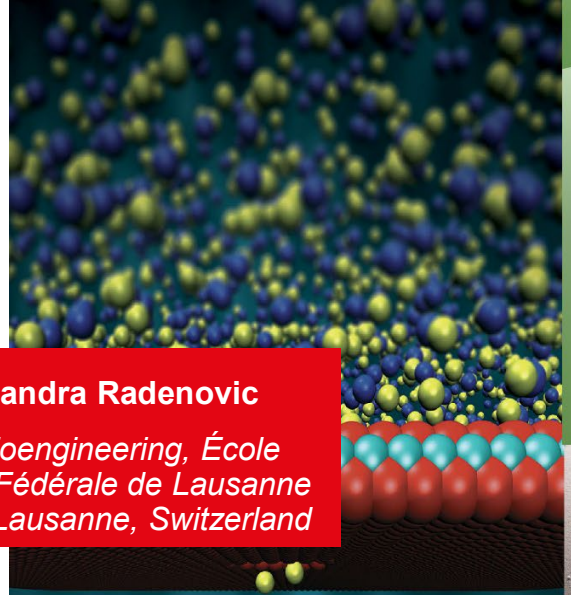
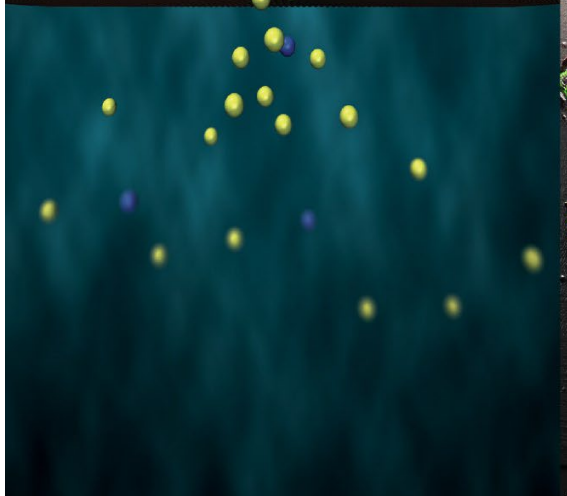
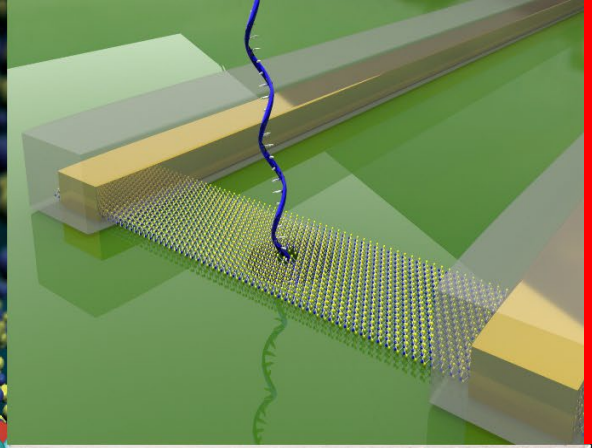
Solid state nanopores from fab to lab

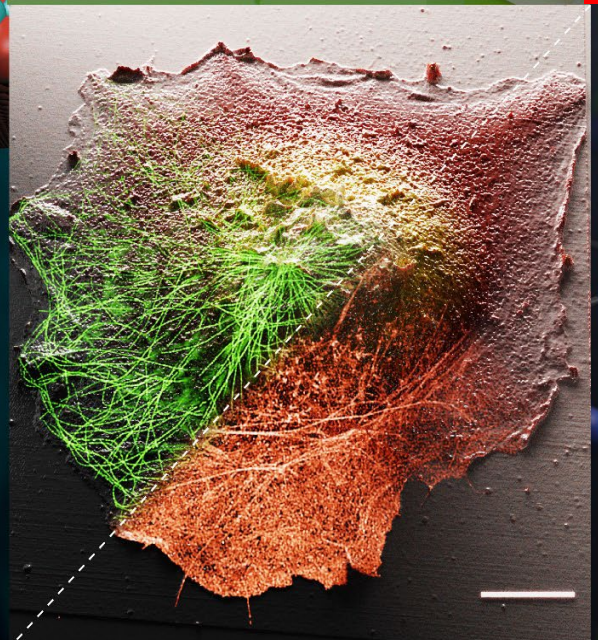
Prof. Aleksandra Radenovic

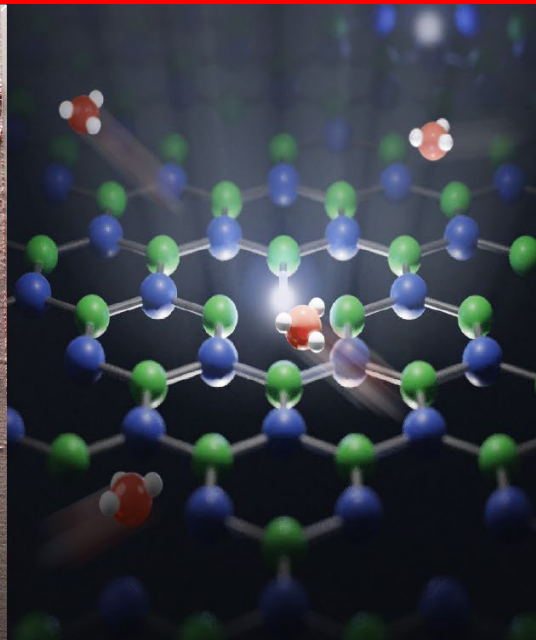
Institute of Bioengineering, École Polytechnique Fédérale de Lausanne (EPFL), 1015, Lausanne, Switzerland





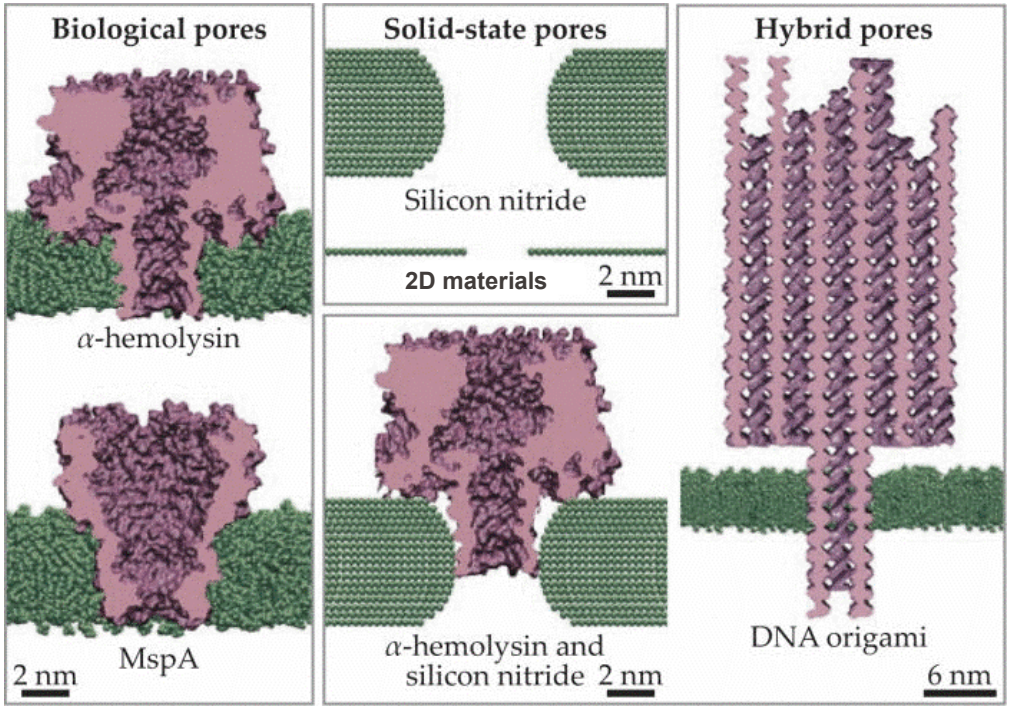




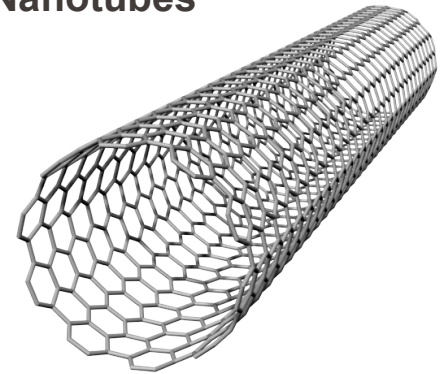


Nanofluidics

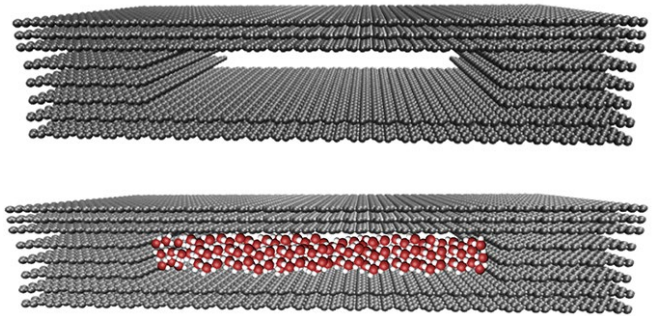
Nanopores



Nanotubes

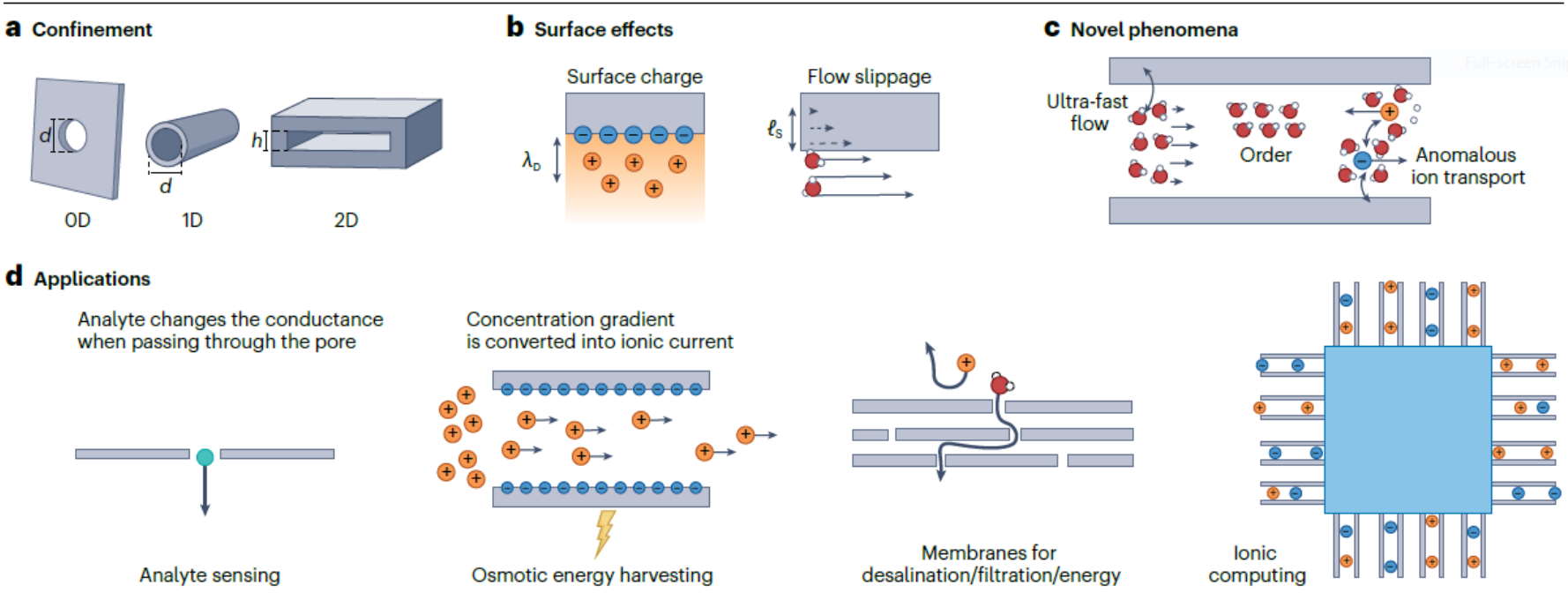


Angstrom-scale slits



- Bocquet, L. *Nanofluidics coming of age.* Nat. Mater. **19**, 254–256

Nanofluidics



Emmerich, T., Ronceray, N., Agrawal, K. V., Garaj, S., Kumar, M., Noy, A., & Radenovic, A. (**2024**). Nanofluidics. *Nature Reviews Methods Primers*, *4*(1), 6.

EPFL **Nanofluidics**

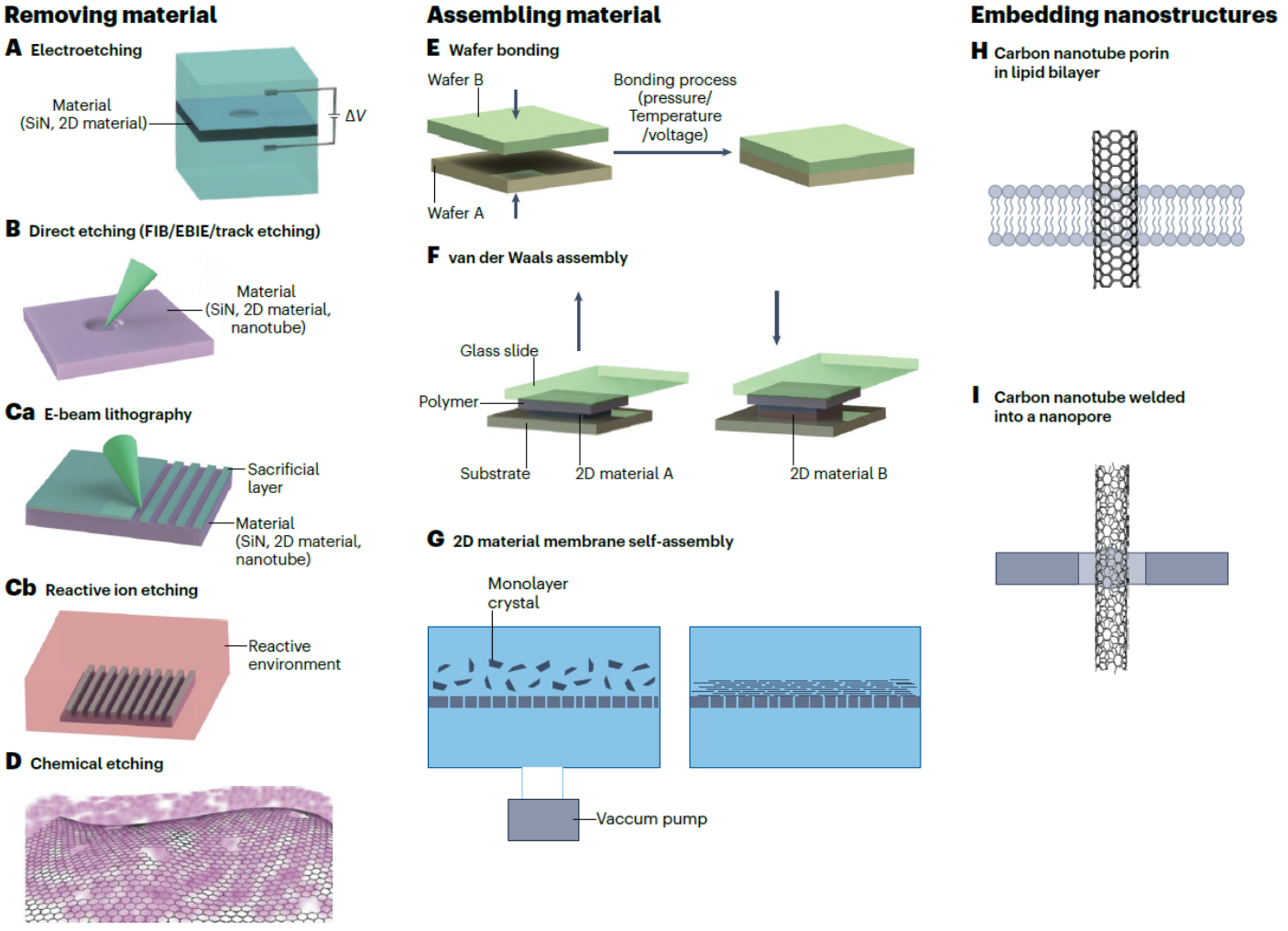


Fig. 2 | Fabrication approaches for nanofluidic devices. A, In situ electro-

Through transfer with polymeric stamps, 2D materials can be stacked on top

- Emmerich, T., Ronceray, N., Agrawal, K. V., Garaj, S., Kumar, M., Noy, A., & Radenovic, A. (**2024**). Nanofluidics. *Nature Reviews Methods Primers*, *4*(1), 6.

Huge Potential of Nanopore Device

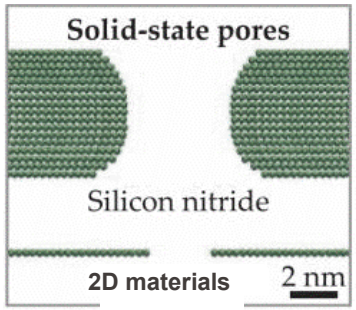
Bio Sensors

Ionic Resources

Blue Energy

Nanofluidics

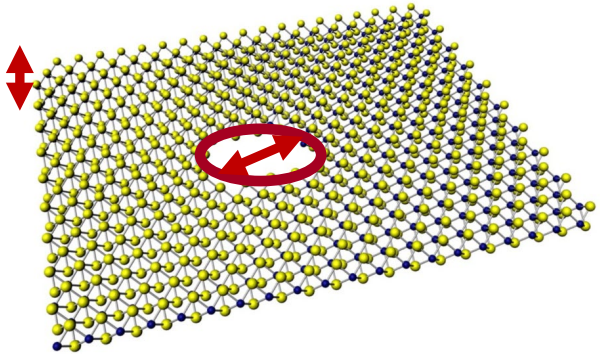
Nanopores

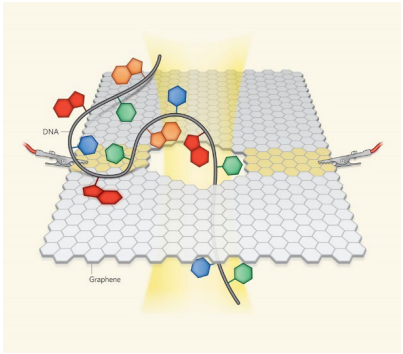


molybdenum disulfide (MoS_2)

L=0.7 nm

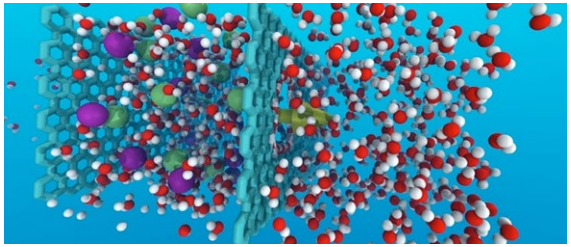
d=tunable

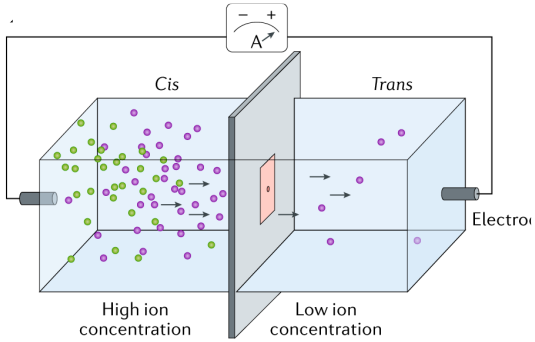




DNA, RNA, protein sequencing

Filtration

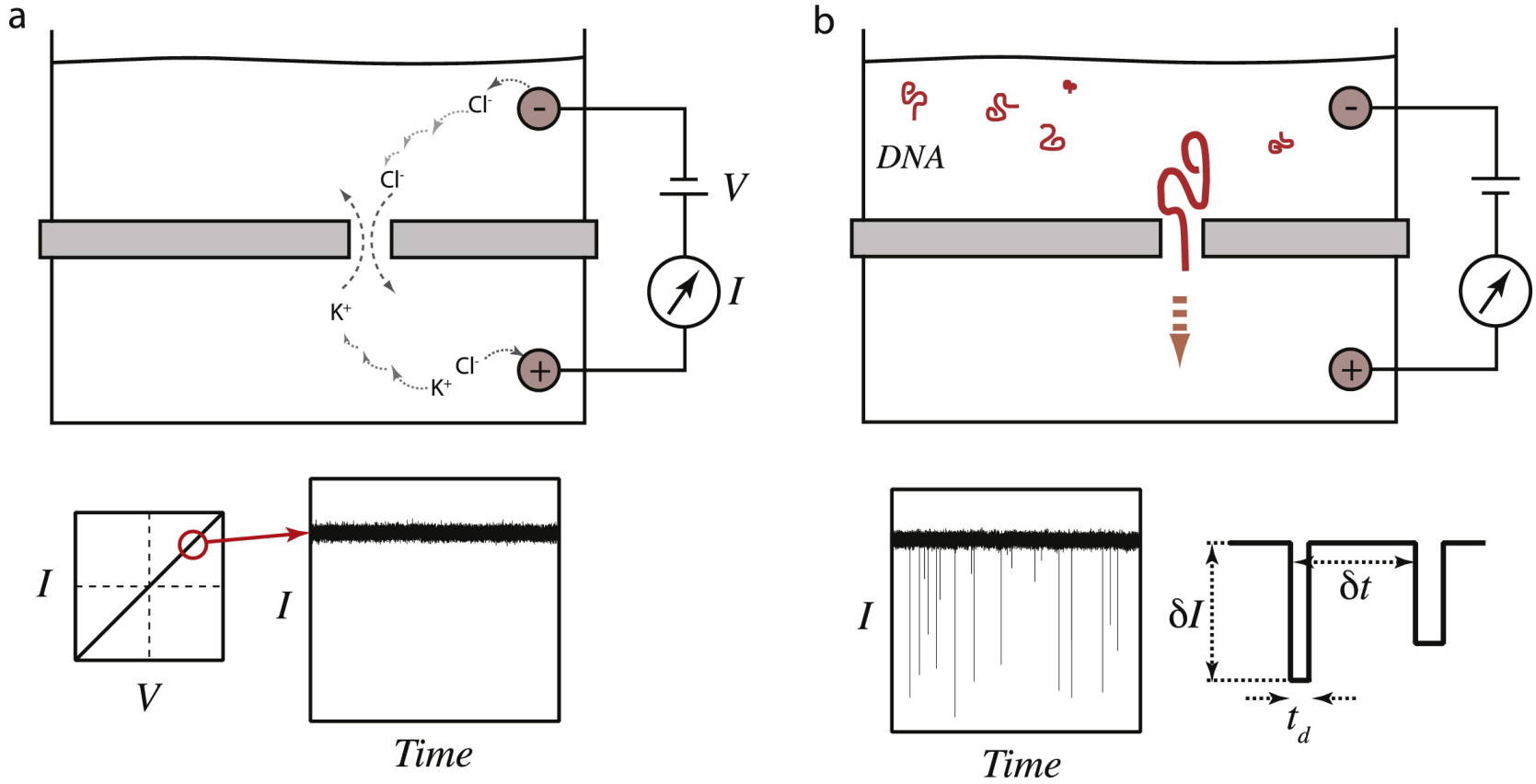




Osmotic power generation

Bocquet, L. *Nanofluidics coming of age.* Nat. Mater. **19**, 254–256

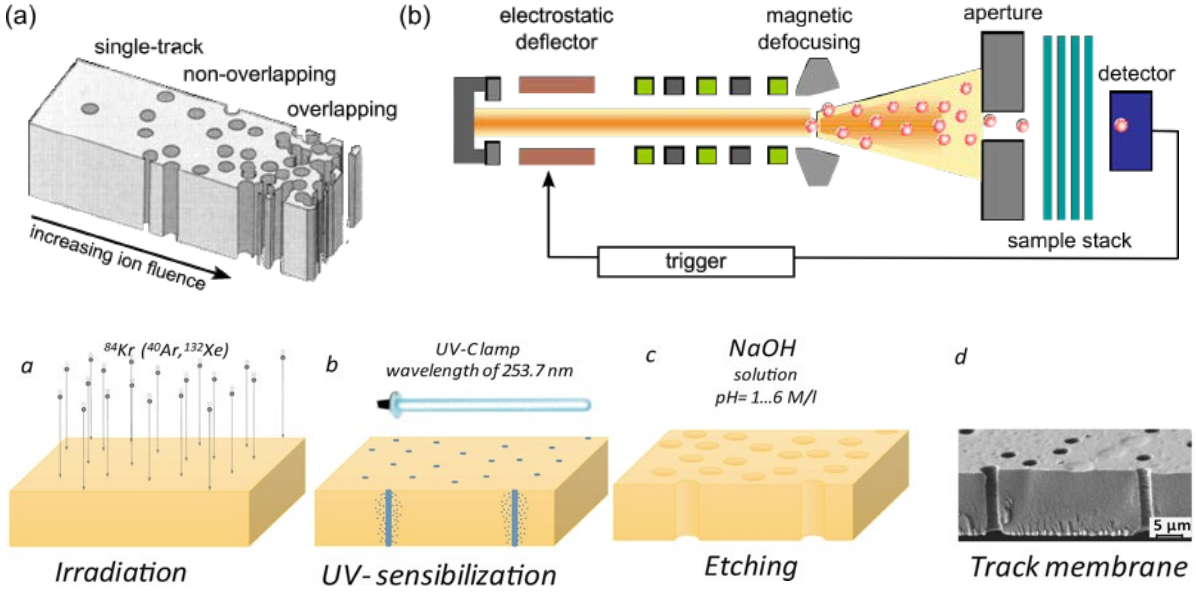
Nanopores



Wanunu, M. (2012). Nanopores: A journey towards DNA sequencing. Physics of Life Reviews, 9(2), 125–158.

