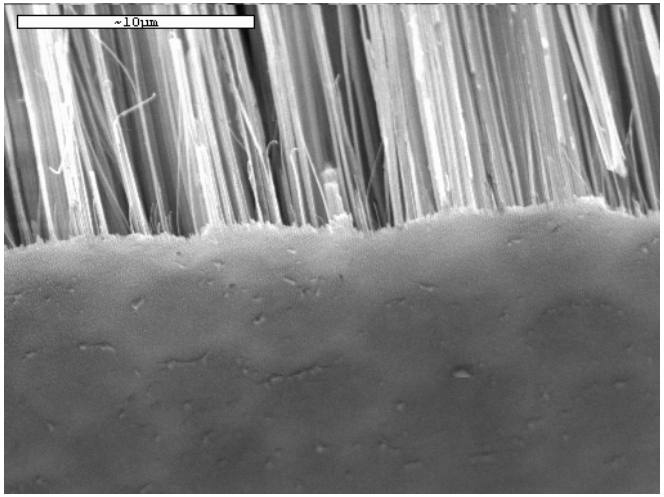


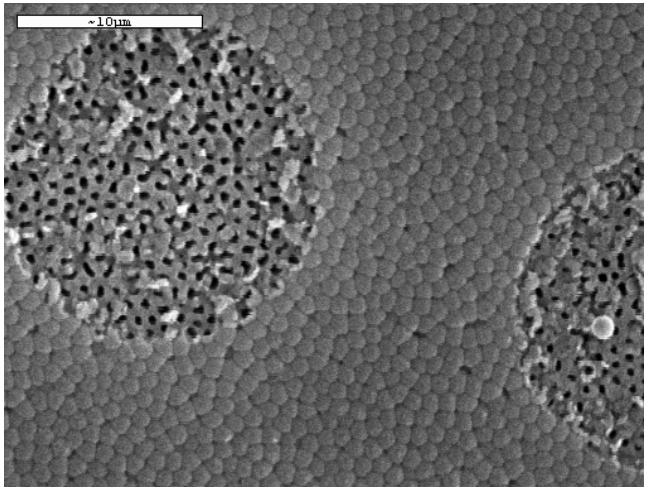




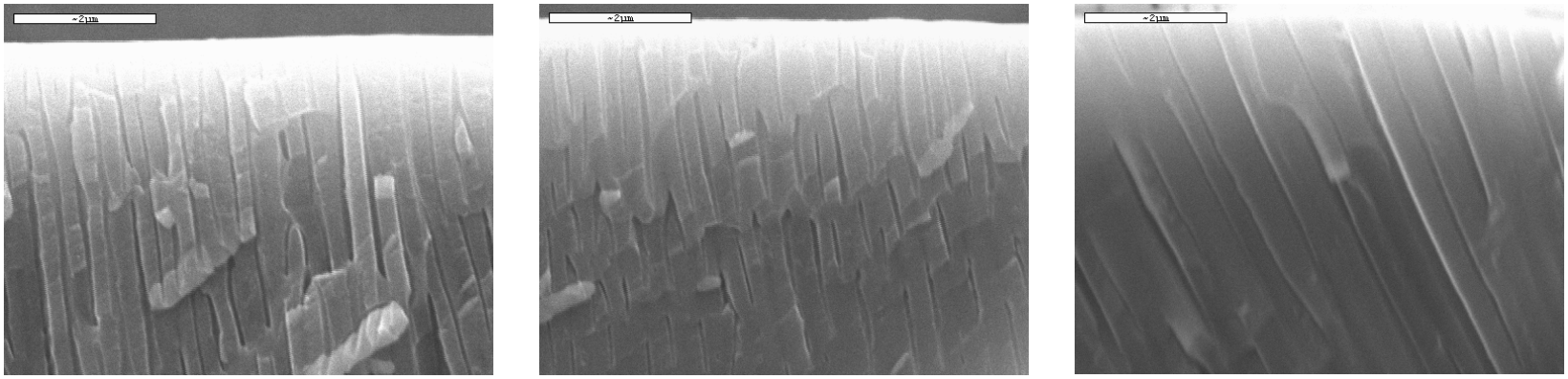
- 25 μm channel diam, 100 μm thickness
- 60% for round channels in *hcp* lattice
- up to 80% in square channels

INTRINSIC ION FEEDBACK BARRIER?