Solid-State Nanopore Technology

Polymer & Silicon Nitride platforms



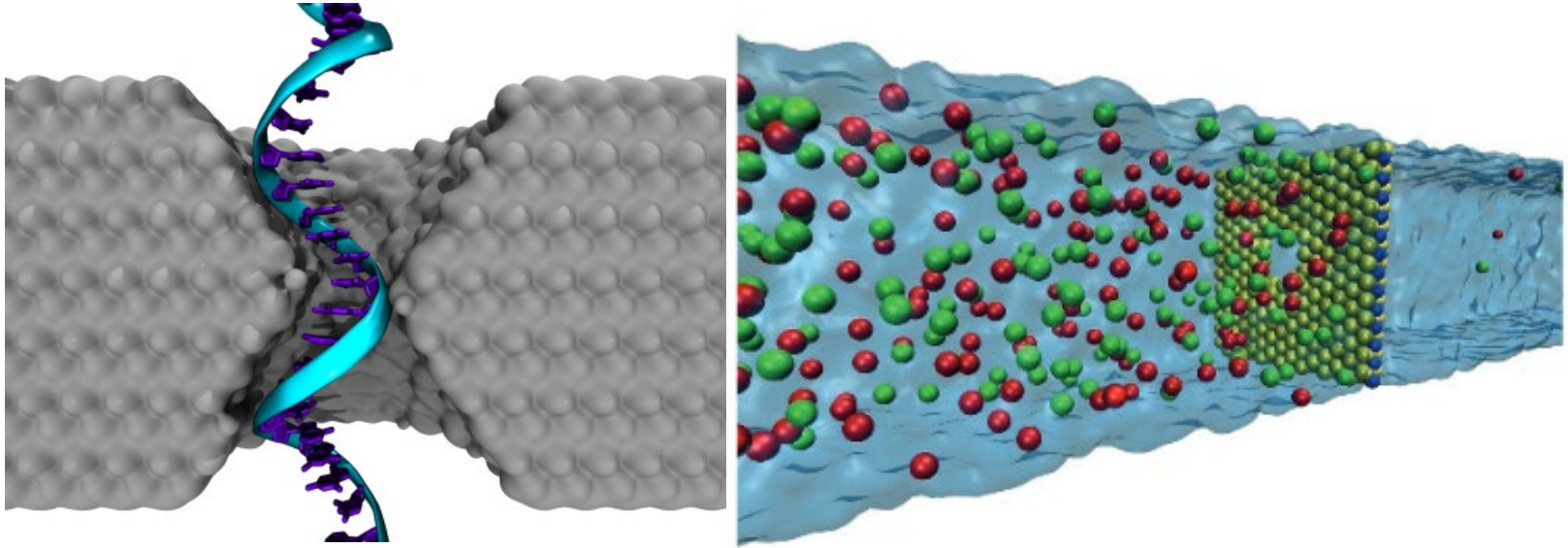
Commercialized Fabrication Technology

Available to various materials & Accessible for different nano-geometry

M. E. Toimil-Molares, Beilstein J. Nanotechnol. 2012, 3, 860.
Kozlovskiy A. et al. Nanocomposites, Nanostructures, and Their Applications. NANO 2018. Springer Proceedings in Physics, vol 221.

Solid-State Nanopore Technology

Polymer & Silicon Nitride Platform (Relatives: Dielectric, 2D Materials, …)

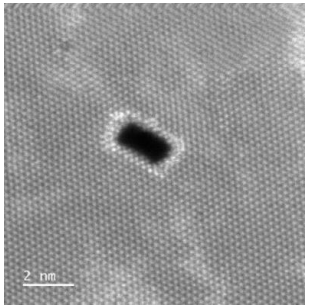


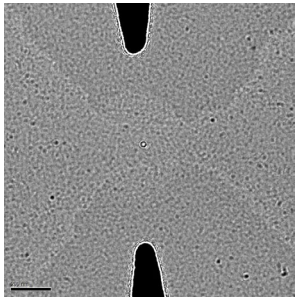
https://phys.org/news/2013-05-advance-nanotech-gene-sequencing-technique.html

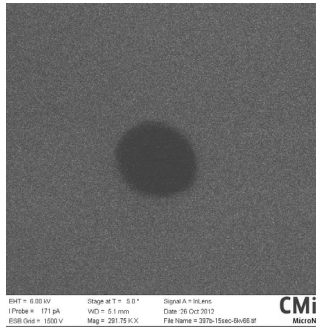
Feng, J. et al. Single-layer MoS2 nanopores as nano power generators. Nature 536, 197–200 (2016).

Solid-State Nanopores

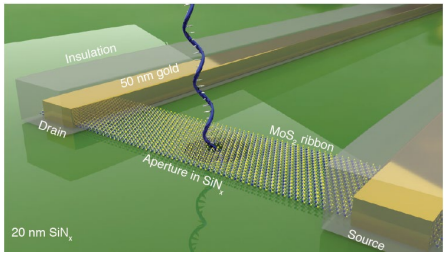
- Different materials



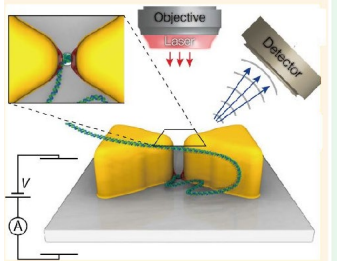




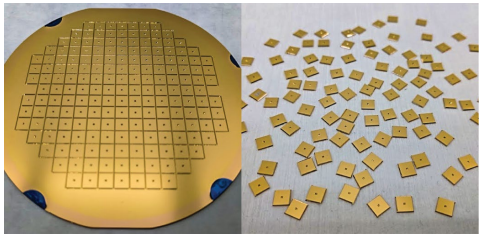
- Integration with electrical sensing

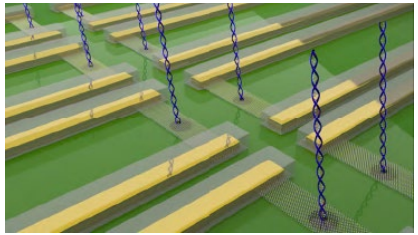


- Integration with optical sensing



- High-throughput production





- Graf, ….Radenovic *Nano Letters* **2019.**

Bioinspired Solid State Nanopores

Background Information

Clean Room Technology

Applications

Further Plan

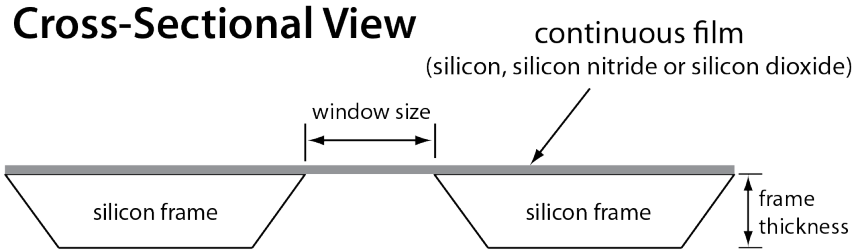
Acknowledgement

From TEM Sample Grid to
Universal Research Platform

Silicon Nitride freestanding membrane is designed for TEM window grid. **(Silicon Oxide)**

- **Simple Substance Composition.**
- **High Surface Flatness.**
- **High Chemical Tolerance.**
- **High Electronic Transparence.**

- **Cleanness**
- **Controllable Structure**
- **Stability**
- **Thickness**



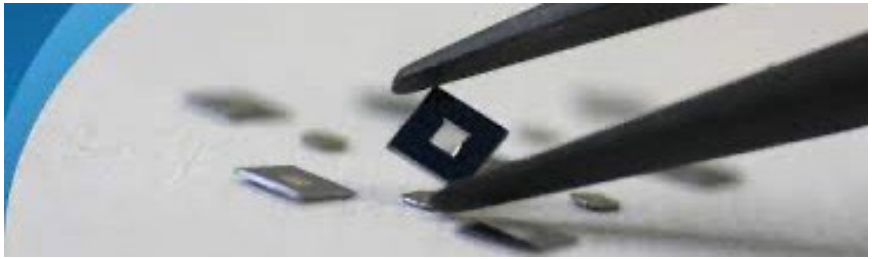
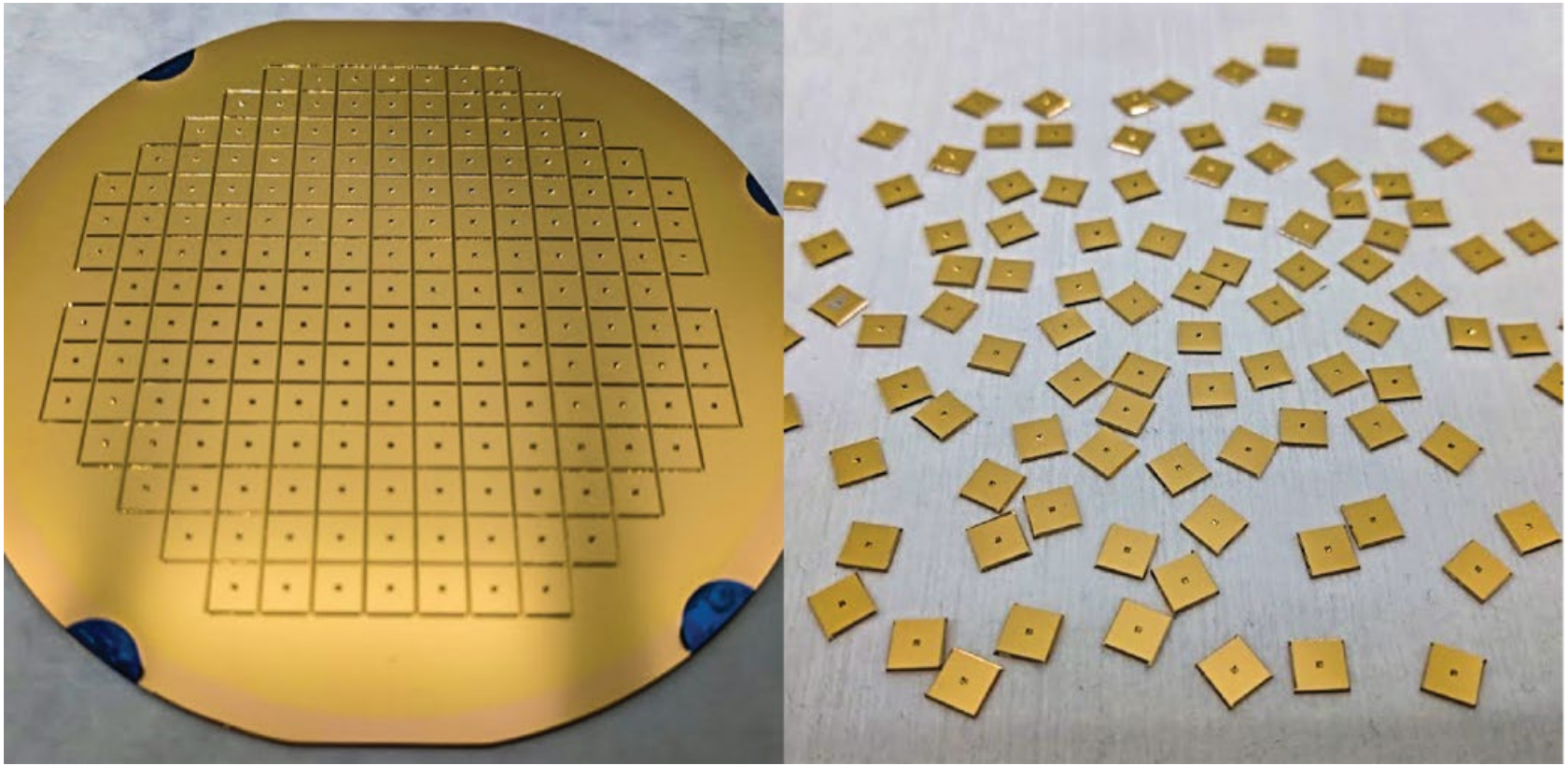


Image from Norcada

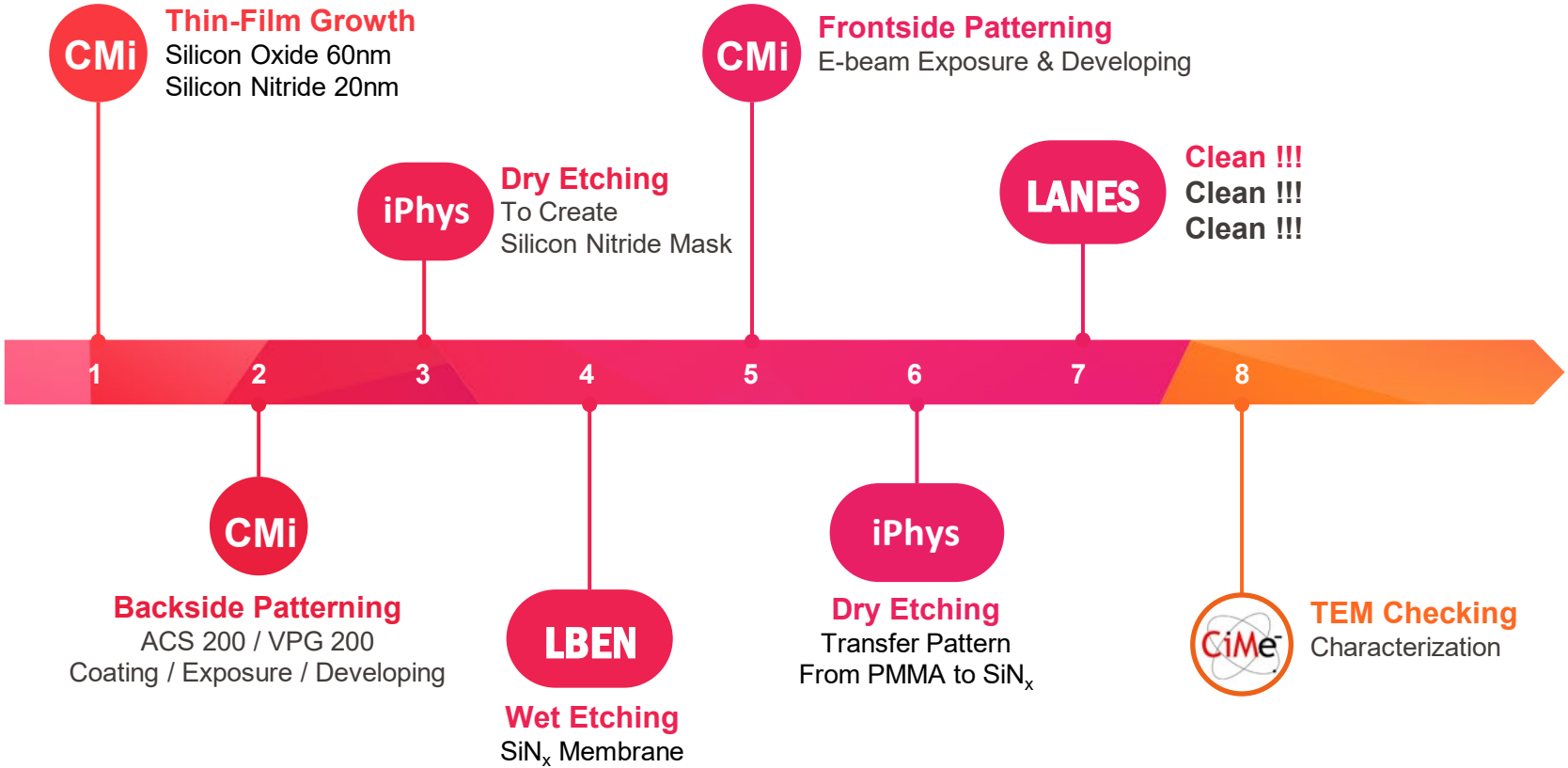
Wafer scale device production



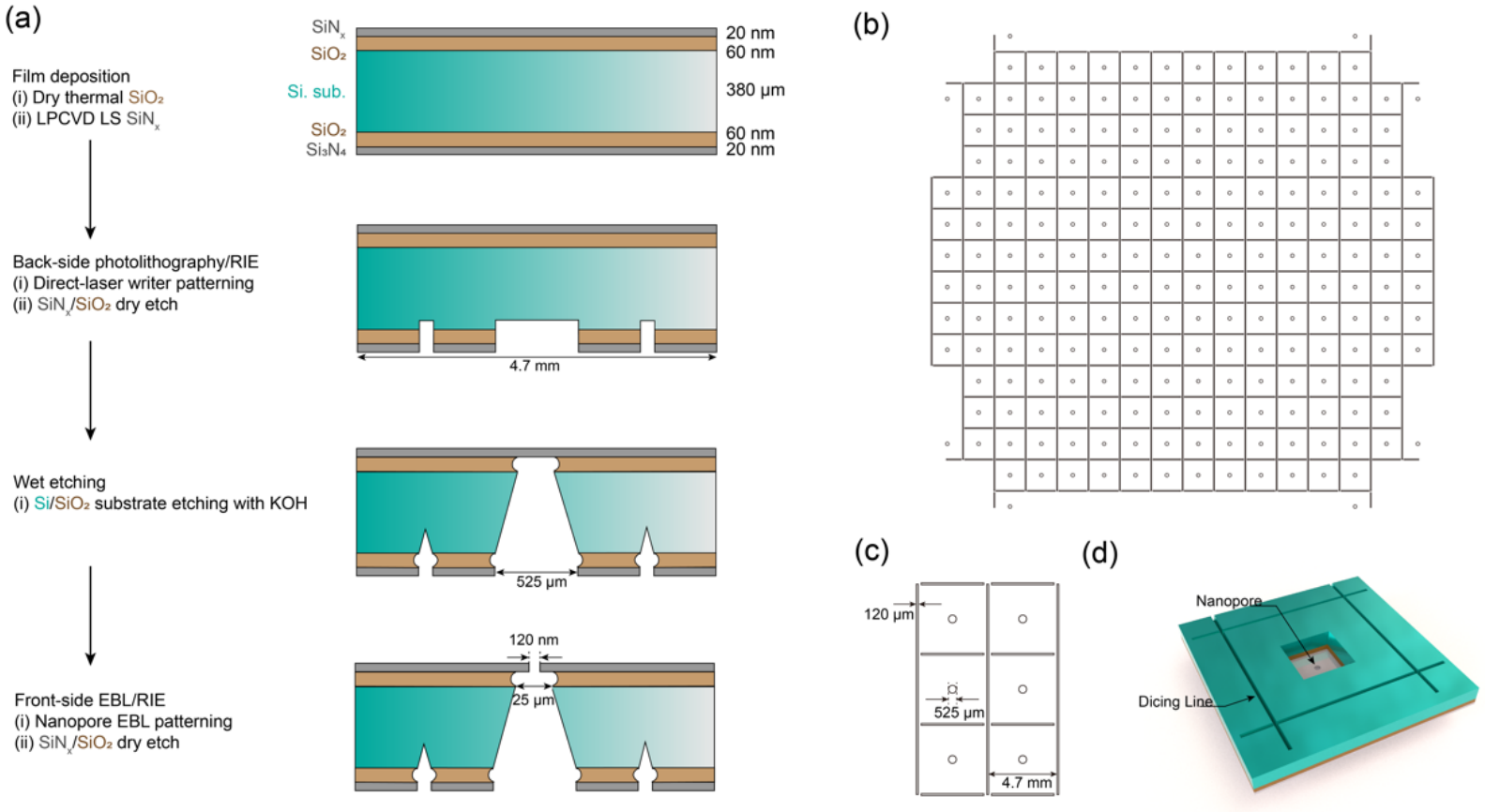
Graf, Lihter, Thakur…Radenovic *Nature Protocols* **2019.**

4-Inches Wafer-Scale Fabrication

Mask-Free Procedure



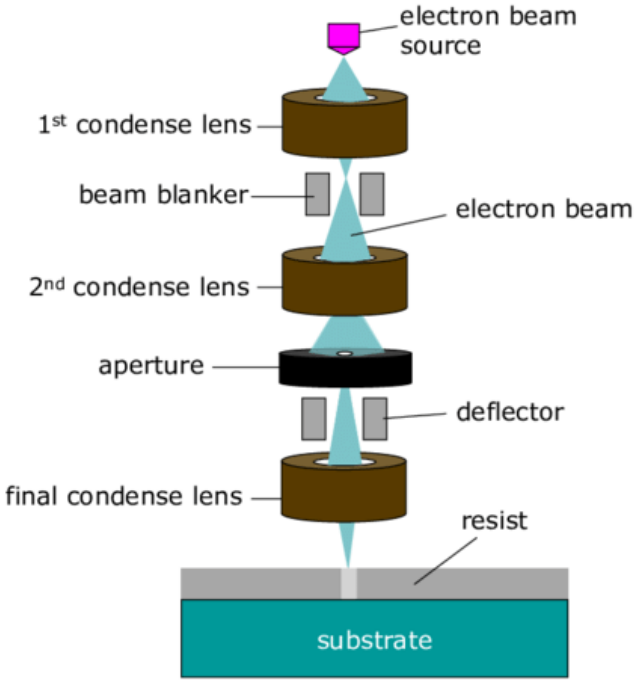
How do we get our SiN_x substrate platforms?