BIAS ANGLE or EQUIVALENT



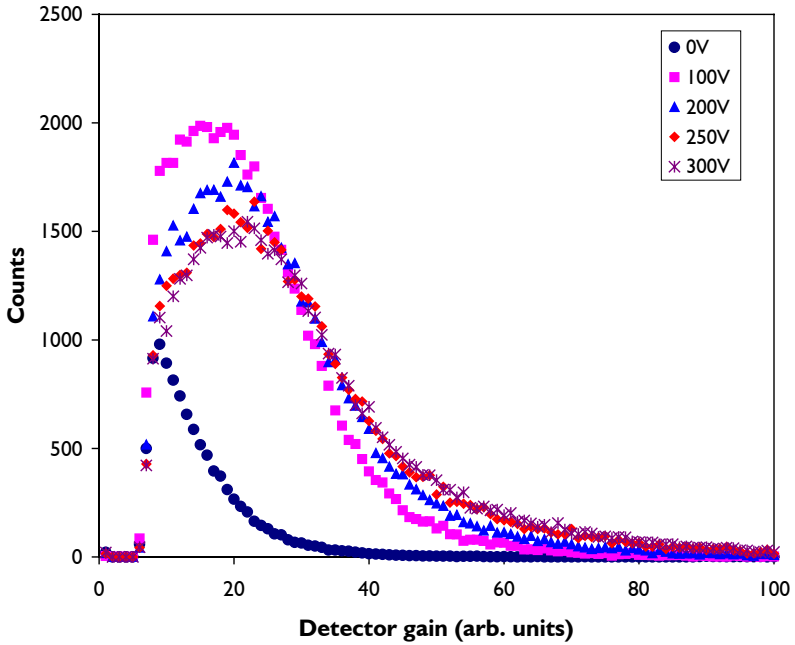
Approach #1: bias the nanopores, then translate into the microchannel bias

- feasibility demonstrated.

Approach #1: funnel-shaped pore entrance (intrinsic pores)

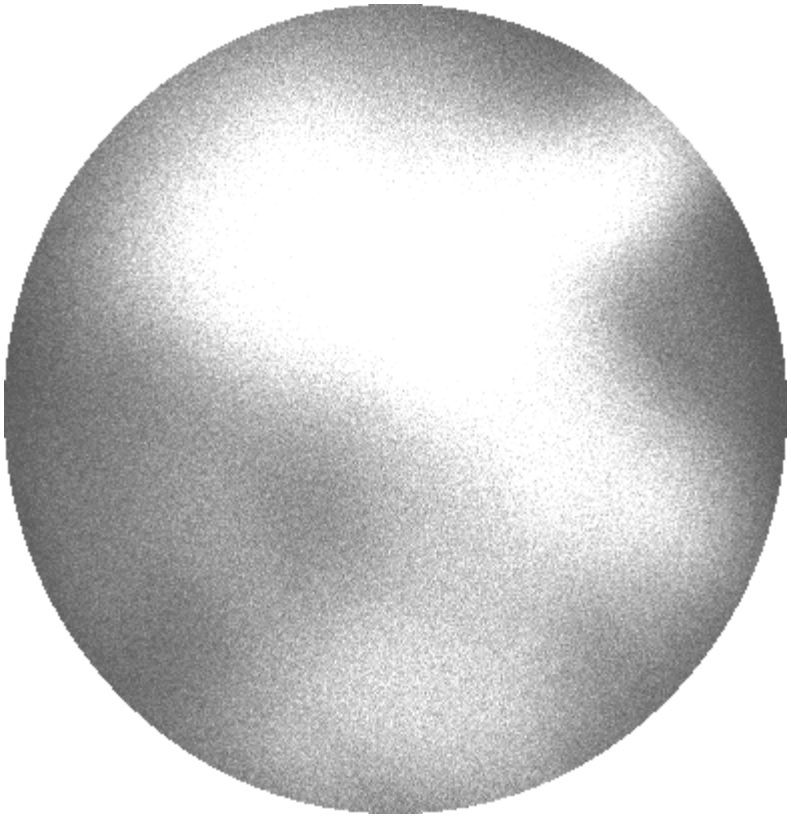
- inherent feature of Synkera free-standing AAO