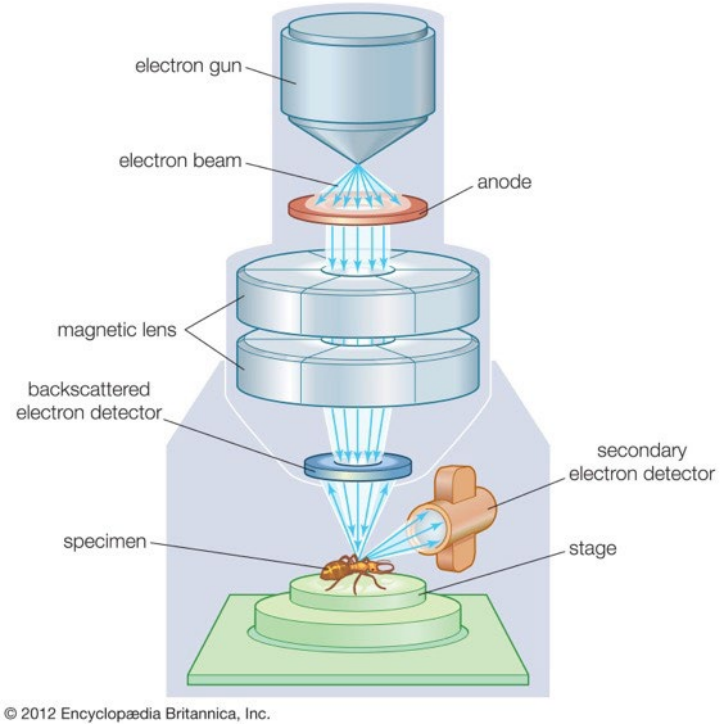


npj 2D Materials and Applications, 2022, under review

Advantage of E-beam Lithography

Direct Writing Technology

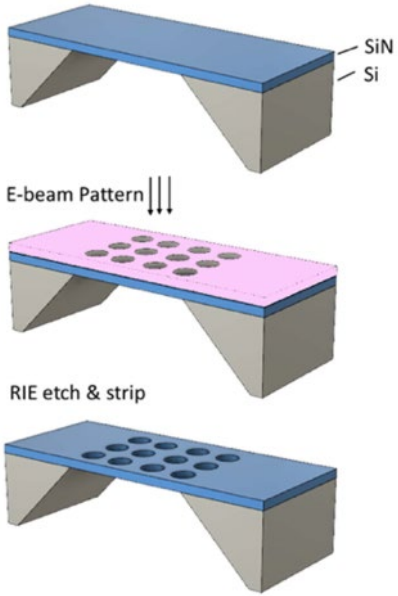


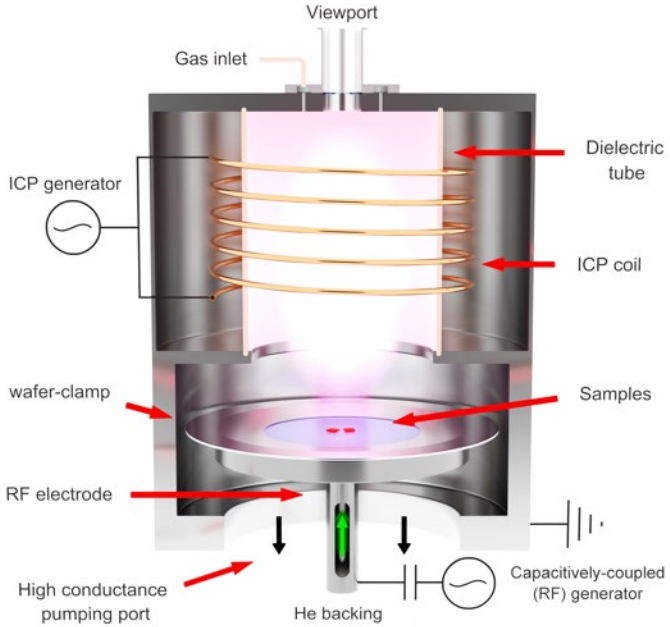


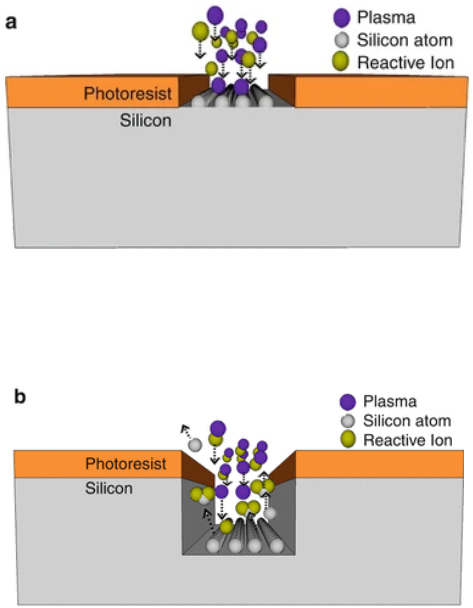
- **Electron Beam Lithography** is similar as **Scanning Electronic Microscopy**
- **Deflector** of E-beam Lithography: Direct Writing Realizing Customizing Design

A. Pimpin, et. al. Eng. J., 16, 1, 37-56, 2011.

Reactive Ion Etching (RIE) – Fabricate Pores





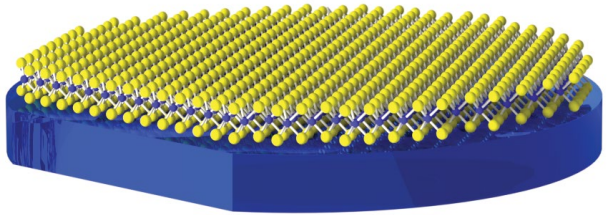


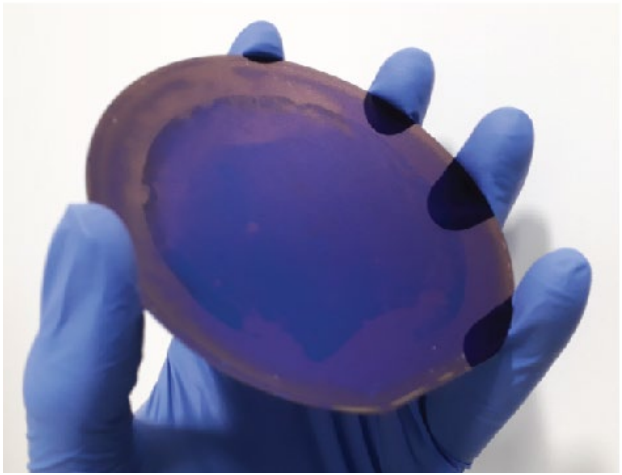
- **Reactive ion etching (RIE)** is a plasma process where radiofrequency (RF) discharge-excited species (radicals, ions) etch substrate or thin films in a low-pressure chamber.
- **High Selectivity & High Anisotropy & High Etching Rate**

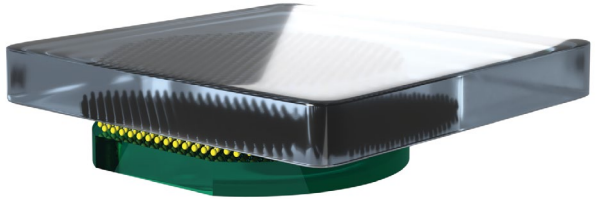
Verschueren, D. V., Yang, W. & Dekker, C. Lithography-based fabrication of nanopore arrays in freestanding SiN and graphene membranes. Nanotechnology 29, 145302 (2018).
Godin, B. et al. Encyclopedia of Nanotechnology. 587–589 (2012).
Hönl, S., Hahn, H., Baumgartner, Y., Czornomaz, L. & Seidler, P. Highly selective dry etching of GaP in the presence of AlxGa1–xP with a SiCl4/SF6 plasma. J Phys D Appl Phys 51, 185203 (2018).

Wafer scale transfer

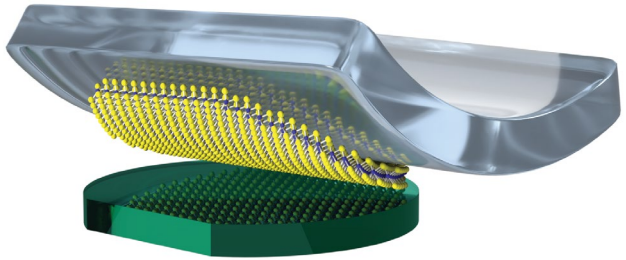
Complete dry-transfer of MoS_2 on SiNx wafer (nanopore devices)



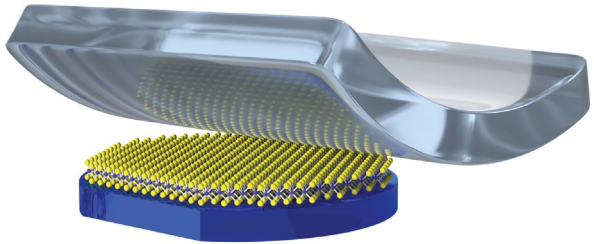




PDMS/MoS_2 on sapphi re subst rate

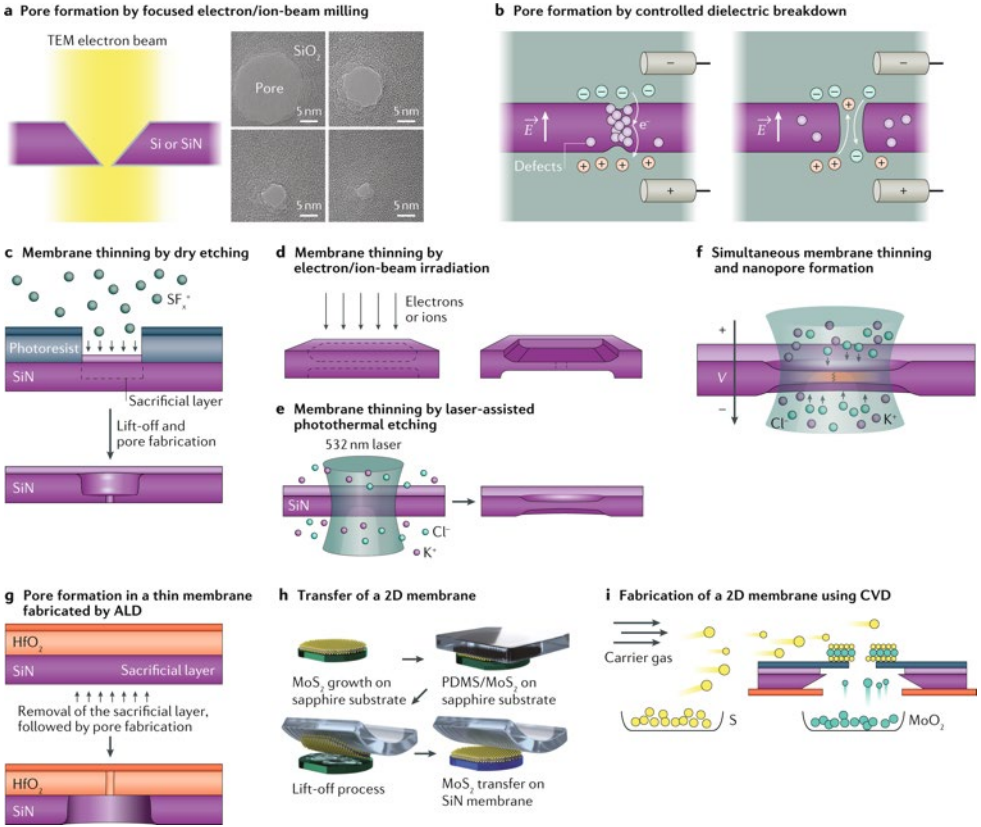


Water-assisted lift-off process



Thakur, Macha…. Radenovic *Small Methods* **2020.**

Different Materials & Fabrication Strategy



Fabrication Strategy

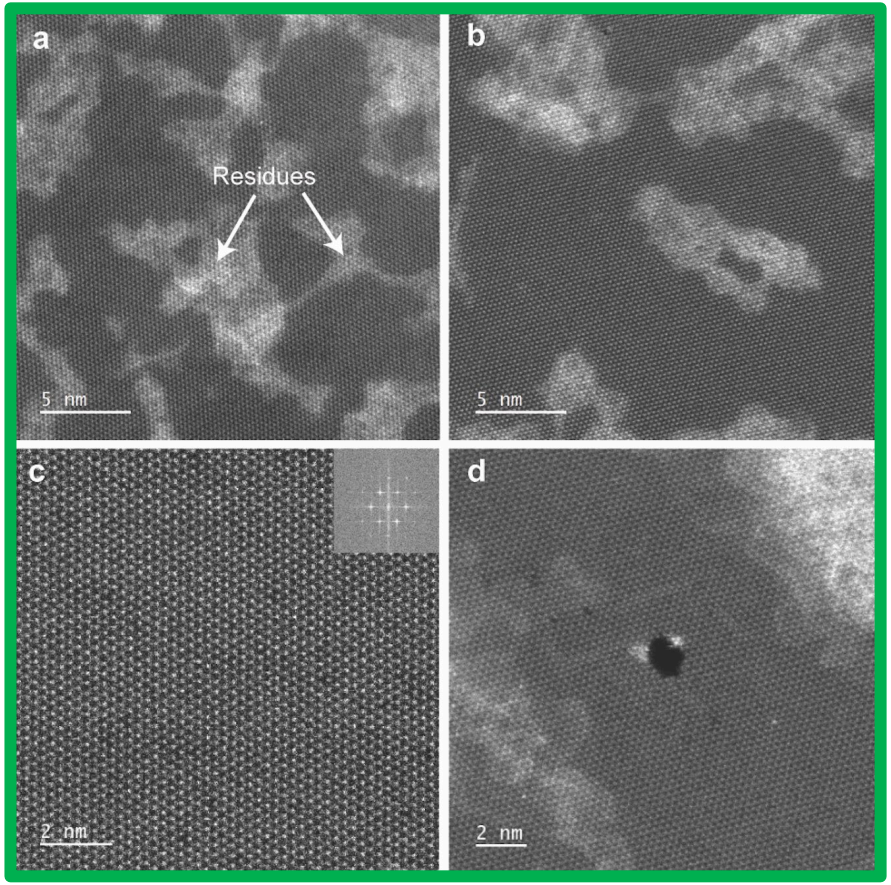
- TEM Drilling
- Dielectric Breakdown
- Dry Etching
- Photothermal Etching
- Drilling on ALD-additional Layer
- Transferred MoS2 Nanopore
- CVD MoS2 Nanopore

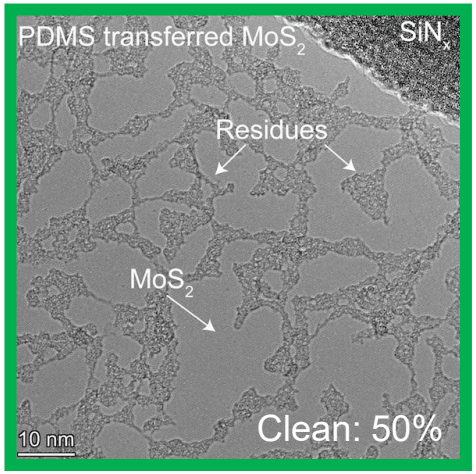
Materials

- Silicon Nitride
- HfO2 (Dielectric materials)
- 2D Materials (MoS2 Graphene …)

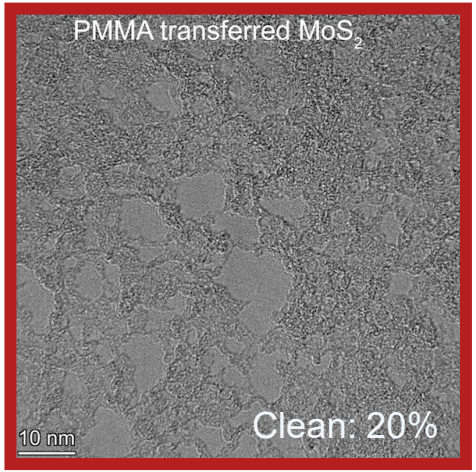
Xue, L. et al. Solid-state nanopore sensors. Nat Rev Mater 5, 931–951 (2020).

TEM: COMPARISON OTHER METHODS





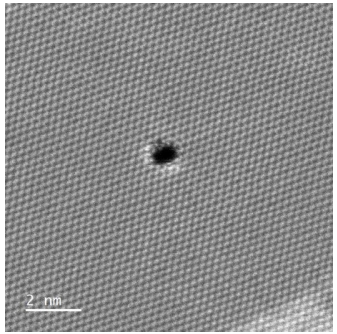
PDMS

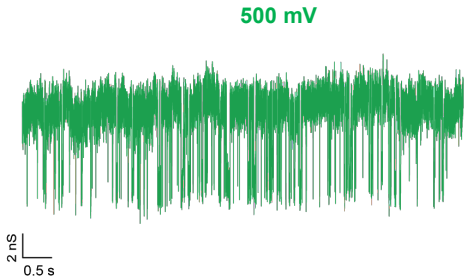


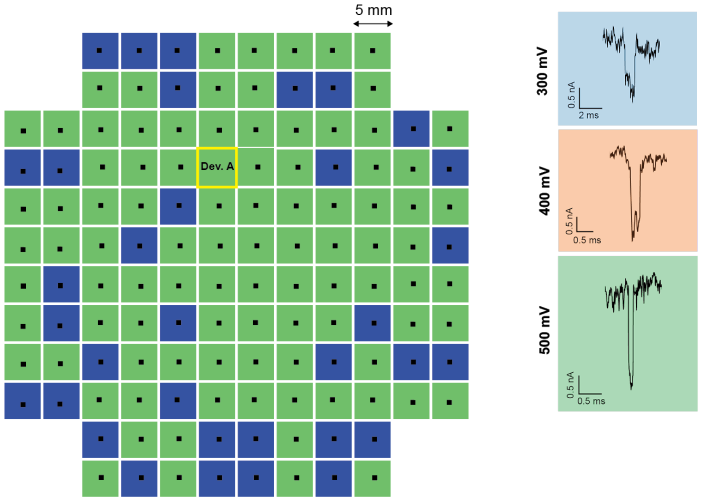
PMMA

Thakur, M., Macha, M. et al. Small Methods, (2020).

Nanopore devices benchmarking



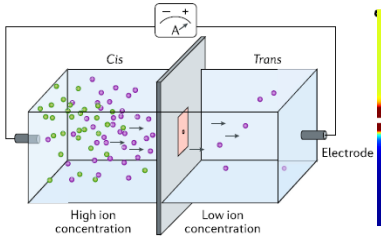


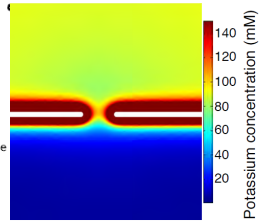


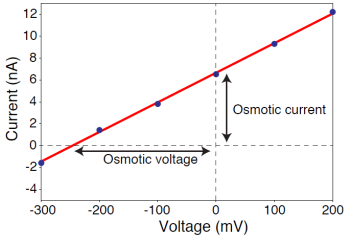
- Thakur, Macha. Radenovic *Small Methods* **2020.**

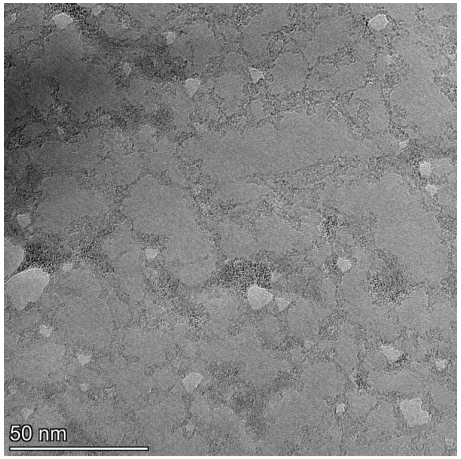
concentration gradient + ion selectivity = net current

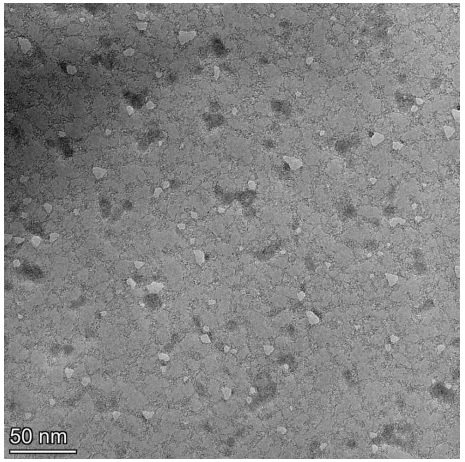
Potassium selective











Feng ….Radenovic *Nature* **2016**.

Macha, Marion… Radenovic *Nature Rev. Materials* **2019**.

Macha, Marion… Radenovic *ACS Nano* **2023**.

Disadvantage of Silicon Nitride

Limited Surface Properties & Relatively Poor Stability

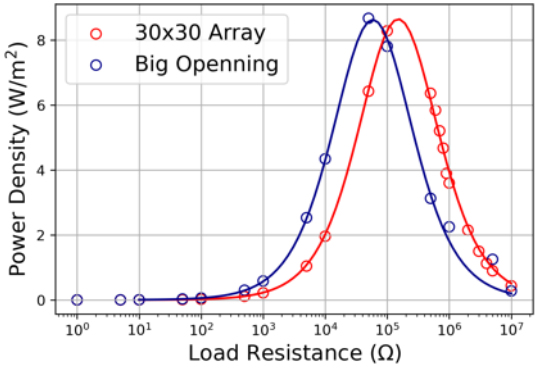
Limited Surface Properties

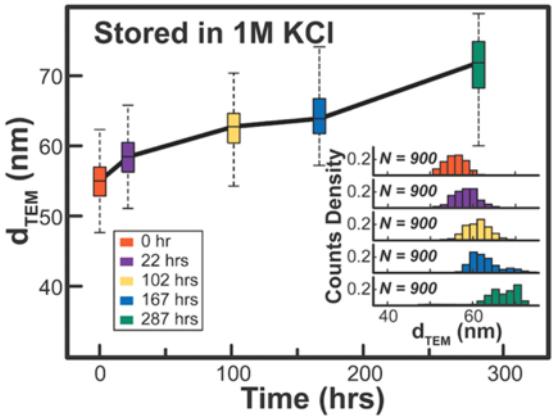
- Limited Surface Charge
- Limited Output in Osmotic Energy Harvesting

Limited Stability

- Nanopore Enlargement due to the Dissolving
- Lower Stability in electrolyte solution
- In Industrial Application Scenarios Require

Working for Months !



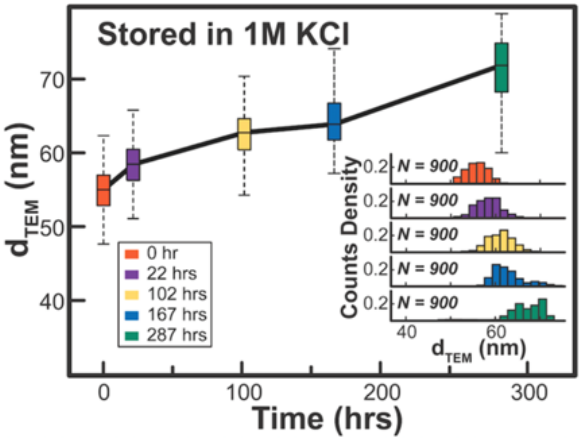


Chou, Y.-C., Das, P. M., Monos, D. S. & Drndić, M. *Acs Nano* **14**, 6715–6728 (2020).

Stability

Limited Stability

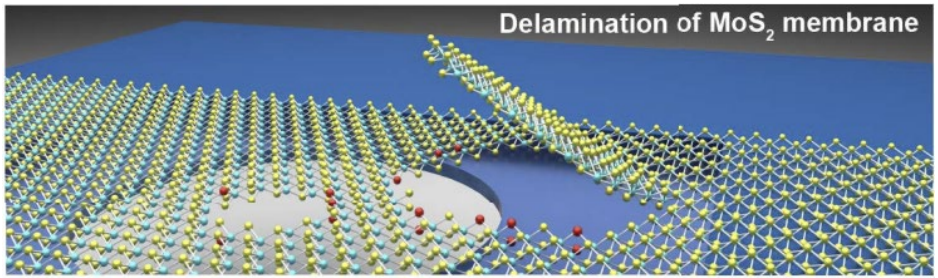
- Nanopore Enlargement due to the Dissolving
- Lower Stability in an electrolyte solution
- Industrial Applications Require long-term stability



Chou,., Das,….. & Drndić, M. *Acs Nano* **2020.**

Working for Months!

- Reinforcing adherence of 2D material to the substrate of the silicon nitride platform



Thakur, Cai,… Radenovic *npj 2D Mater Appl* **2023**

Thin-film Technology

How to Improve SiNx Nanopore with Microfabrication Compatible Process.

Thin-film Technology

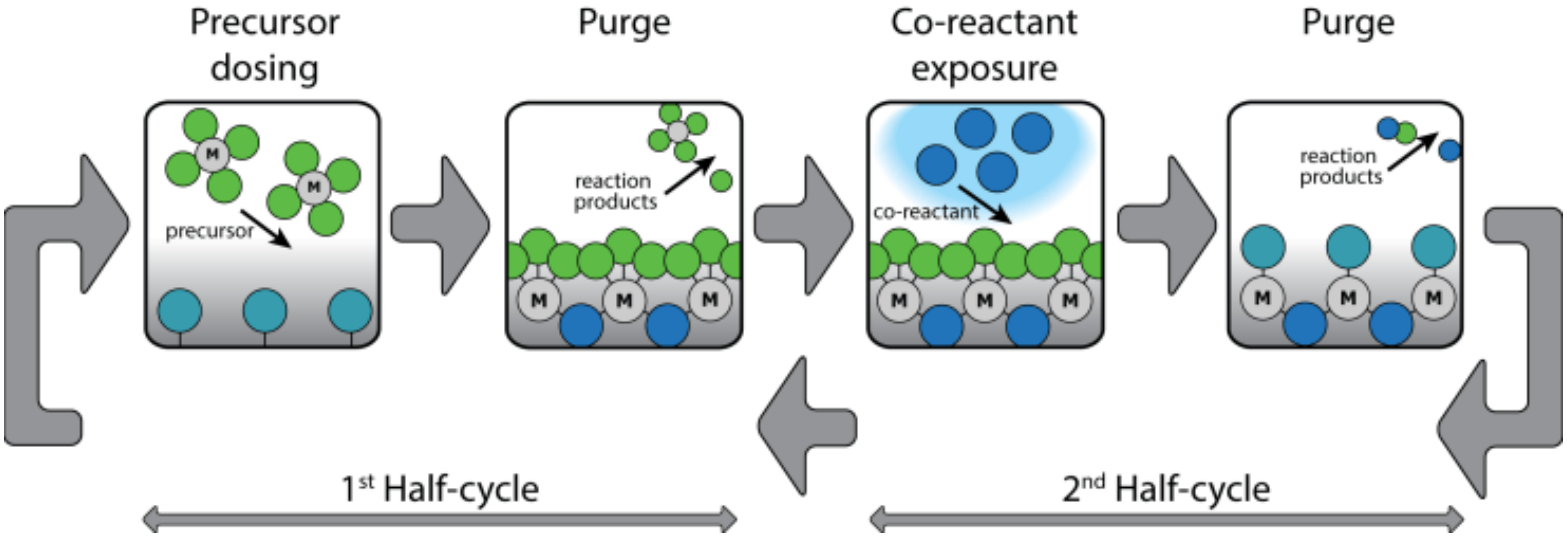
- Introducing thin layer of specific materials
- Usage
  - Changing Surface Property
  - Providing / Avoiding Electric Conducting
- Deposition Method
  - Chemical Deposition
  - Physical Deposition

CVD
Chemical Vapor Deposition

ALD
Atomic Layer Deposition

How ALD Work?

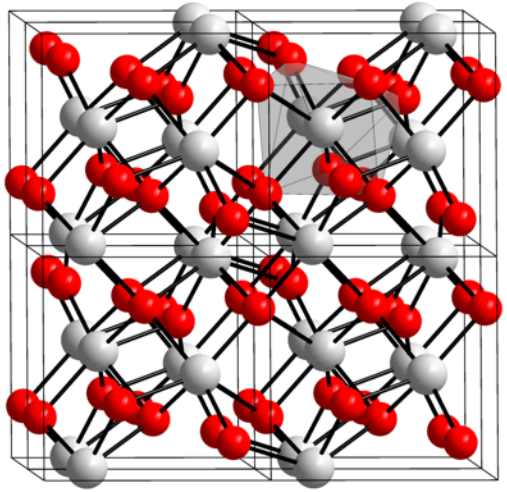
Atomic Layer Deposition

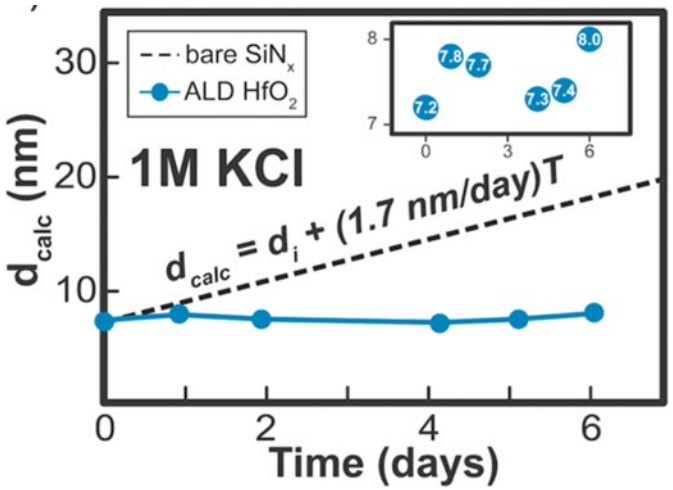


- **Atomic layer deposition (ALD)** is a thin-film deposition technique based on the sequential use of a gas-phase chemical process; it is a subclass of chemical vapor deposition **(CVD).**
- **Uniformity & Self-Limiting**

Hafnium(IV) oxide Functionalized Nanopore

High Stability Materials





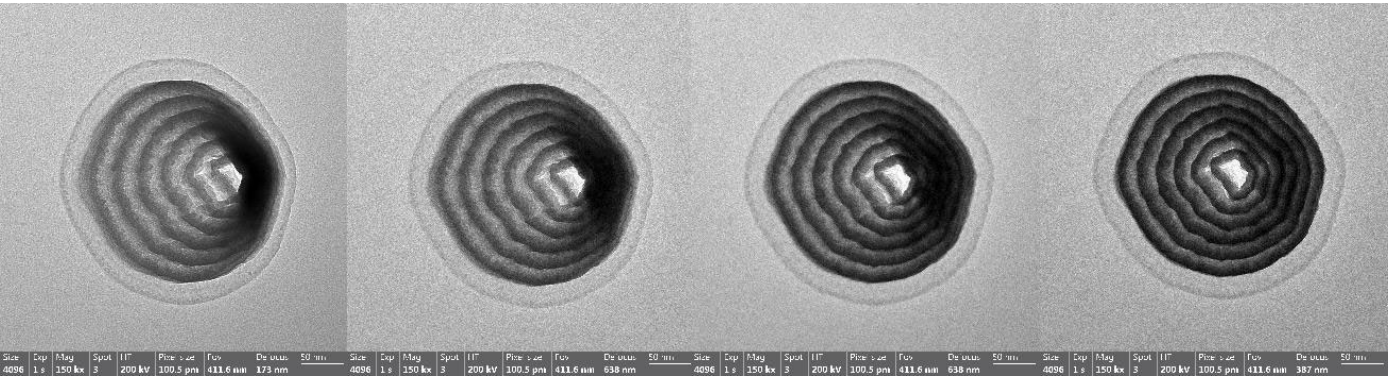
- **Hafnium(IV) oxide**
- Introduced by **Intel** for gate insulator in field-effect transistors
- The dielectric constant of HfO_2 is **4–6 times higher** than that of SiO_2

- **Nanopore Application [Present Research]**
- HfO2 functionalized Nanopore (ALD)
- Stability in Room Temperature KCl Solution **[6 Days]**
- Stability in 60°C KCl Solution under 532nm Laser Irradiation **[10 mins]**

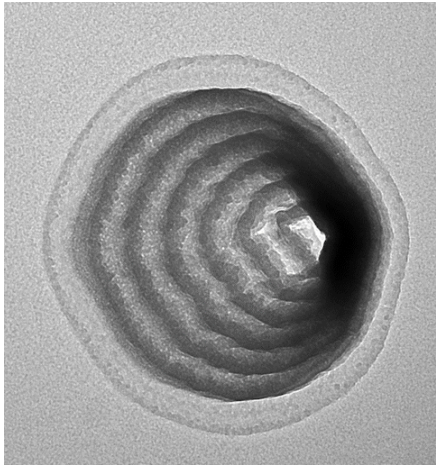
Chou, Y.-C., et. al. *Acs Nano* **14**, 6715–6728 (2020).
Yamazaki, H.,et. al. *Acs Nano* **12**, 12472–12481 (2018).
en.wikipedia.org/wiki/Hafnium(IV)_oxide

HAADF STEM imaging

Insight on the 3D Morphology by Tilting

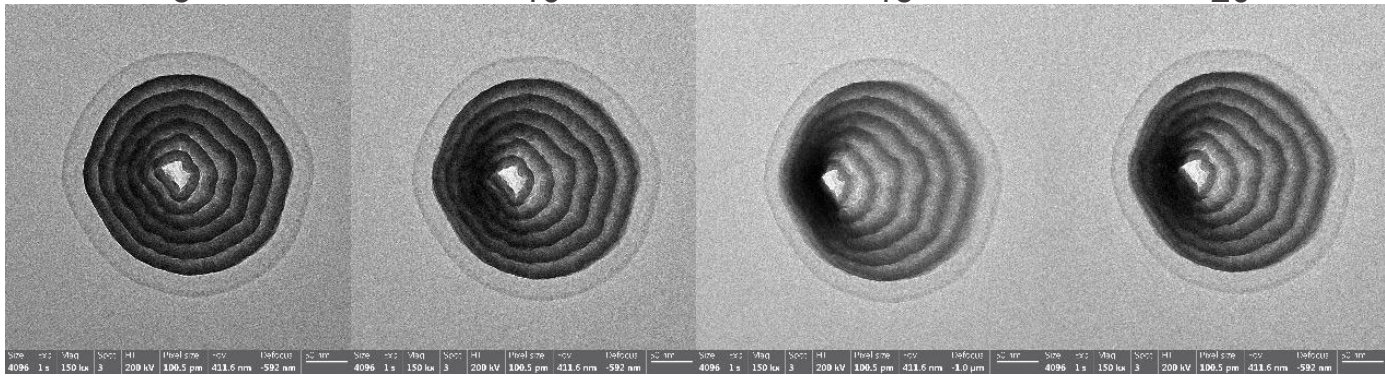


-20 -15 -10 -5



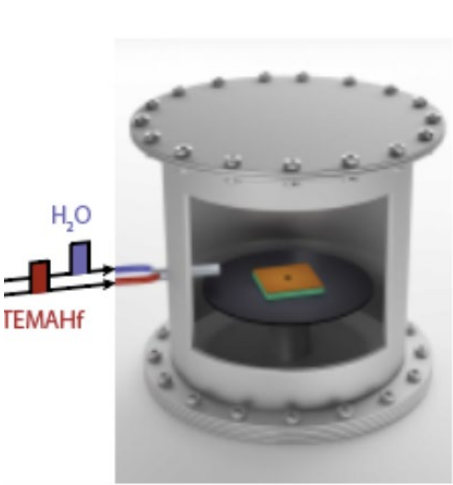
Tilt angles

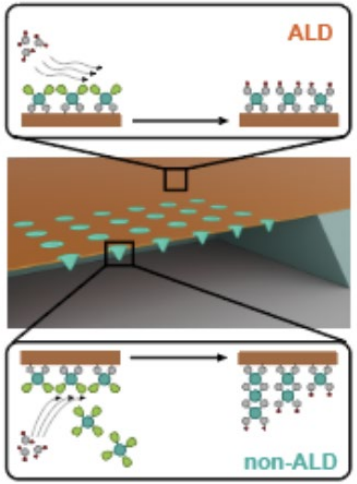
5 10 15 20

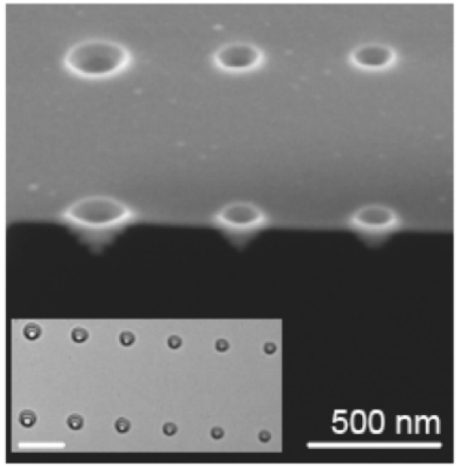


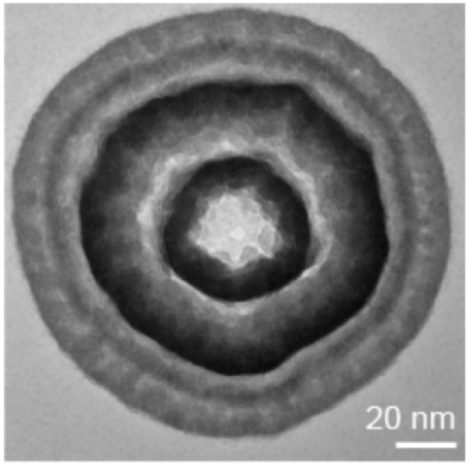
Thin-film Technology for Silicon Nitride Aperture Functionalization

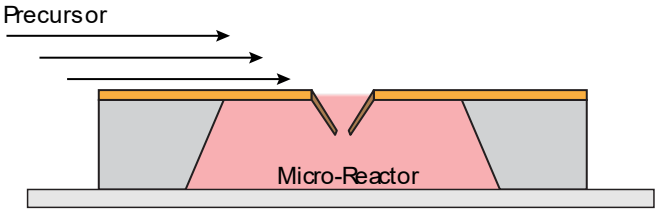
Atomic Layer Deposition (H_2O + TEMA-Hf)

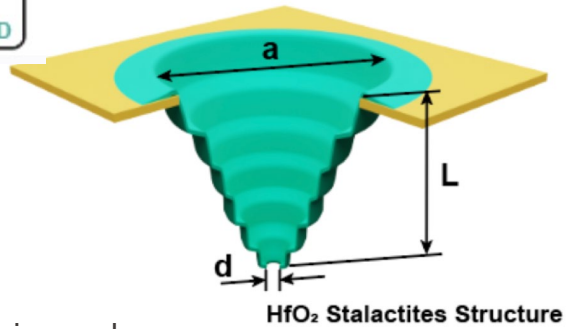


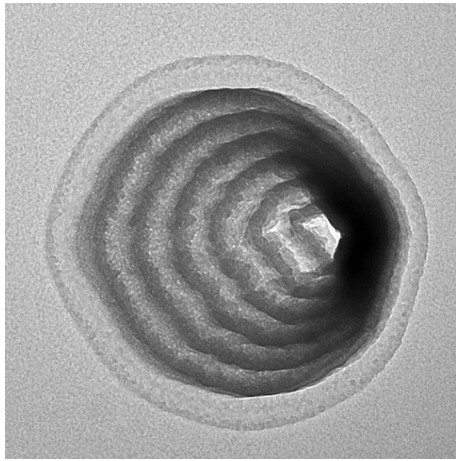












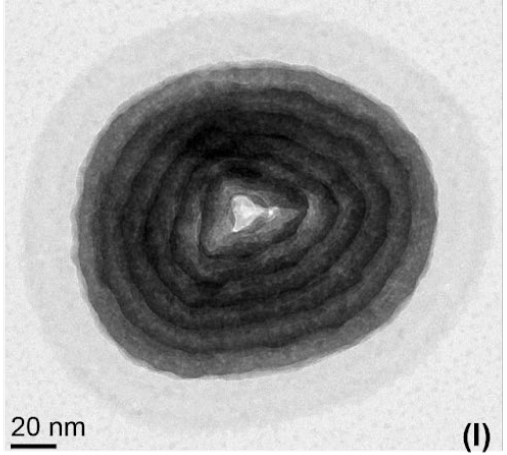
Nature-inspired stalactite nanopores for biosensing and energy harvesting Chernev *, Teng*, …….Radenovic* Advanced Materials **2023**

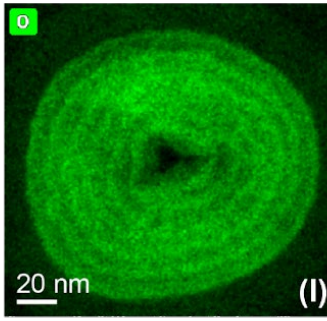
- PCT 23 170 779.5

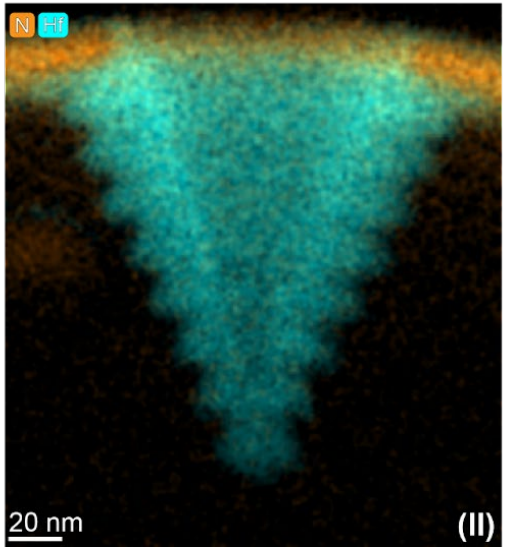
HfO_2 Stalactite-like Nano-Structure

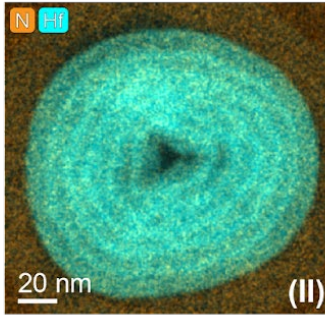
Elemental analysis with STEM

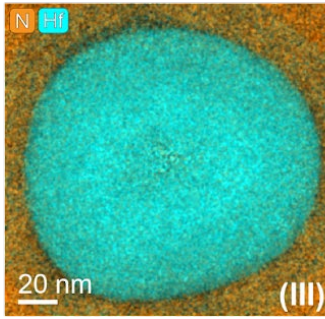
- **Cross-section**
  - Localization Hf and N
  - Corresponding to HfO_2 and Silicon Nitride
- **Composition recognition**
  - HfO_2
  - Silicon Nitride
  - Homogenous distribution inside HfO_2 nanostructure.











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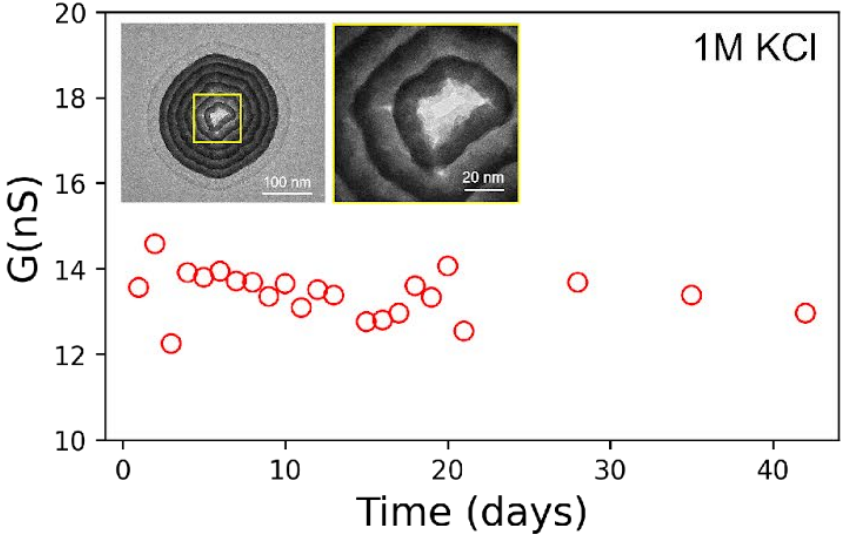
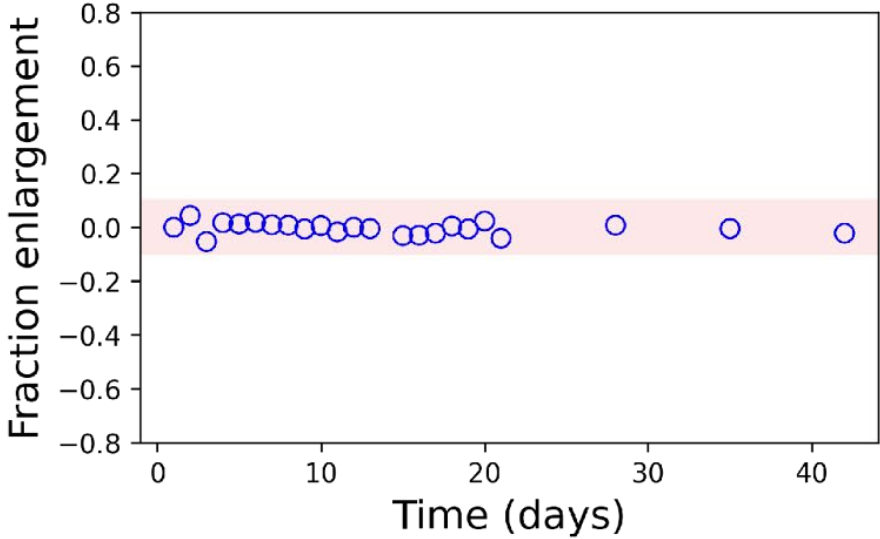
Stability of HfO_2 Nano-Structure

- **Conductance of 1M KCl electrolyte solution in 42 days**
- **Variation with in 10%**





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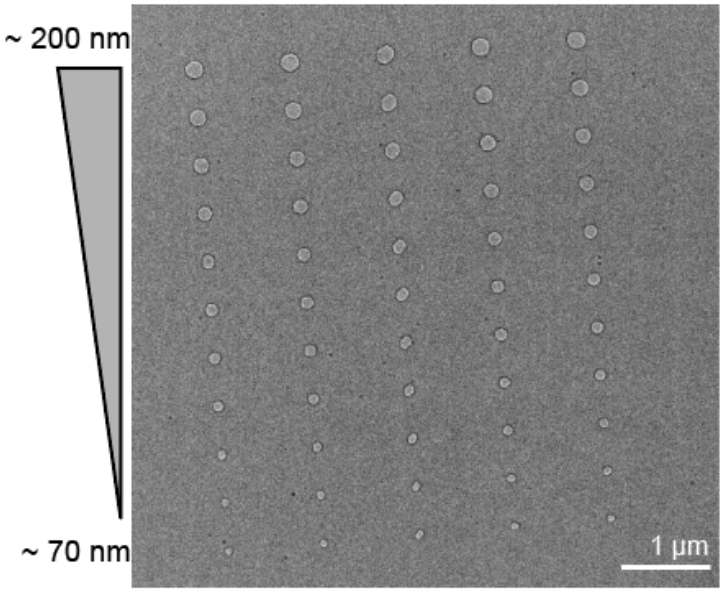
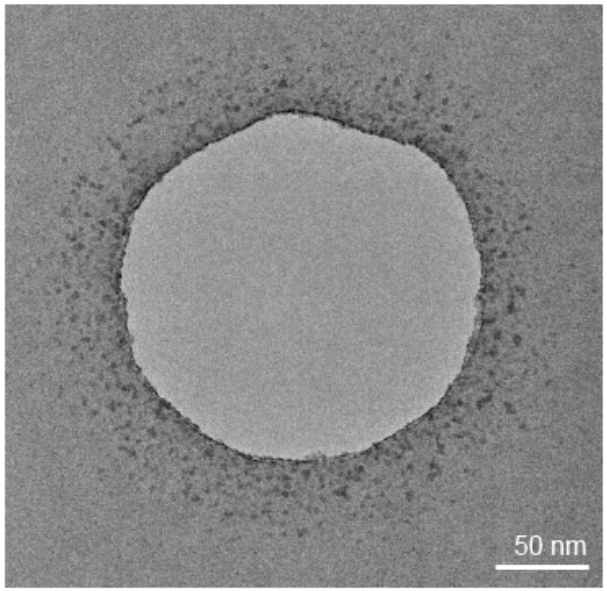
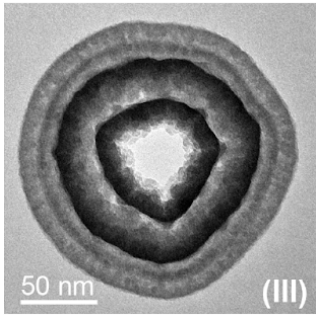
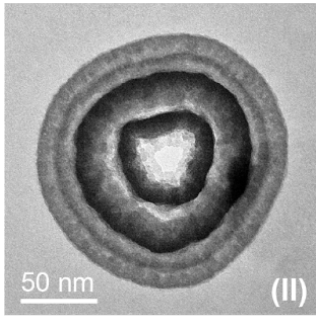
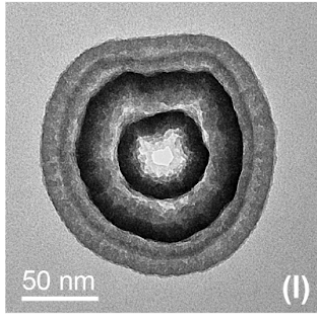
Tuning Growth Behavior with Geometry / Process parameters/