TESTING AT UC BERKELEY

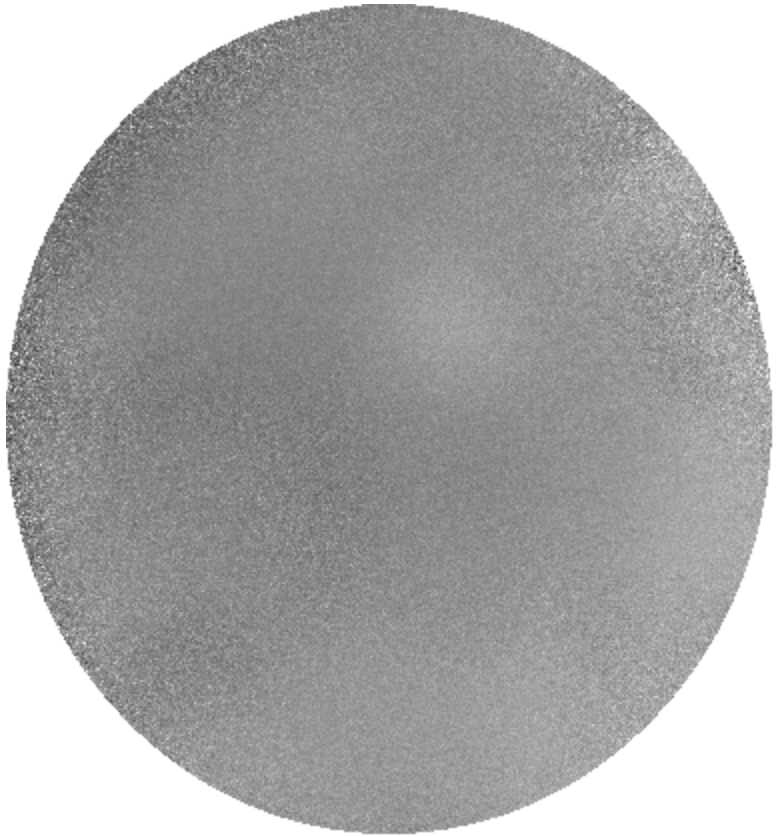


- Channel diameter 10 μm; D/L = 10
- Tested at UC Berkeley, Space Science lab
- Ceramic MCP (bias varied) was in front of two glass MCP (1500V bias).
- Testing demonstrated appreciable gain and sensitivity to UV photons
- Breakdown voltage up to 1000V
- "Self-healing" of the breakdown defects during ramp-up

INITIAL TESTING AT UC BERKELEY



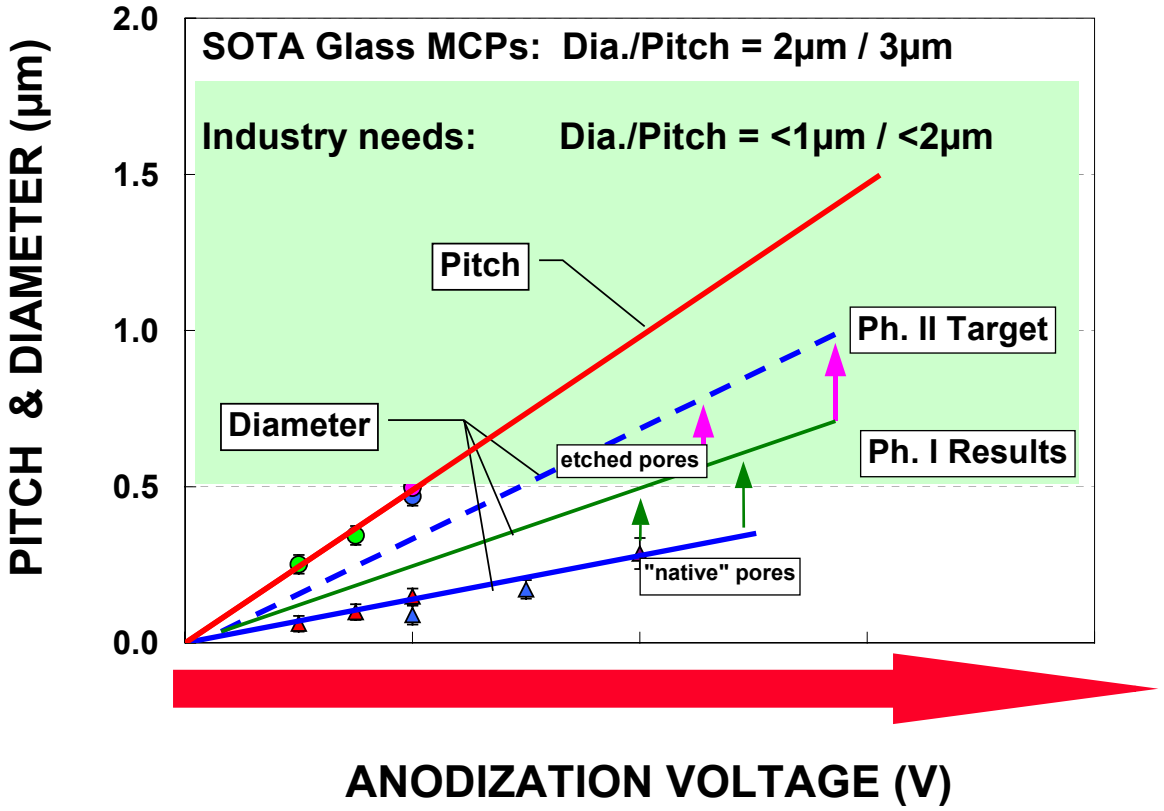
- Full flood image at 2537Å
- Alumina + 2 glass MCP stack

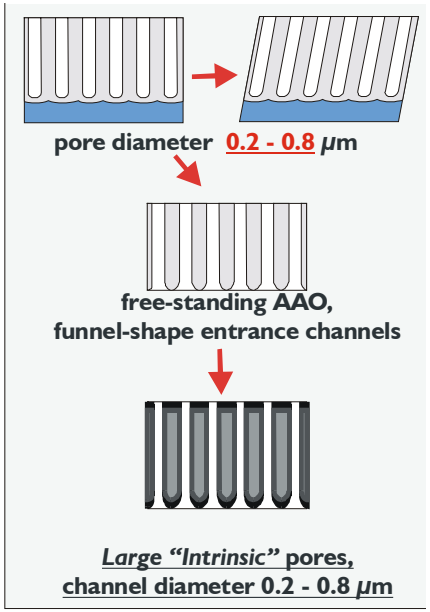


- Full flood gain map at 2537Å
- Alumina + 2 glass MCP stack

INTRINSIC CHANNELS

ACHIEVING TARGET CHANNEL DIAMETER / PITCH





Pore Pitch:

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

Pore Diameter:

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

Challenge: increasing anodization voltage

- Factors to consider
  - Upper voltage limit
  - Anodization rate

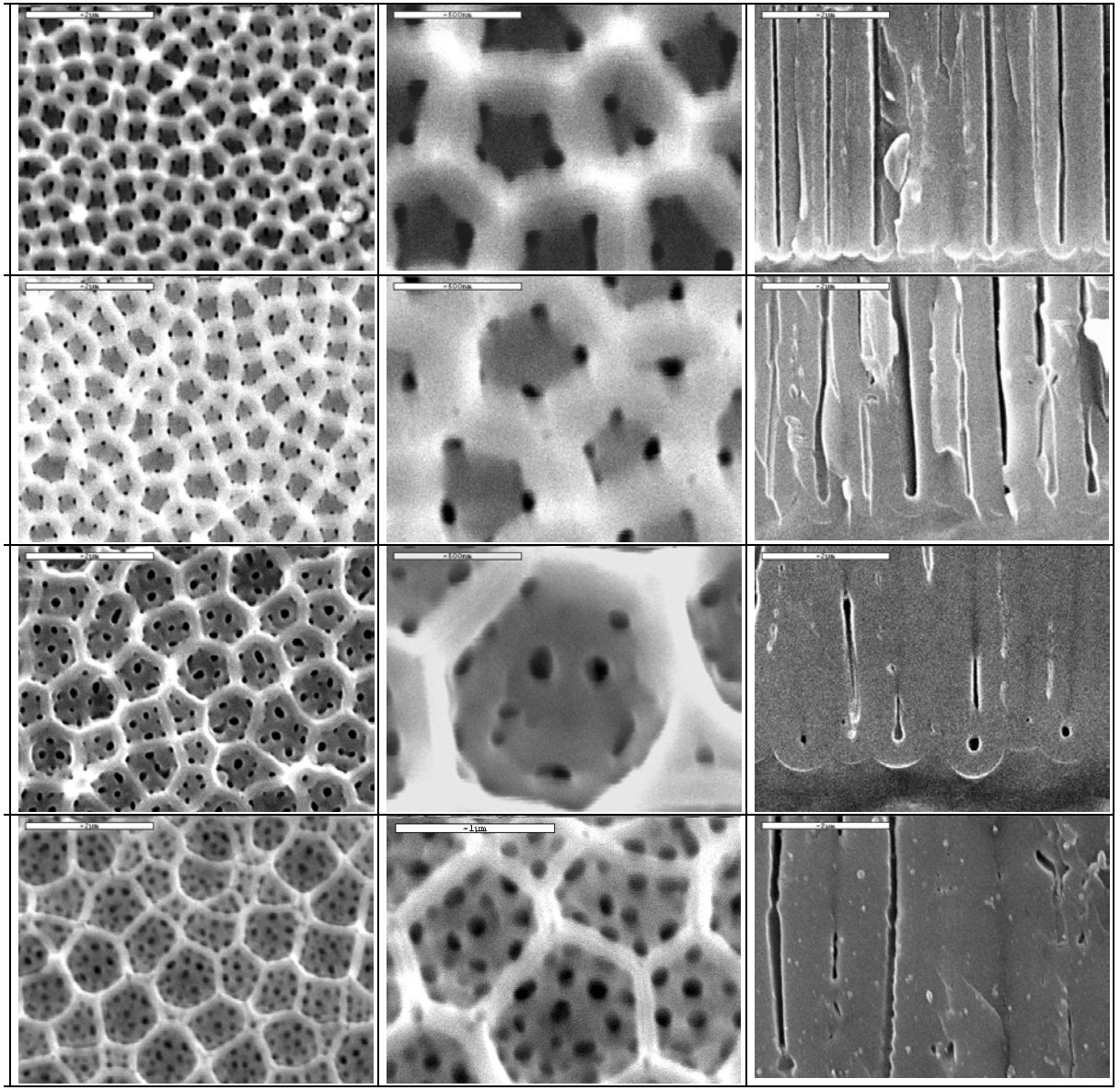
Approach: new electrolytes & procedures

U

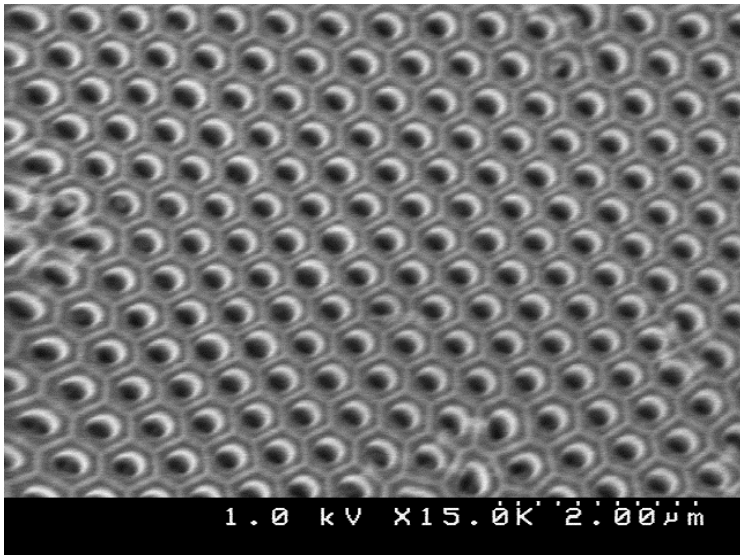
1.5xU

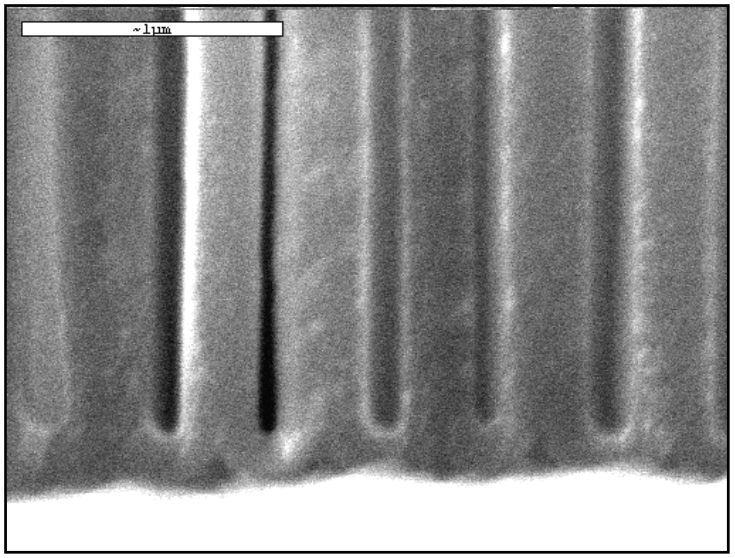
2xU

2.5xU



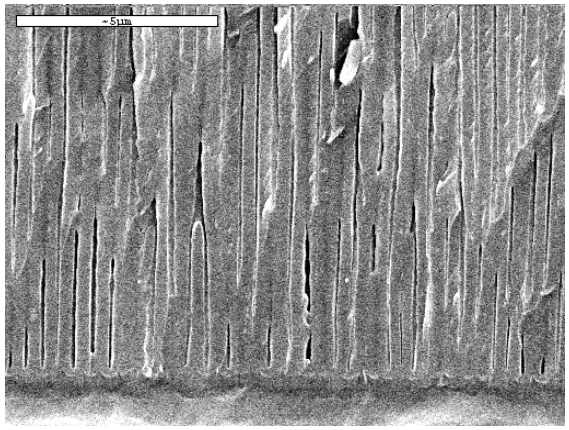
PORE ORDERING



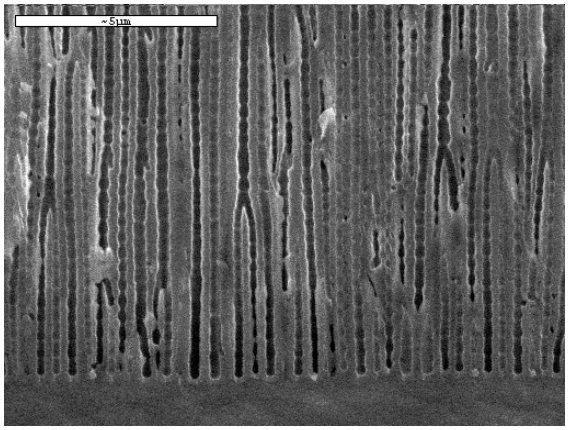


- Al was pre-anodized and anodized at 195 V, followed by pore widening
- Resulting pore diameter ~0.3 µm, pore period ~0.55 µm
- Number of methods available to improve pore ordering:
  - Pre-anodization - well-known (Masuda papers, Synkera proprietary procedures)