- The number of steps in HfO_2 Nanostructure (n) => Steps of the growth process (n+1)
- Critical Parameter in Nanostructure (Diameter)=> Different Starting Diameter of Aperture
- Whether Starting the non-ALD Growth Process=> Different Purging Time

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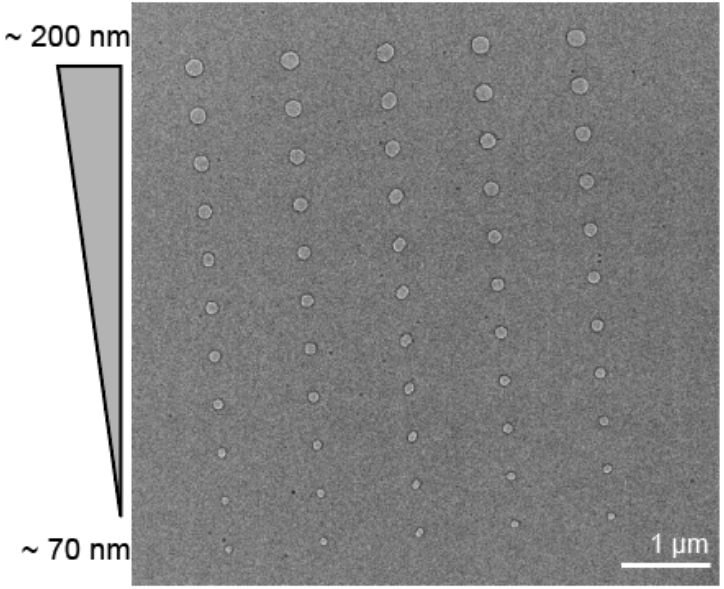

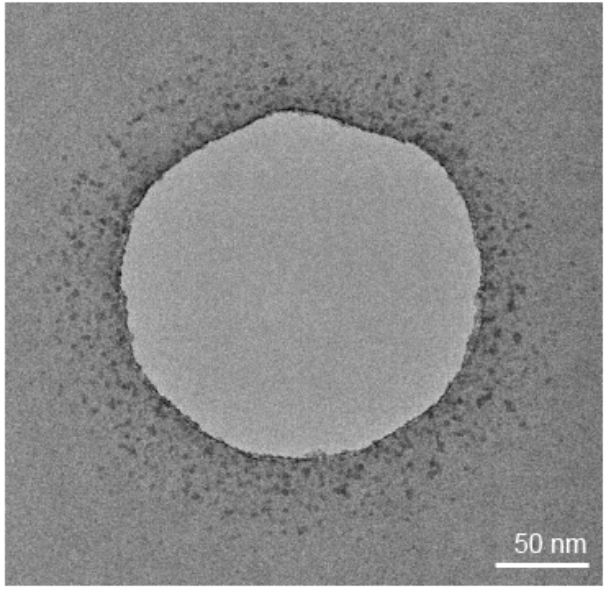

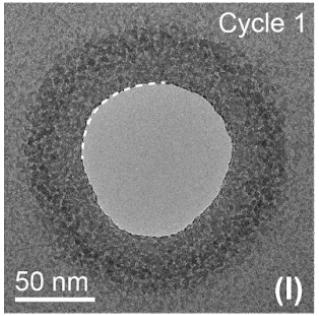

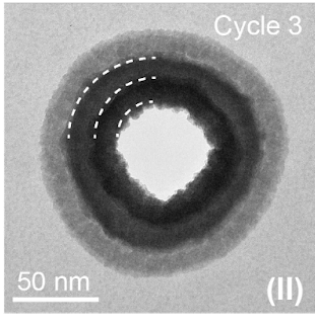

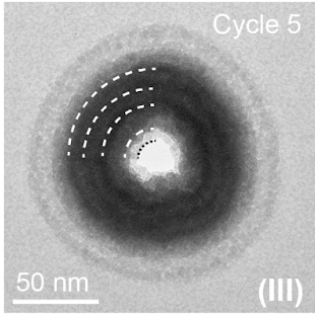

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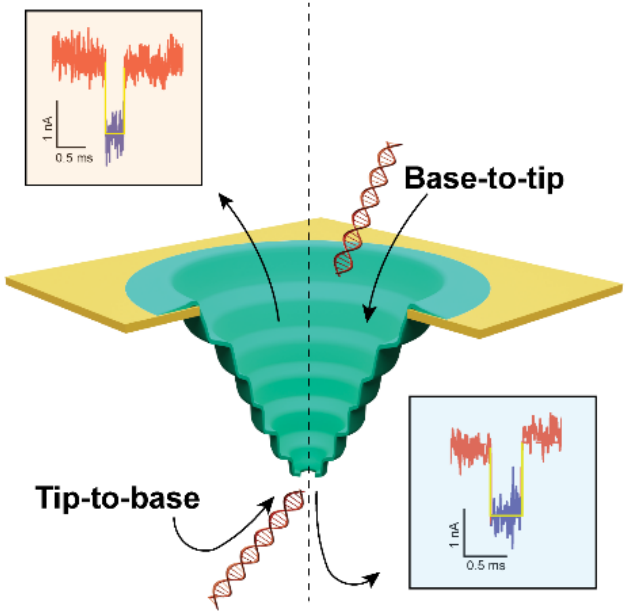
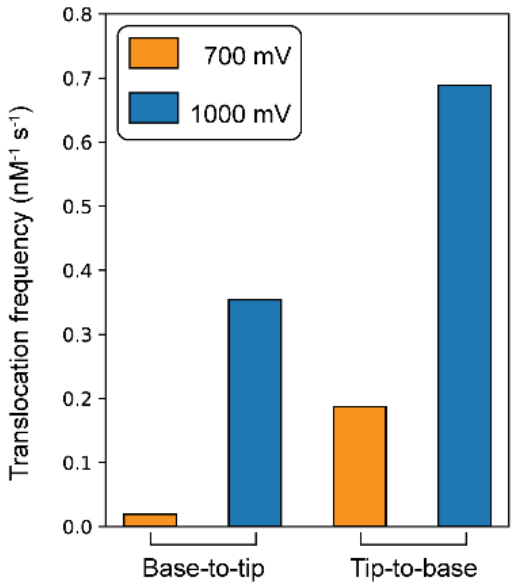
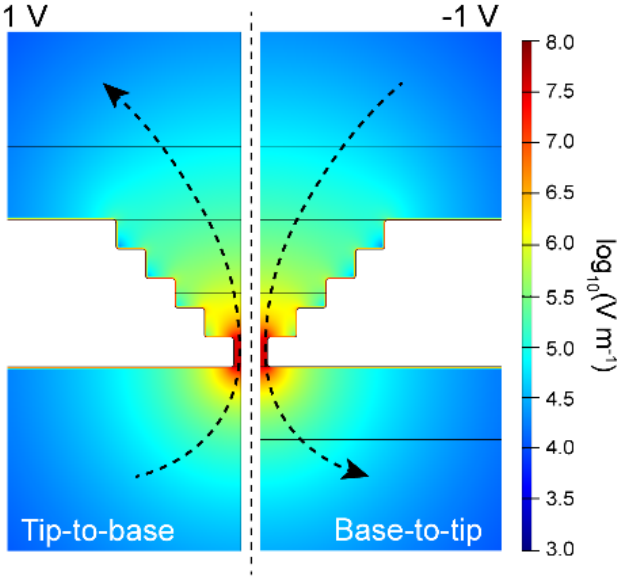
Biosensing experiment







- **Geometry Defines Two Direction: Tip-to-base / Base-to-tip.**
- **Translocation events : Tip-to-base is always more than Base-to-tip.**

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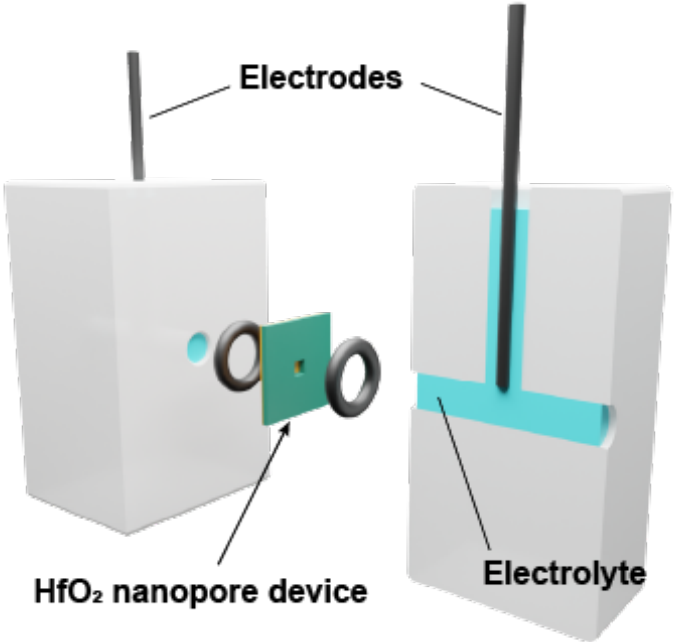
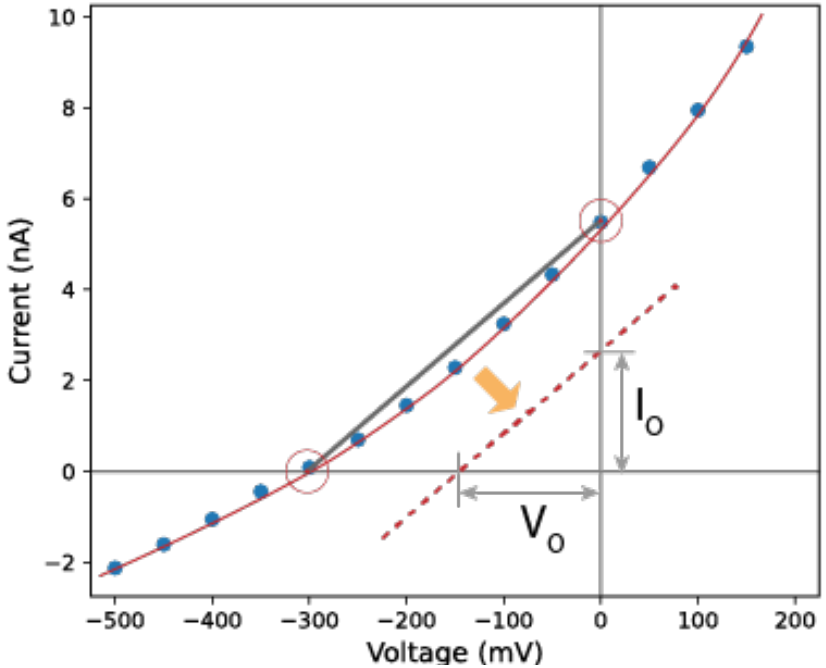
Osmotic Energy Conversion – Single Nanopore Devices

Set up and Basic Characteristics





$$\varepsilon_{osm} = \frac{k_B T}{ze} S \ln \frac{c_h}{c_l}$$

S: Ionic Selectivity

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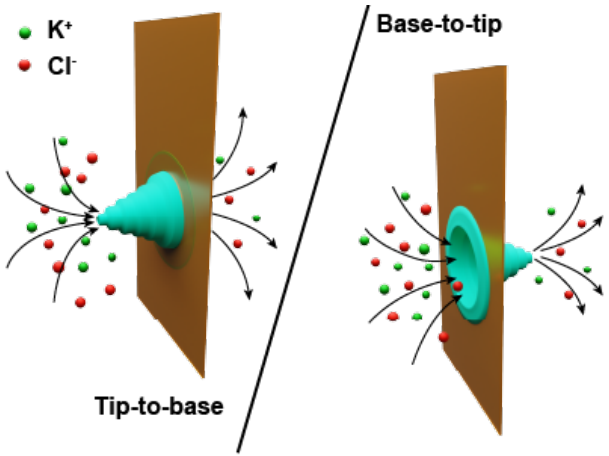
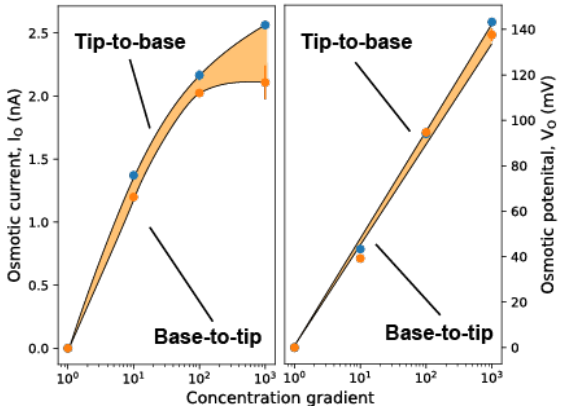
Osmotic Energy Conversion – Single Nanopore Devices

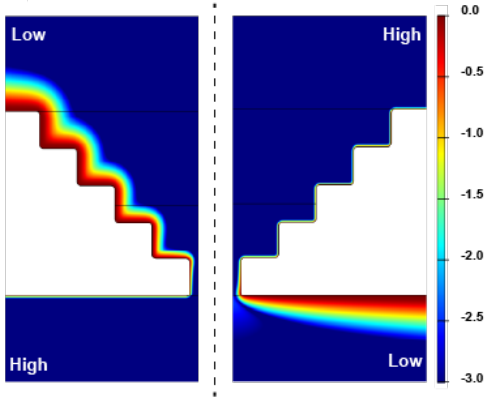
Asymmetric Diffusion from Asymmetric Geometry





Relative Distribution

log(Local distribution bias)



$$Local\ distribution\ bias = \left|\frac{c_{K^+} - c_{Cl^-}}{c_{K^+} + c_{Cl^-}}\right|$$

- Geometry Defines Two Direction: **Tip-to-base** / **Base-to-tip**.
- Osmotic Conversion Efficiency: **Tip-to-base** is always higher than **Base-to-tip.**
- Finite Element Simulation : Localized Relative Ions Distribution Difference

  Electric Double Layer expanded inside the nanochannel provide more selectivity (Left)

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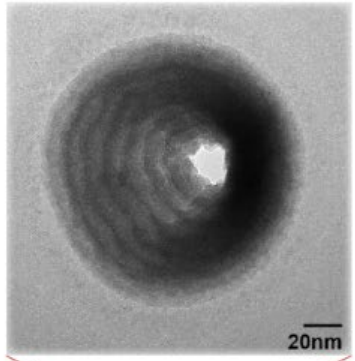
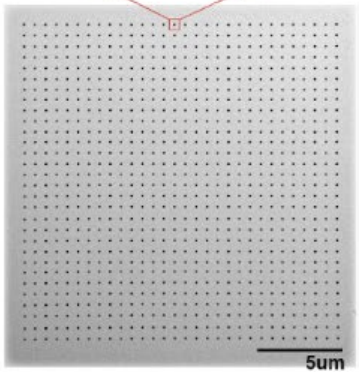
Our Result: ~0.35 nW / pore

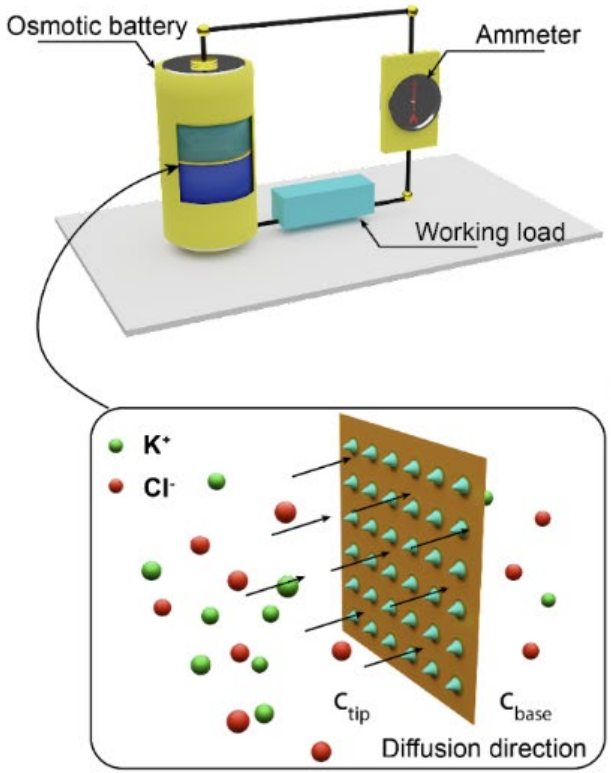
| MoS_2: ~1 nW / pore

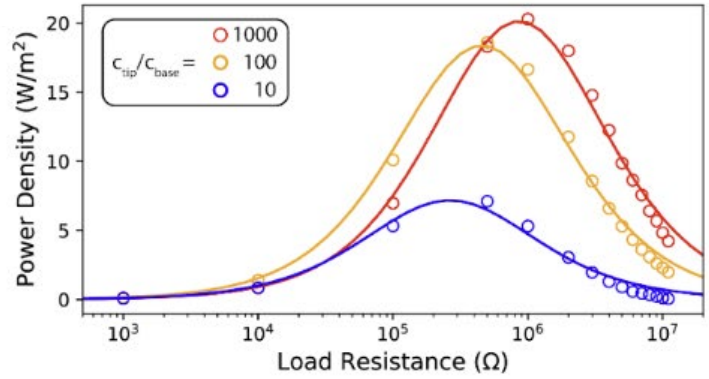
| hBN Nanotube: ~0.02 nW / Tube

Osmotic Energy Conversion – Array of Nanopores 900

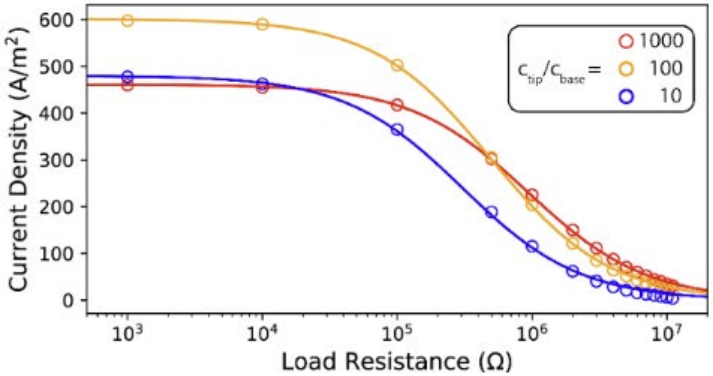








(d)



Nature-inspired stalactite nanopores for biosensing and energy harvesting Chernev *, Teng*, …….Radenovic* Advanced Materials **2023**

- PCT 23 170 779.5

Conclusion

New adaptable platform in terms of geometry and material composition – with inherent scalability, stability

Applications

basic nanofluidics research

biosensing -multi-omics

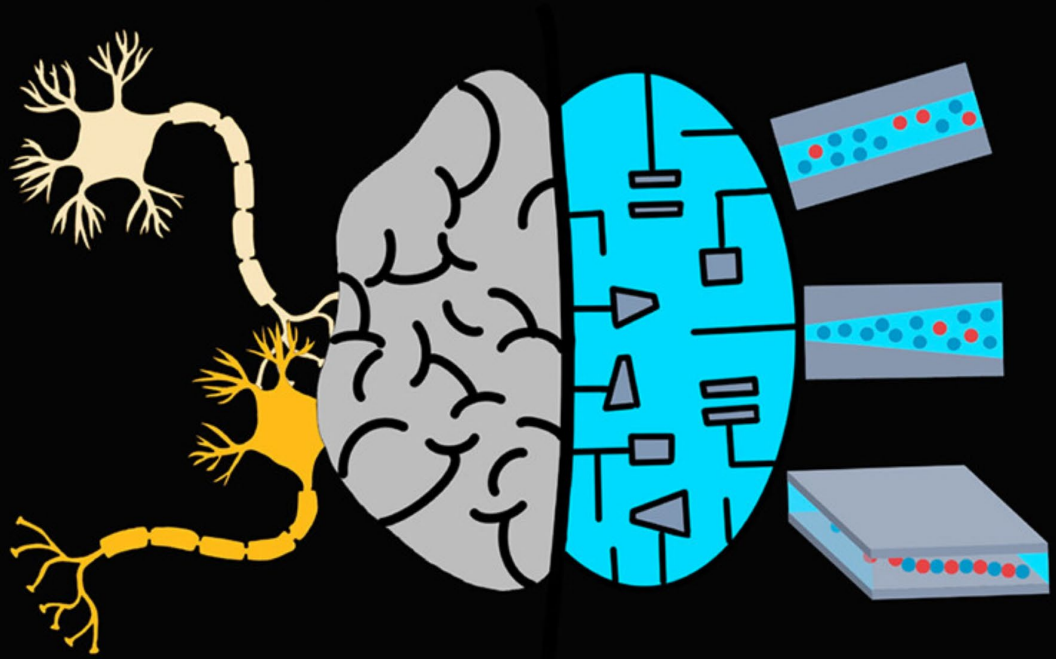
ionotronics

energy harvesting

filtration

Learning from the Brain: Bioinspired Nanofluidics

Brain-inspired nanofluidic iontronics



Advantages

- Multiple information carrier
- Low energy consumption
- In-memory computing
- Hardware-level plasticity
- Good biocompatibility
- …

Potentials

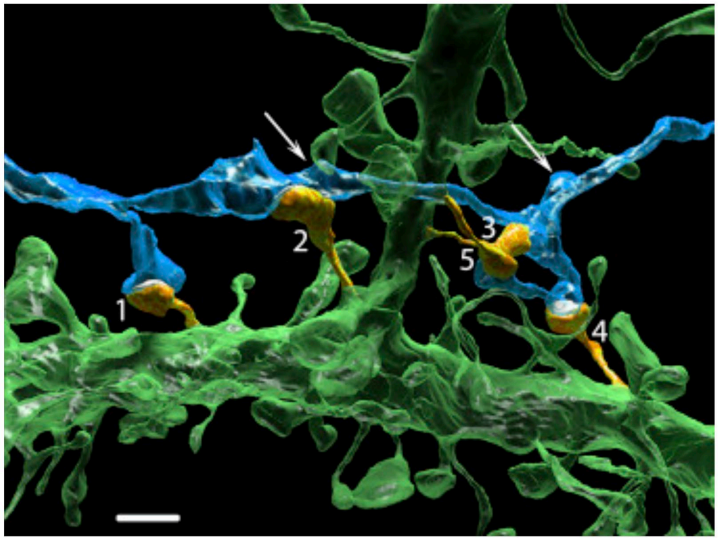
- Brain-like computing
- Brain-computer interface
- Neurological disease treatment
- …