  - Nanoimprint lithography - DO NOT HAVE TO PATTERN EVERY PORE
    - *conventional nanoimprint methods and tools*
    - *nanosphere imprint (Fournier-Bidoz, Kitaev, Routkevitch, et al, Highly Ordered Nanosphere Imprinted Nanoporous Anodic Alumina, Adv. Mater., 16 (23-24), 2193-2196 (2004)).*
  - Other methods (FIB; e-beam, conventional &interference lithography)

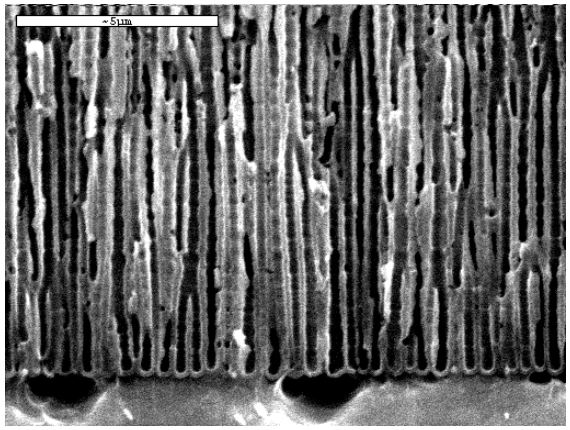
CONFORMAL CHANNEL ETCHING



As anodized



90 min



135 min

RESULTS TO DATE

Early MCP prototypes with intrinsic or “native” AAO pores:

- Channel geometry: diam. 0.2 - 0.6 μm, pitch up to 1.2 μm
- Open area ratio up to 80% with funnel-like channel entrance on one face
- Feasibility of controllable bias (0 to 15°) confirmed.
- Size / Format: 25 mm produced; larger sizes (up to 150 mm) supported.
- Channel Modification: precision ALD modification of the resistance; secondary emission enhancement

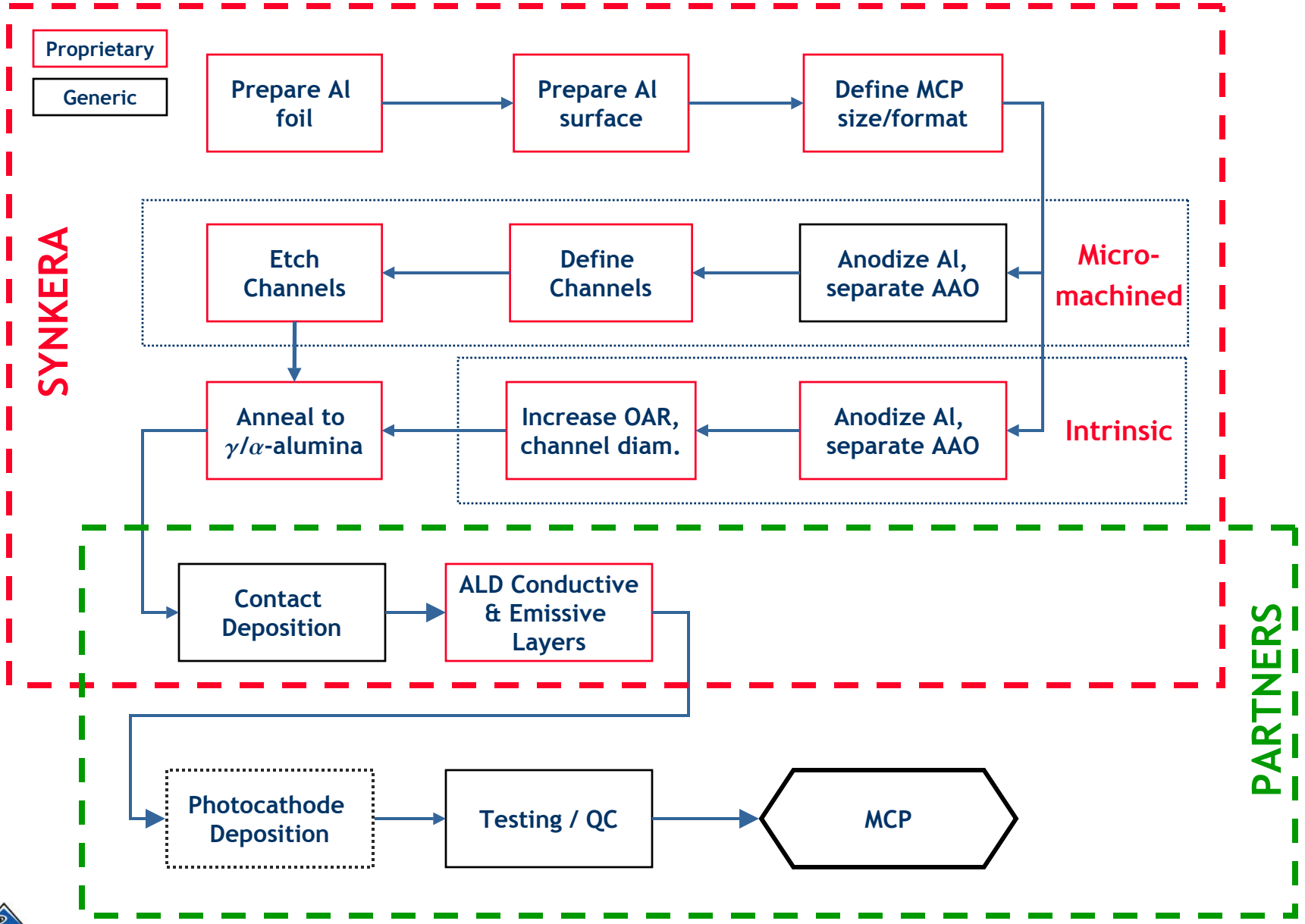
Performance to date

- Excellent voltage breakdown resistance (up to 1000V).
- Resistance leveling off with voltage, as compared to a steady decline for Si or glass MCPs.
- Great resistance stability after a short stabilization period.
- Positive TCR (0.006 to 0.01/°C), eliminating "thermal runaway"
  - could enable high bias and high count rate
- Withstand temperatures up to 1100°C without loss of integrity

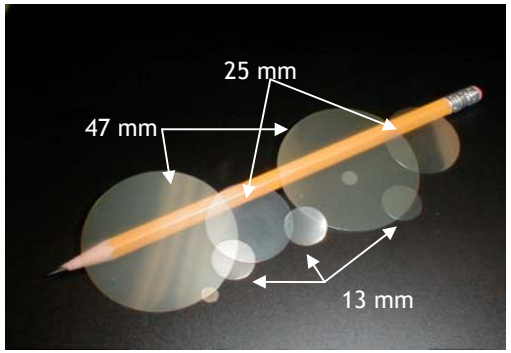
Evaluation of the electron amplification is in progress.

SCALE-UP AND PROTOTYPING

MANUFACTURING OF AAO MCP & s-MCP



AAO SCALE-UP



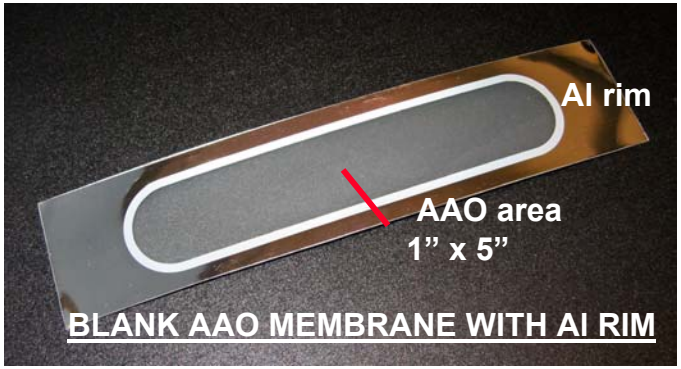
Current AAO Membrane Products

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

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

STATUS of AAO-BASED MCPs

PARAMETER	CURRENT AAO MCP STATUS AT SYNKERA		RISK
	Micromachined	Native pores	
Max Size	25 mm current prototypes	25 mm prototypes, up to 11" x 18" feasible	**scaling new AAO processes**
Development status	prototype development	early feasibility	**producing functional prototypes**
Channel Size, µm	2-3 (for 20:1) needs dev. for high L/D	up to 0.8 µm, (target 1 - 1.5 µ)	**achieving high resolution in required size & AR**
L / D	up to 40 (for >10 µm)	10-200	**achieving needed L/D with required resolution in practical format**
Channel Bias	needs development	feasibility demonstrated	**validating in practical scale, is it needed?**
Resistance	basic process developed		**fine-tuning the physics; scaling to size**
TCR	0.006 to 0.01/°C		**reduced thermal runoff**
Temperature Range	up to 1100°C confirmed		**-**
Mechanical Durability	robust	very robust	**-**
Lifetime / Stability	unknown	unknown	**validating assumptions**
Competitive Posision	drop-in replacement	undefined (large area?)	**lack of a thorough performance evaluation, validating assumptions**