Trends: ion channel-like → synapse-like → neural network-like → brain-like

Hou et al . J.Phys. Chem. Lett. 2023, 14, 11, 2891−2900

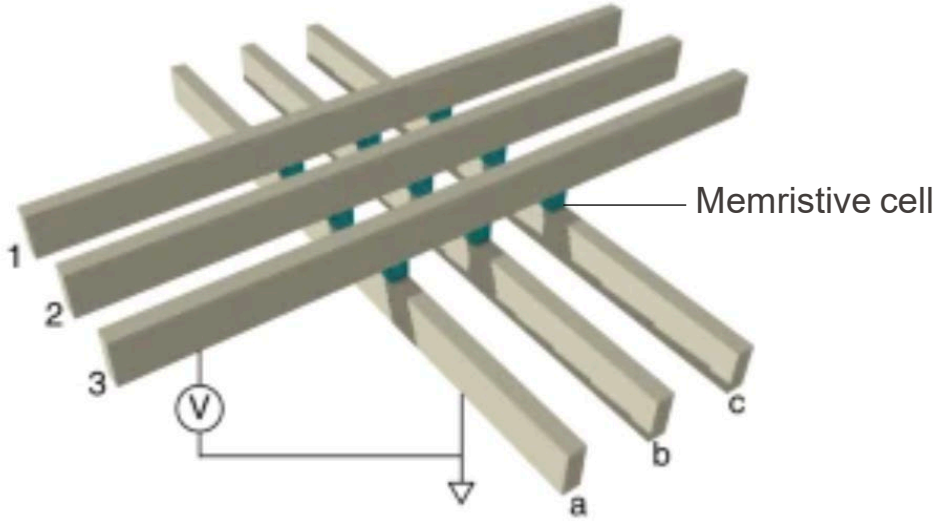
Ionotronics- towards artificial ionic computing

N. Kasthuri et al. Cell. 2015



Biological neural network
Ions as information carrier

Shuang Pi et al. Nature Nanotechnology. 2019



Artificial neural network (ANN)
electrons as information carrier

Need ionic ANN for accurate biomimicking

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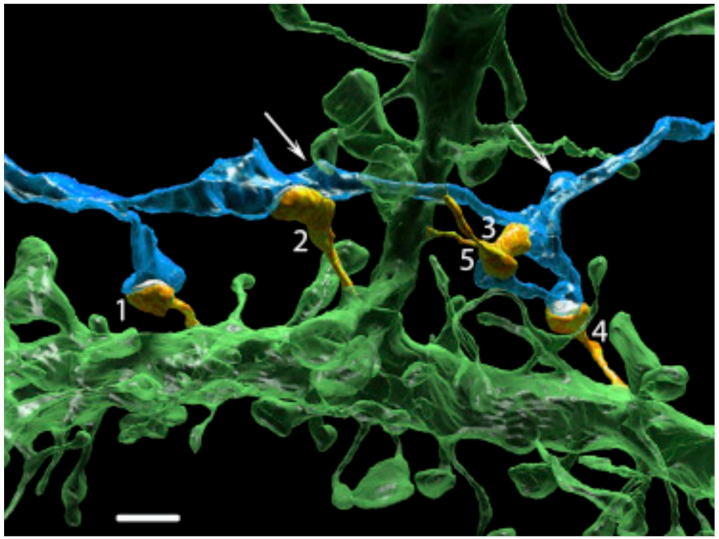
N. Kasthuri et al. Cell. 2015



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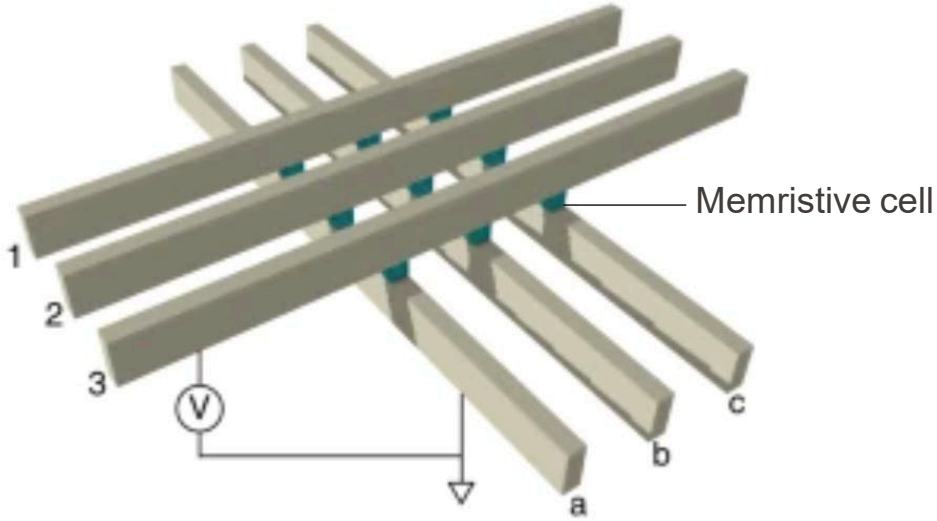

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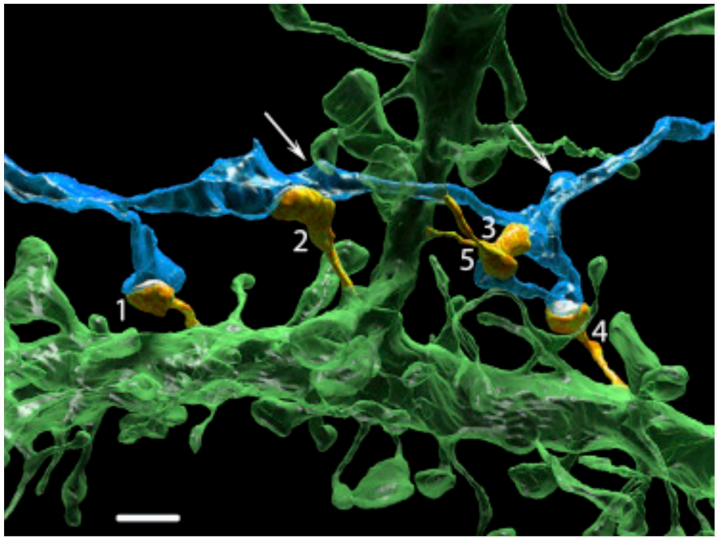

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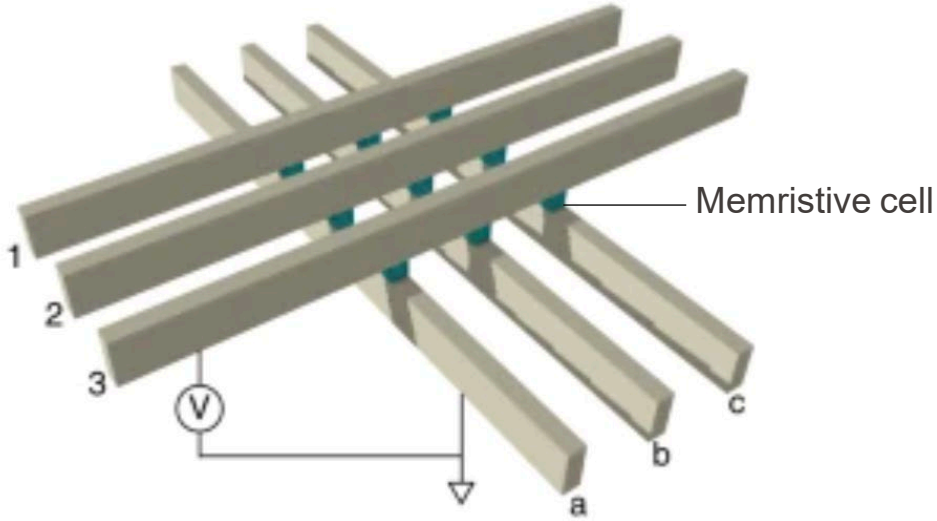


Artificial neural network (ANN)
electrons as information carrier

Need ionic ANN for accurate biomimicking

Roadmap: Neuro-inspired computing chips

Leon O. Chua concept of memristor

The idea of brain-like computing

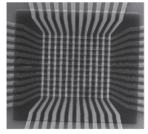
Hebbian Learn Rule

1948 1949 1971 2008 2010 2012 2014 2016 2018 2020 10+ years

Chip and system 5+ years

Device and techonology

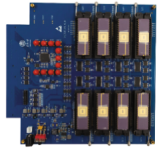
Memristor array (12×12, Analog)



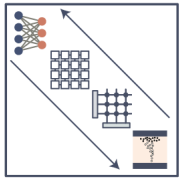
Memristor macro (1 Mb, Digital)

Fully memristor chip (160 kb, Analog)

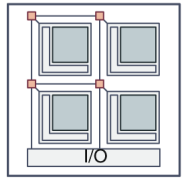
Fully memristor system (16 kb, Analog)



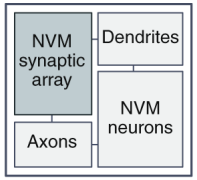
Codesign EDA tool chain



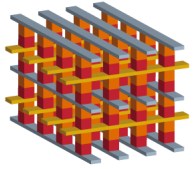
General-purpose NVM based chip



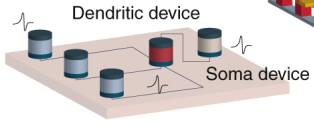
'Brain' chip



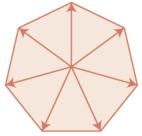
3D integration techonology



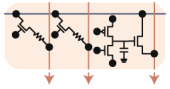
Neuromorphic devices and integration techonology



Optimized performance meet application requirements

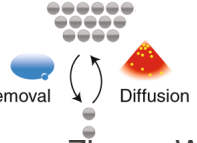


2T2R + 3T1C Unit cell

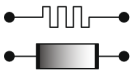


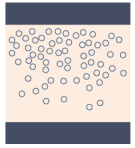
Diffusive memristor

Analogue memristor



Device linked to memristor

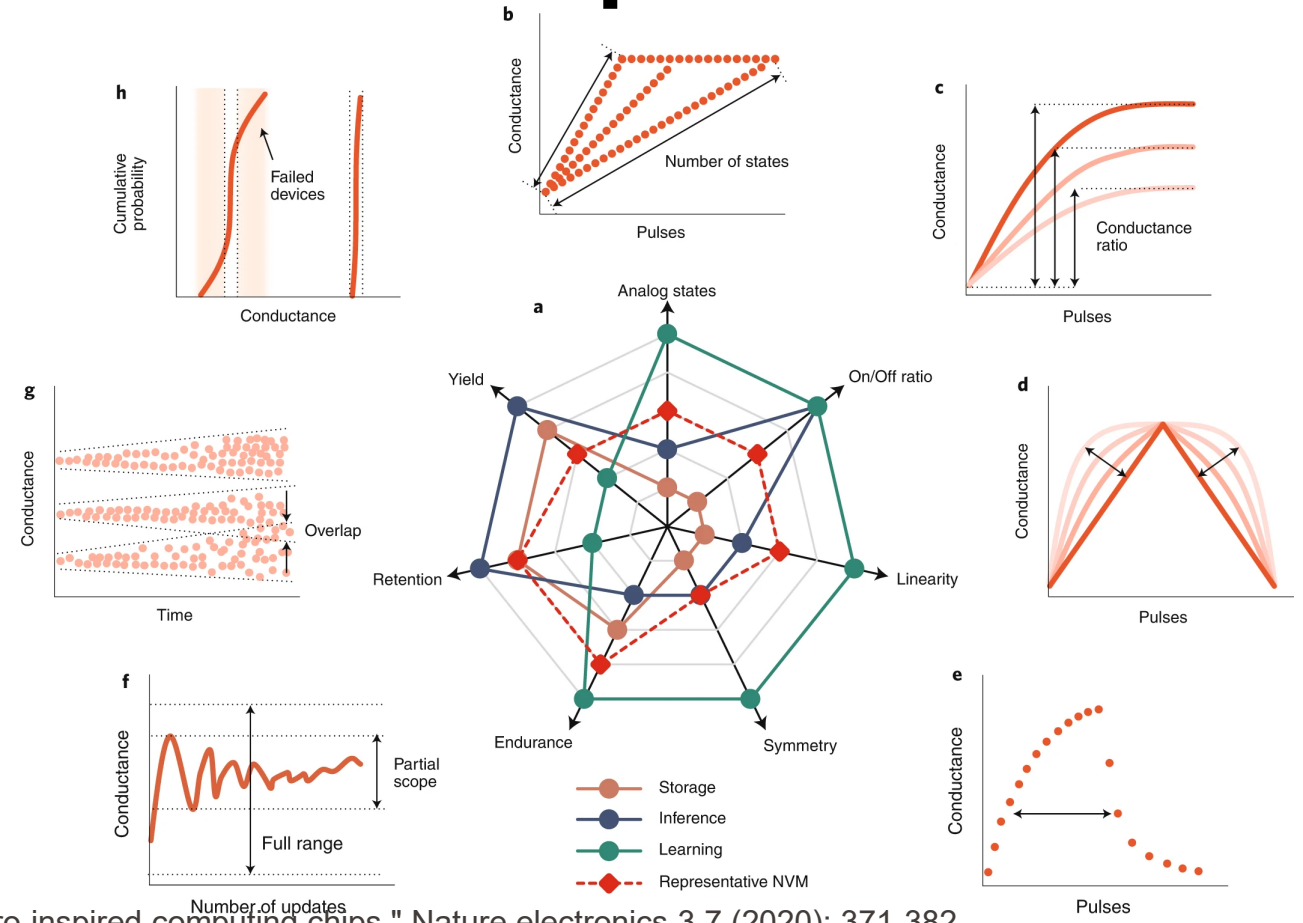




Zhang, Wenqiang, et al. "Neuro-inspired computing chips." Nature electronics 3.7 (2020): 371-382.

Metric for memristive material platforms

- Ion migration materials
- Phase change materials
- Conductive filament-based materials
- Magnetics
- Ferroelectrics
- 2D materials



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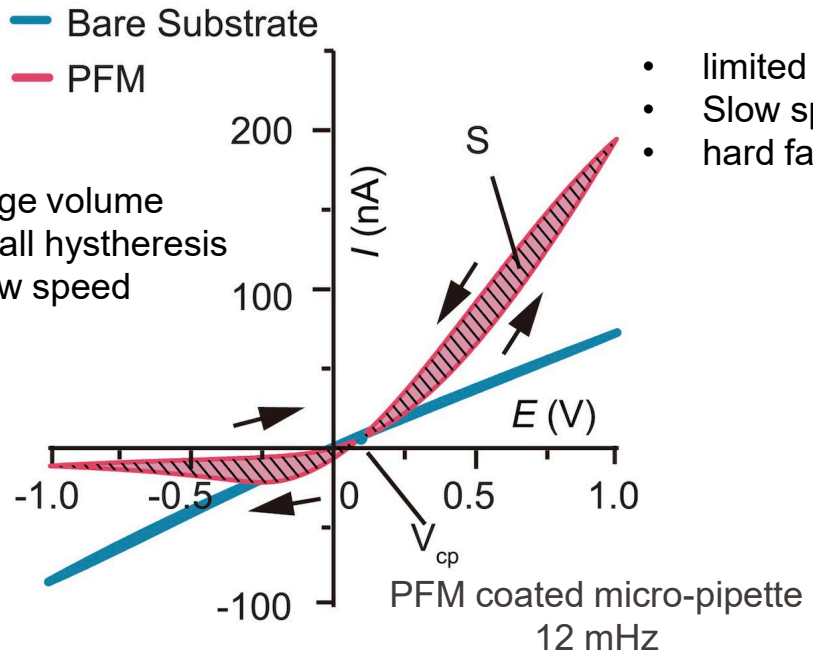
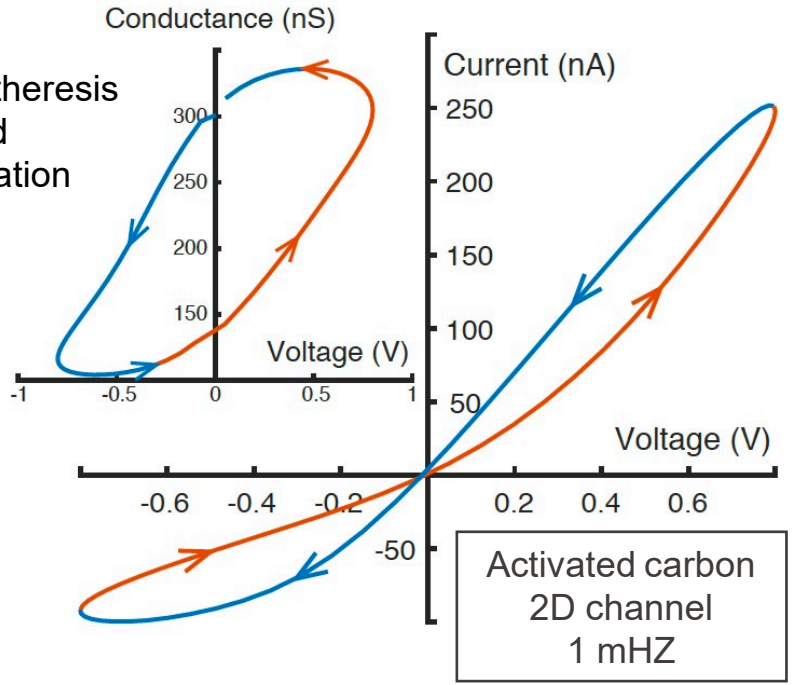
Limitations of fluidic memristors

- Large volume
- Small hystheresis
- Slow speed



T. Xiong *et al*. *Science*. 2023

- limited hystheresis
- Slow speed
- hard fabrication



P. Robin*,T. Emmerich*, A.Ismail* *et al*. *Science*. 2023

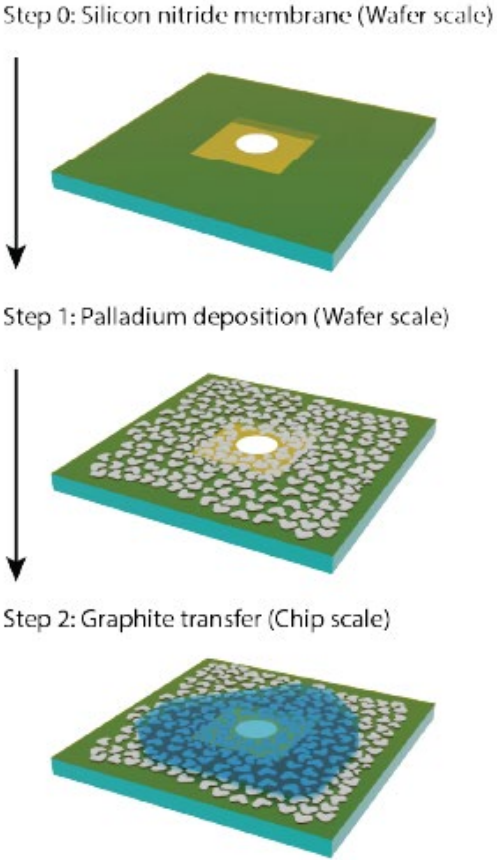
Need for a compact, performant, reliable, and scalable device to build ionic ANN and move forward iontronics

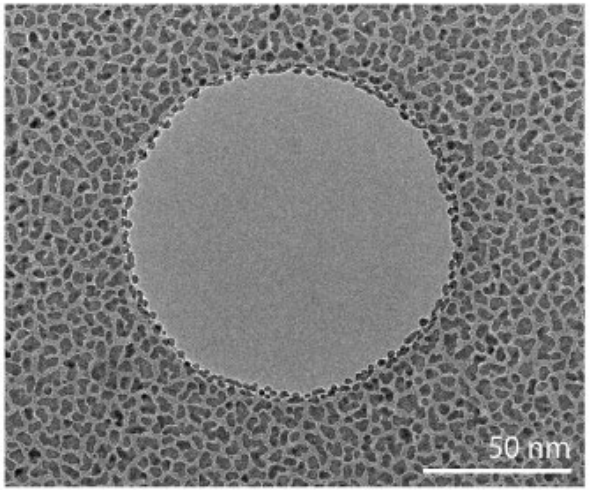
Logic: a new device for network scale iontronics-HACs[46]

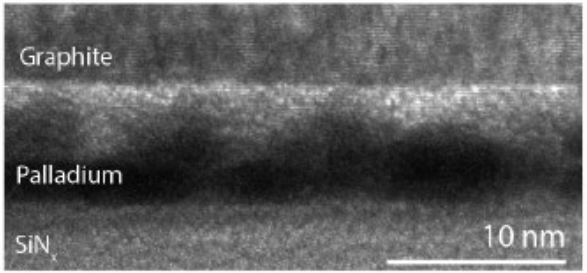
Fast and scalable fab:

10s of devices per week

Most of the steps at Wafer-scale production







- Ionic logic with highly asymmetric nanofluidic memristive switches Emmerich*, Teng*, Ronceray *, …….Radenovic* https://arxiv.org/abs/2306.07617

Logic: a new device for network scale iontronics-HACs[47]

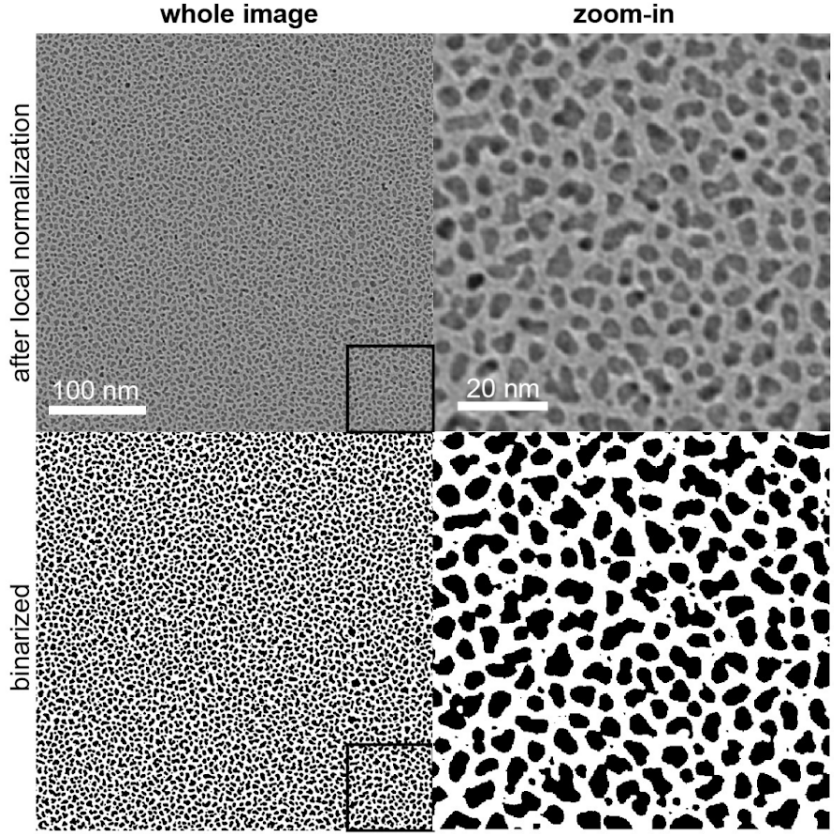
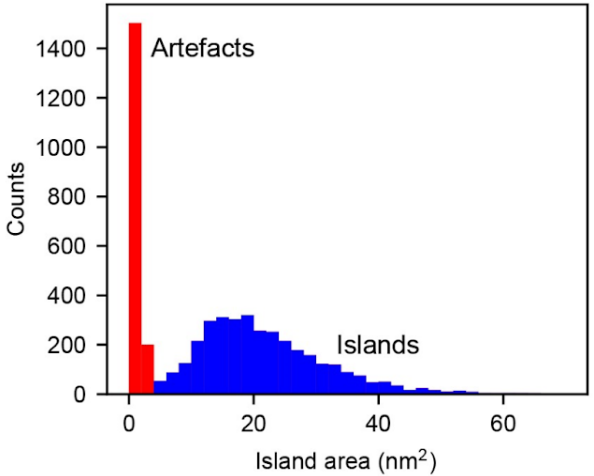