DIFFERENTIATION & BENEFITS

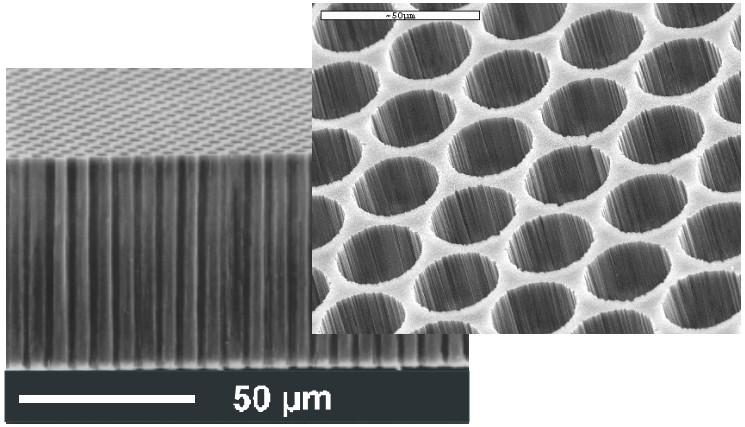
Differentiation

- Micromachined or intrinsic nanochannels in refractory ceramic
- Simplified fabrication, lower cost
- Significant performance and reliability advantages over conventional glass MCPs

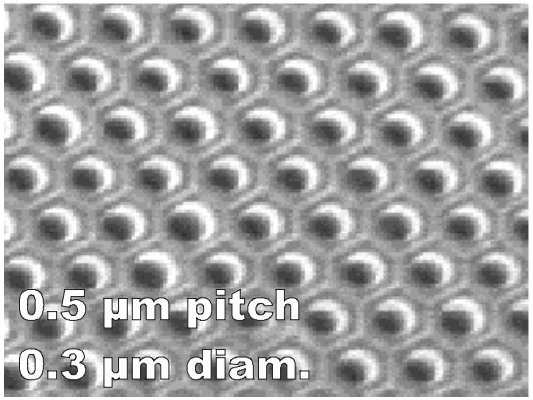
Projected Benefits

- Increased spatial and temporal resolution
- Improved image quality, (0.5 - 3 µm channels)
- High temperature integration of photocathodes
- Improved performance (signal-to-noise, dynamic range, local and global count rate)
- Excellent breakdown voltage (up to 10V/µm)
- Positive TCR eliminating "thermal runaway"
- Better stability and longer lifetime
- Platform for large area MCPs
- Lower cost, scaleable to mass-production

Synkera Ceramic MCP (micromachined)



Synkera Ceramic s-MCPs (intrinsic)



HOW CAN SYNKERA PARTICIPATE

CORE STRENGTH

- LEADER IN USING AAO AS MATERIALS AND TECHNOLOGY PLATFORM
  - LARGE NUMBER OF TECHNOLOGIES AND PROCESSES
  - SEVERAL PRODUICT LINES IN THE PIPELINE
- EXTENSIVE AAO SYNTHESIS, PROCESSING AND SCALE-UP CAPABILITIES
- HIGH THROUGHPUT ALD IN HIGH SURFACE AREA, LARGE SIZE SUBSTRATES
- FOCUS ON PRODUCT DEVELOPMENT AND END USER NEEDS

SPECIFIC CONTRIBUTION

- DEVELOPMENT OF “MICROMASHINED” AND “NATIVE” AAO SUBSTRATES
- ALD OF RESISTIVE AND EMISSIVE COATINGS
- FABRICATING AND SCALING-UP MCP PROTOTYPES
- FINANCIAL LEVERAGE THROUGH EXISTING NSF FUNDING

FUNDING / PARTNERS

NSF Phase II SBIR Project "New Ceramic Sub-Microchannel Plates"

- Grant #IIP-0724478
- $500K, 09/01/07 - 08/31/09
- Phase IIB opportunity: additional 1:2 match for non-SBIR funding

NIH Phase I SBIR Project, "Ceramic Microchannel Plates for Biomedical Imaging"

- Grant #1R43EB003700
- $200K, project ended
- Phase II opportunity ($750K - $1.5M)

Partners

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

CONTACTS

Product Development

- Dmitri Routkevitch, Ph.D.
  Product Manager & Principal Scientist - Nanostructured Ceramics
  droutkevitch@synkera.com
  720-494-8401 x102

- Debra Deininger
  Product Manager - Sensors
  ddeininger@synkera.com
  720-494-8401 x105

Corporate

- Stephen S. Williams, Ph.D.
  President and CTO
  swilliams@synkera.com
  720-494-8401 x103

- Brian Sperry
  Vice President, CFO
  bsperry@synkera.com
  720-494-8401 x116