Image processing steps:
i) Local normalization (10 nm window)
ii) Gaussian blur (sigma =0.6 nm)
iii) Binarize (threshold: equidistant from peaks)

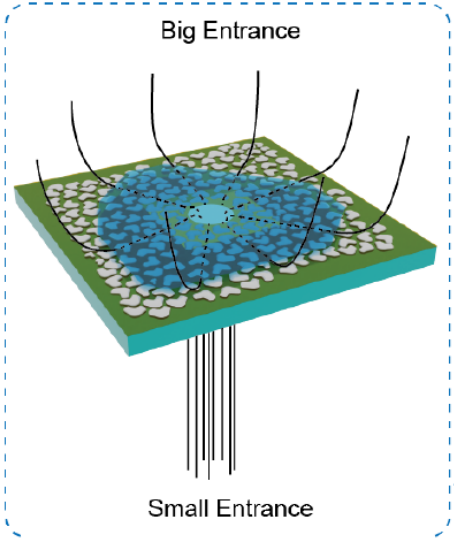


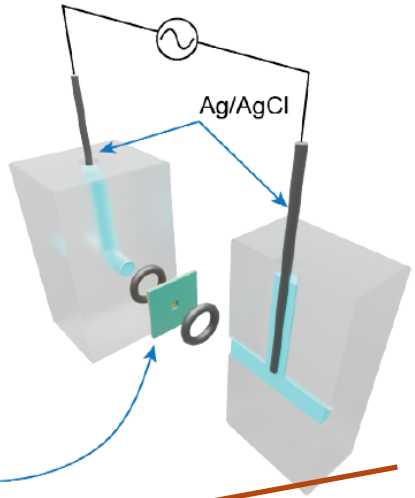
Island coverage 45%
Mean Island area 22nm

- Ionic logic with highly asymmetric nanofluidic memristive switches
  Emmerich*, Teng*, …….Radenovic* https://arxiv.org/abs/2306.07617

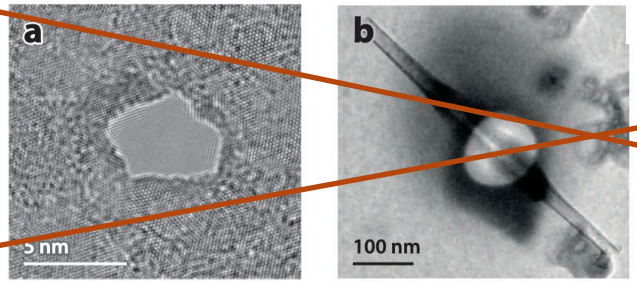
Logic: HACs represents a new nanofluidic geometry

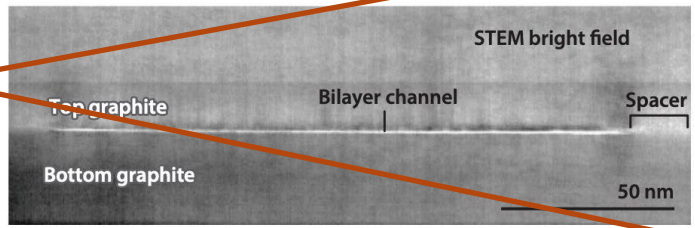
Combination of giant aspect ratio (100s fold) between both entrances and single-digit confinement in the presence of a charged material





HAC Nanofluidics





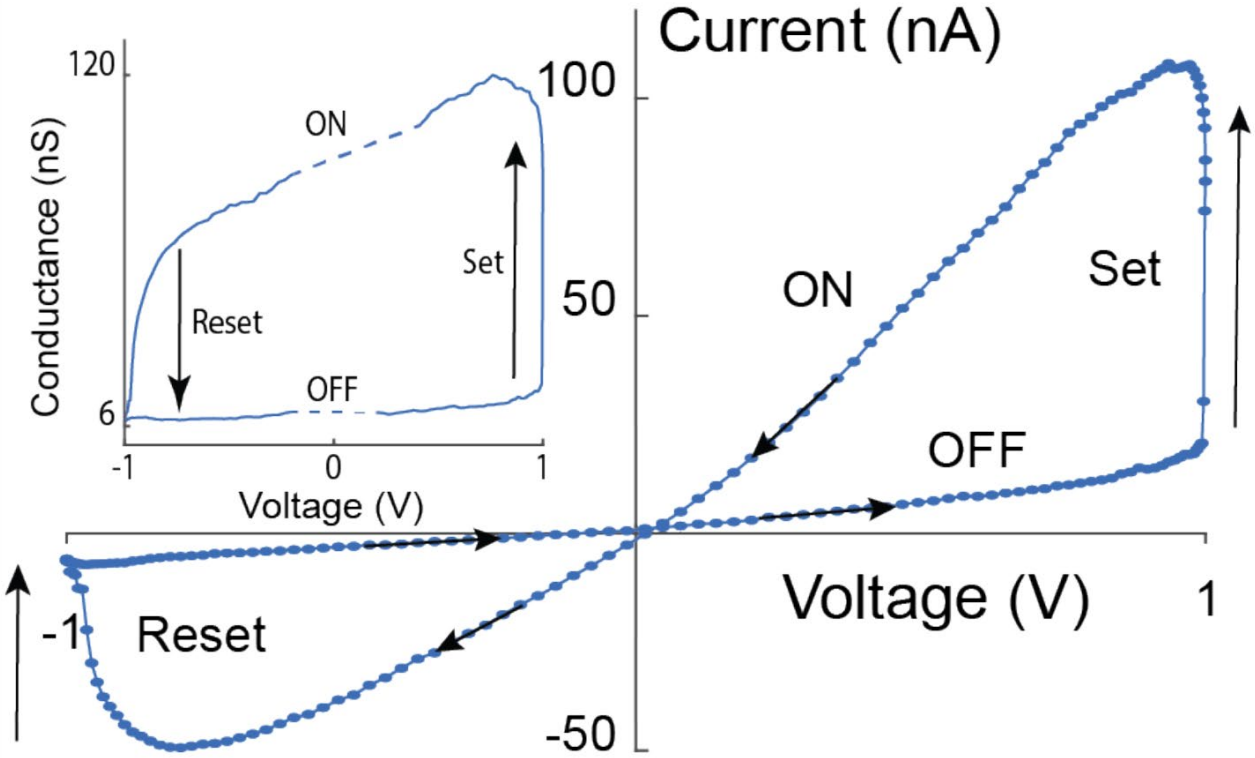
Ionic logic with highly asymmetric nanofluidic memristive switches
Emmerich*, Teng*, …….Radenovic* https://arxiv.org/abs/2306.07617

Logic: the memory effect in HACs

Performances 2 orders of magnitude better than previous report unique discontinuous behavior (threshold for abrupt switching)

1M KCl

50 mHz



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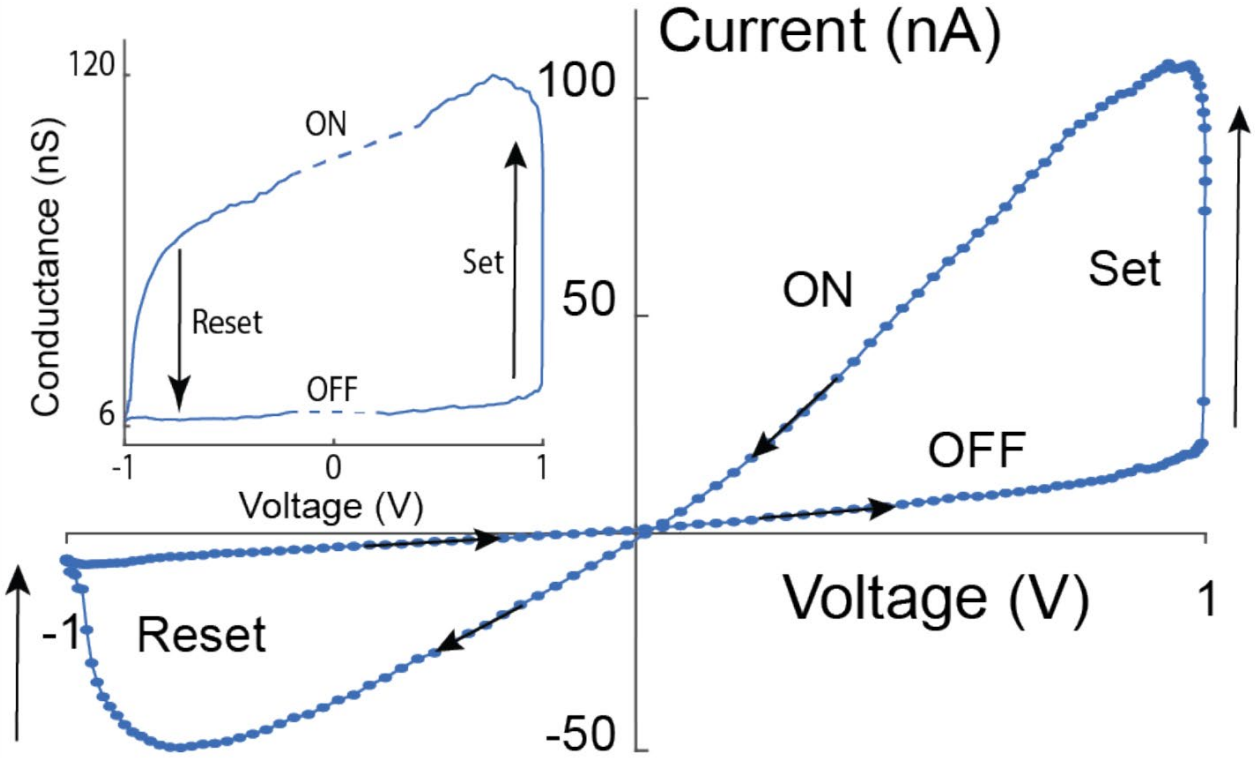

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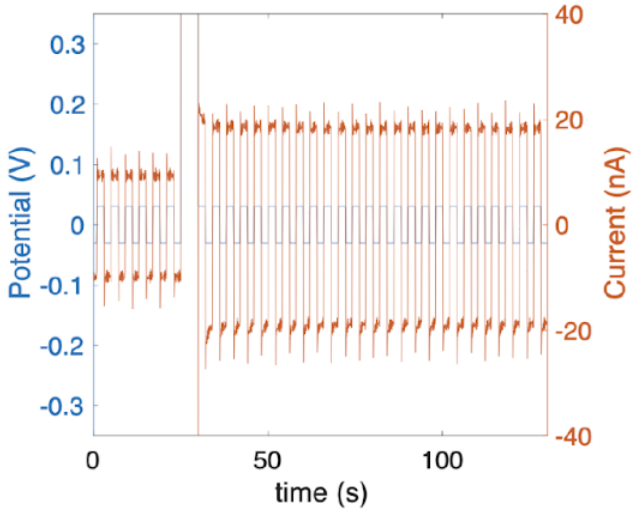
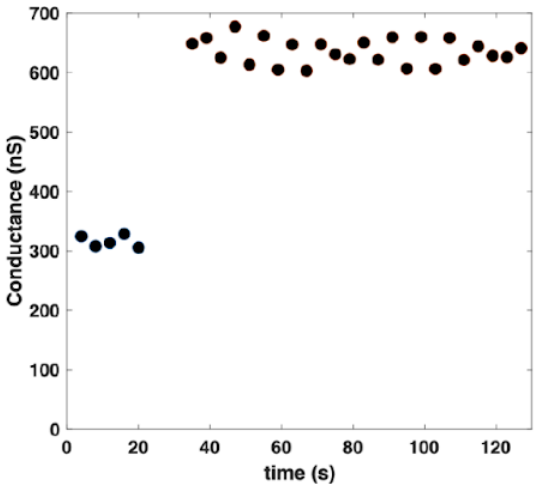
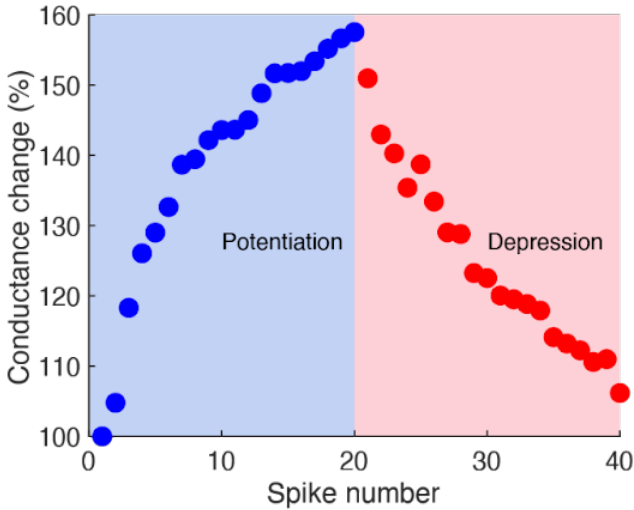
long term potentiation

- The device is set with a single positive voltage pulse of 1V
- The conductance is measured with voltage pulse train oscillating between plus and minus 30 mV..

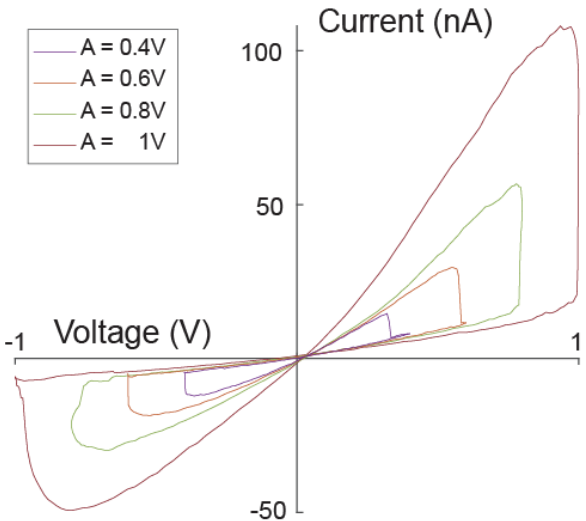


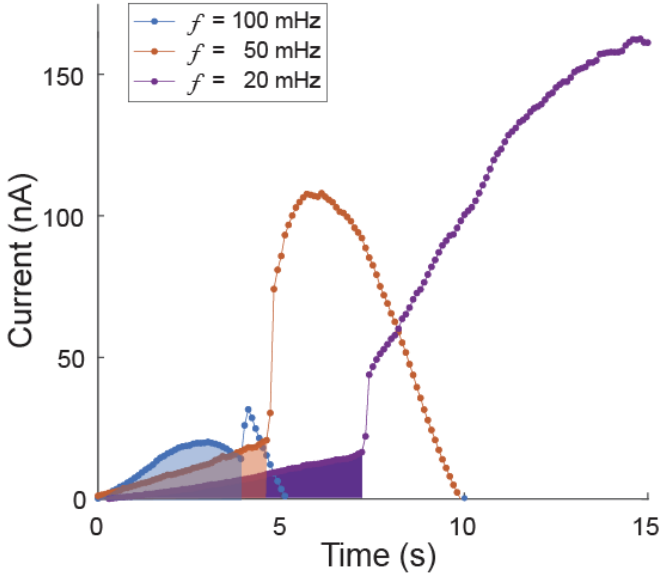


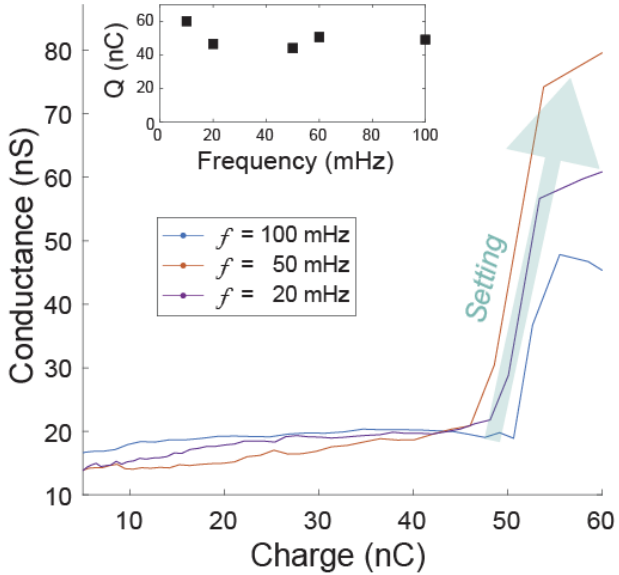


Threshold origin

Not a voltage threshold







When a given amount of ions has flown through the device switching occurs

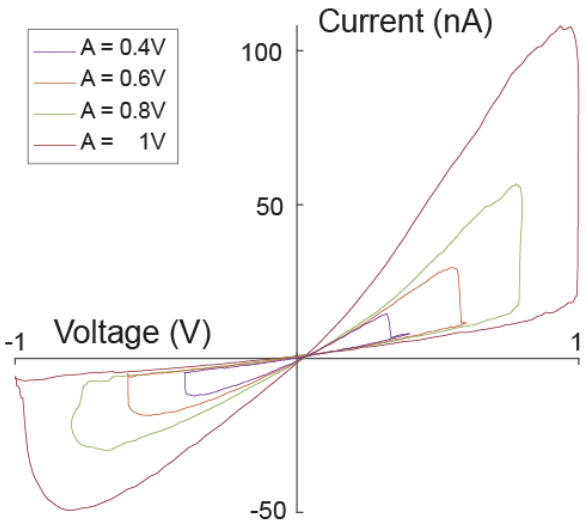
Charge threshold

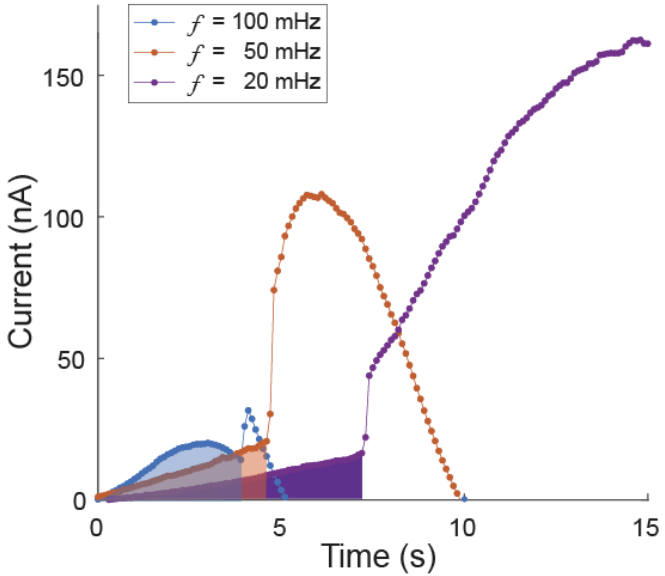
- Ionic logic with highly asymmetric nanofluidic memristive switches
  Emmerich*, Teng*, …….Radenovic* https://arxiv.org/abs/2306.07617

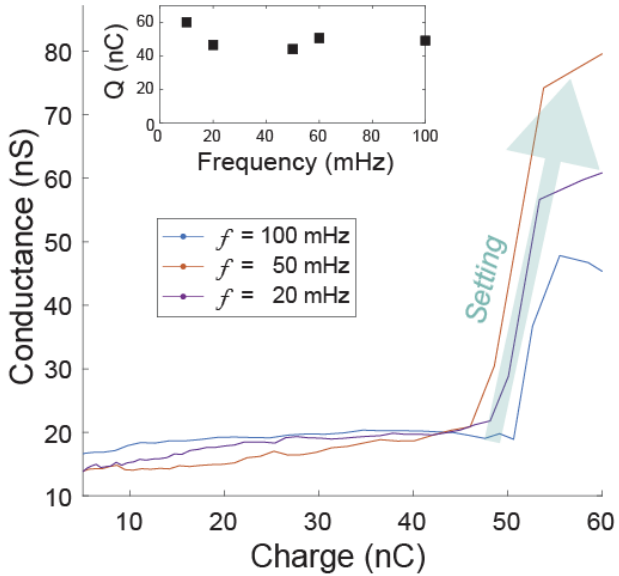
1M KCl

Threshold origin

Not a voltage threshold







When a given amount of ions has flown through the device switching occurs…..

Charge threshold

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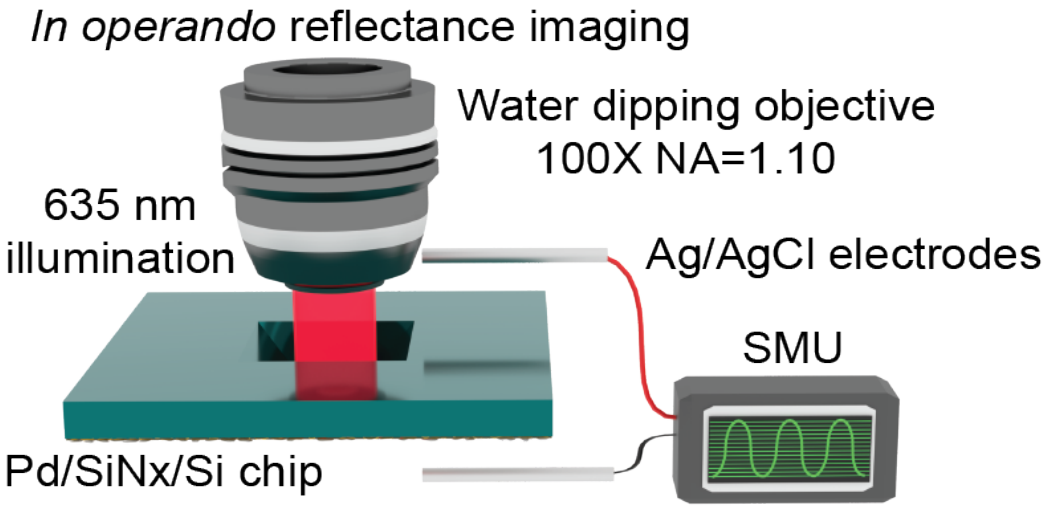
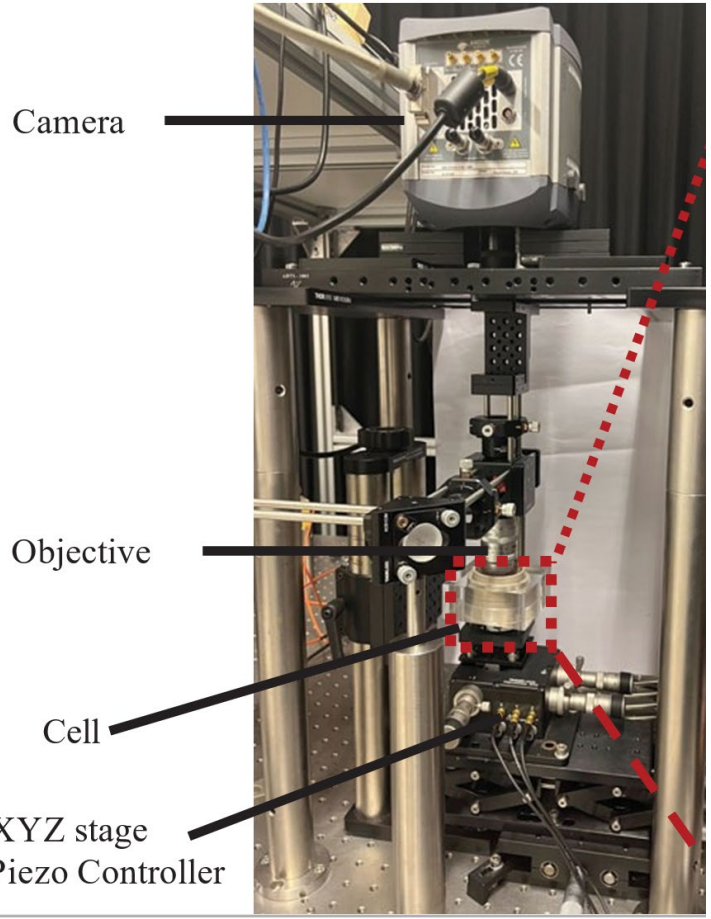
1M KCl

"You can **observe** a lot by **watching**."

- Yogi Berra

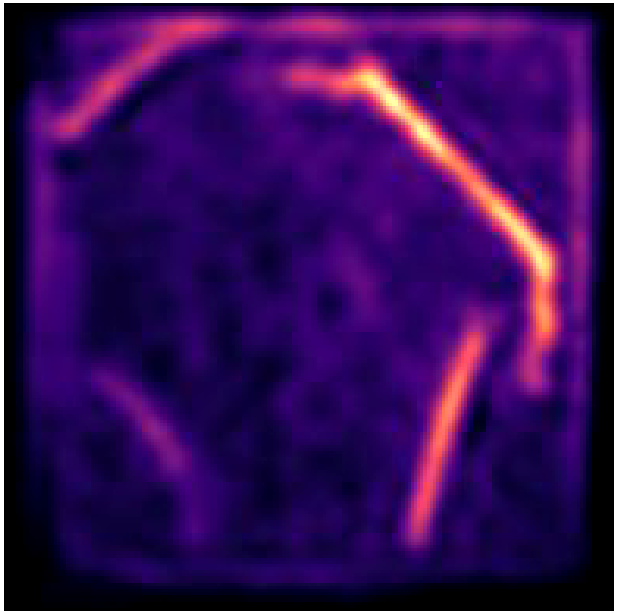
In-operando reflectance imaging

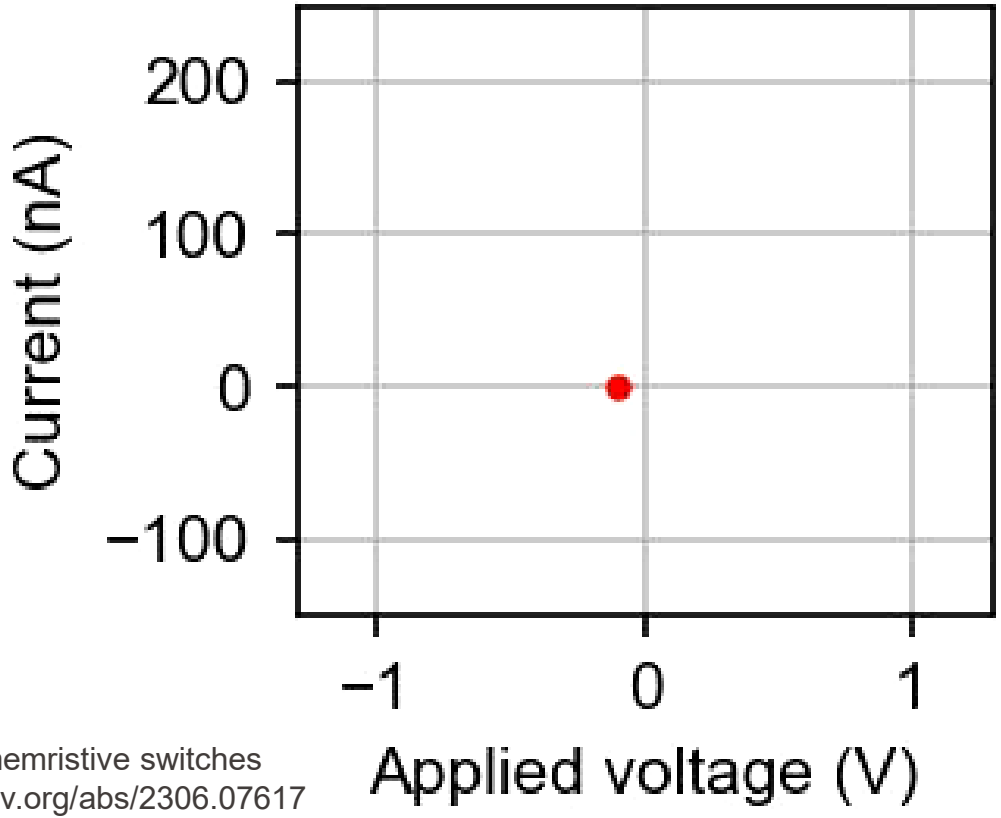




- Ionic logic with highly asymmetric nanofluidic memristive switches Emmerich*, Teng*, …….Radenovic* https://arxiv.org/abs/2306.07617

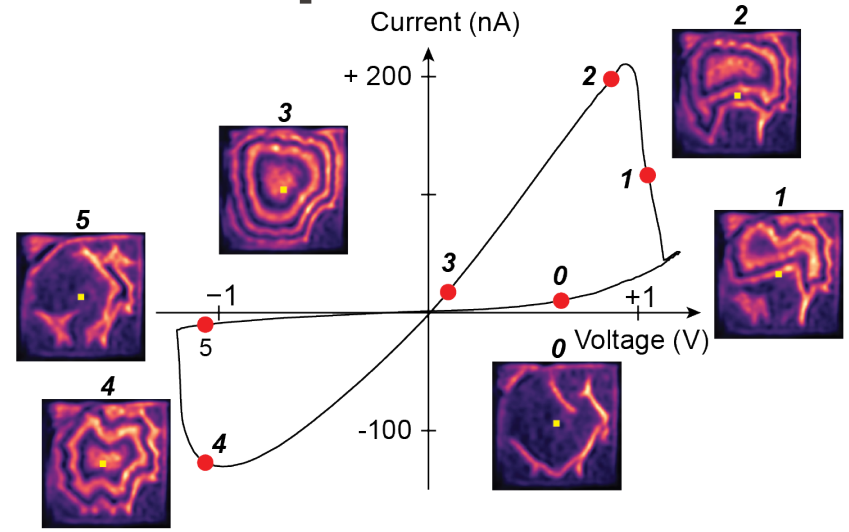
Watching devices while measuring Ionic transport

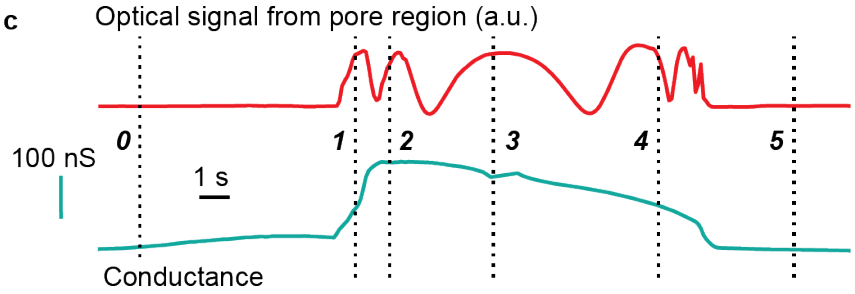


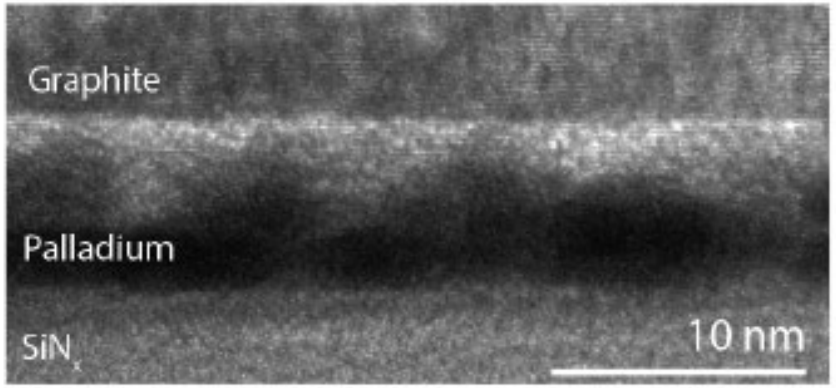


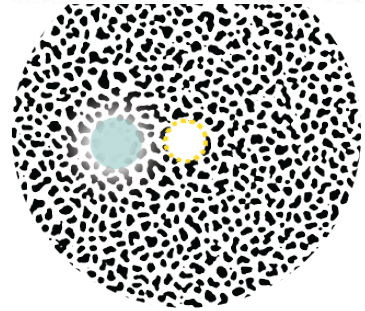
Ionic logic with highly asymmetric nanofluidic memristive switches
Emmerich*, Teng*, …….Radenovic* https://arxiv.org/abs/2306.07617

Proposed mechanism ionic transport in HACs

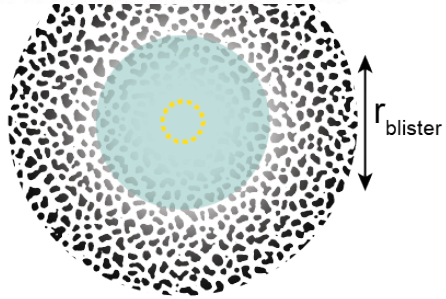








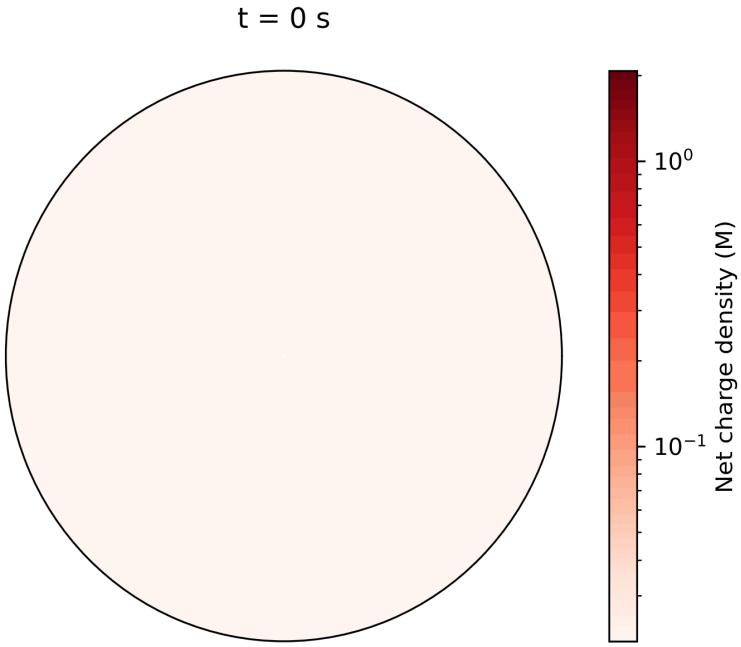
$R_{OFF} \sim \log(r_{ext}/r_{in})$



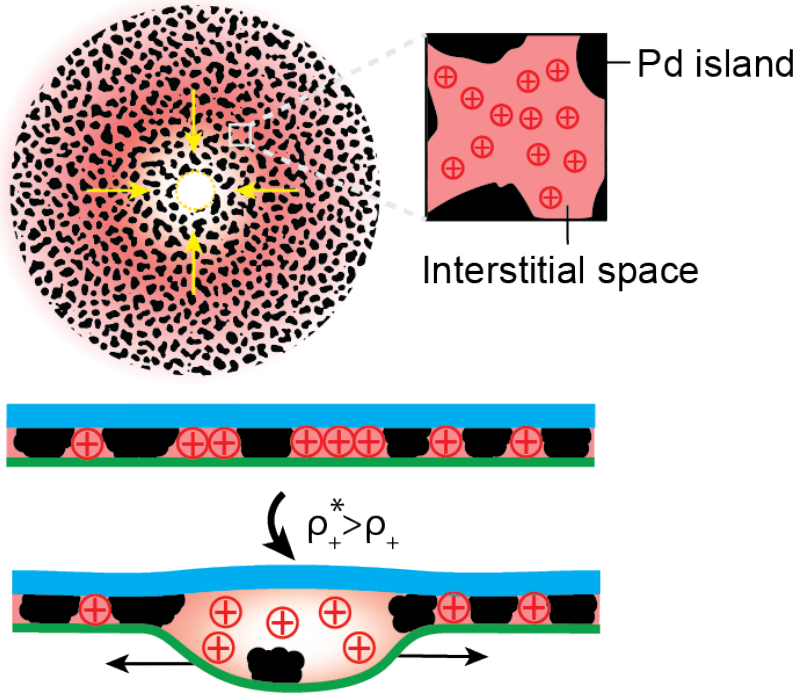
$R_{ON} \sim \log(r_{ext}/r_{blister})$

- Ionic logic with highly asymmetric nanofluidic memristive switches
  Emmerich*, Teng*, …….Radenovic* https://arxiv.org/abs/2306.07617

Proposed mechanism ionic transport in HACs



Charge focusing induces blister formation



Graphite 100 mC/cm²

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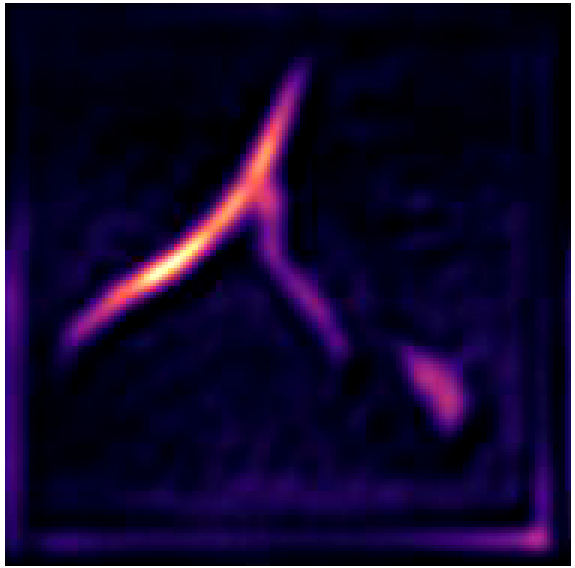
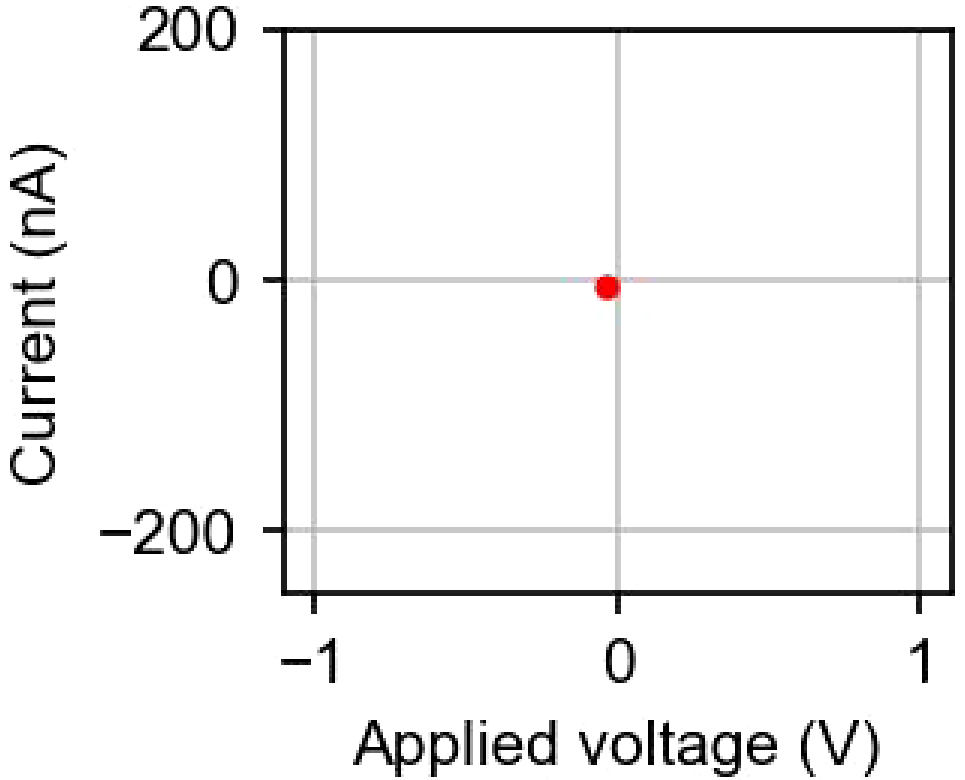
Mica control



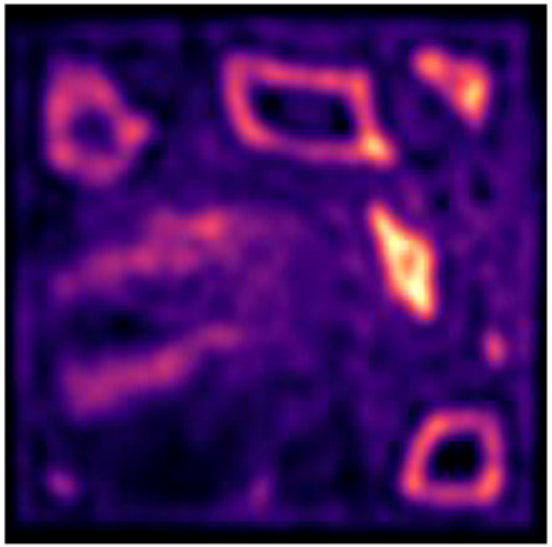


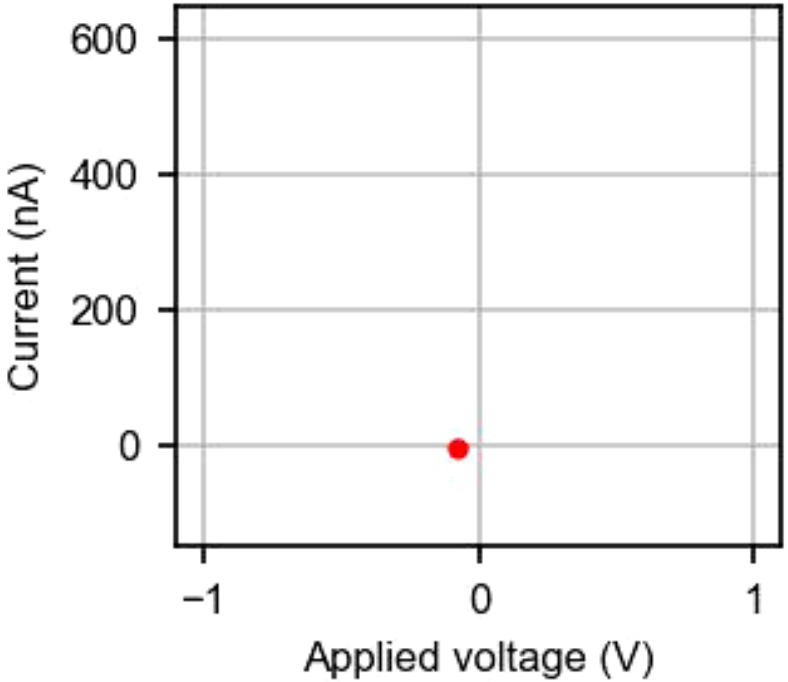
Endurance measurements

Voltage ramp from 0.2 to 1.2 V

300 mHz - Repeatability over 17 cycles

- blister bridges the pore to somewhere closer to the outer region





Endurance measurements

300 mHz - Repeatability over 17 cycles

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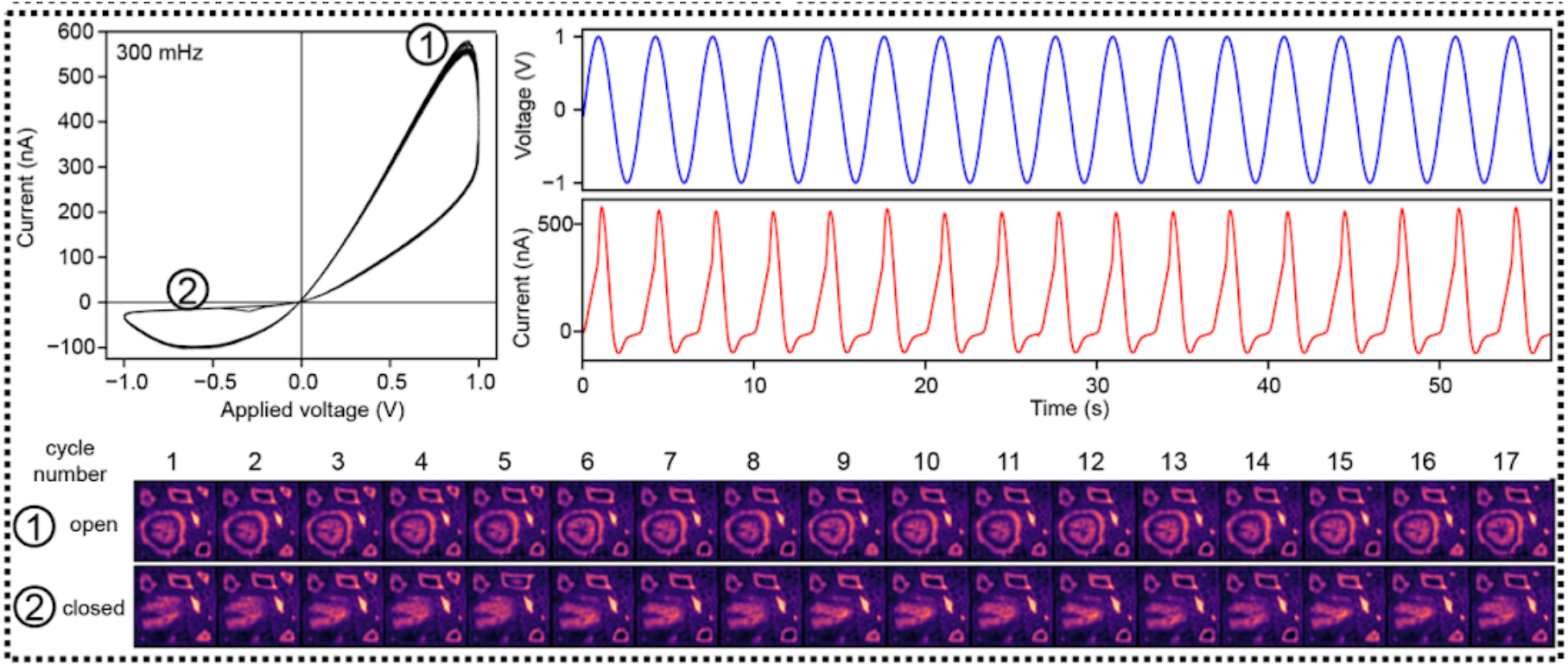
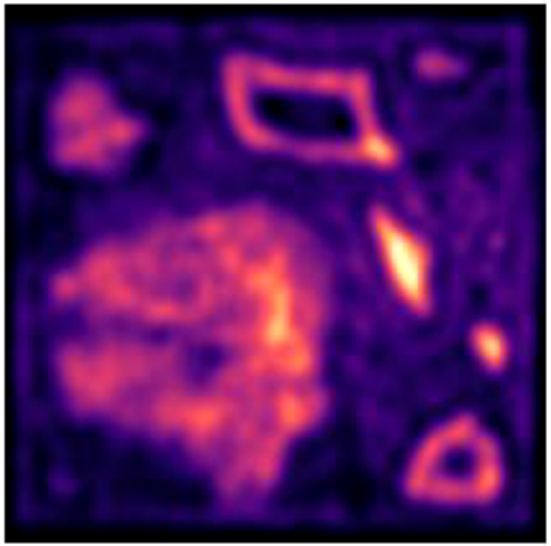
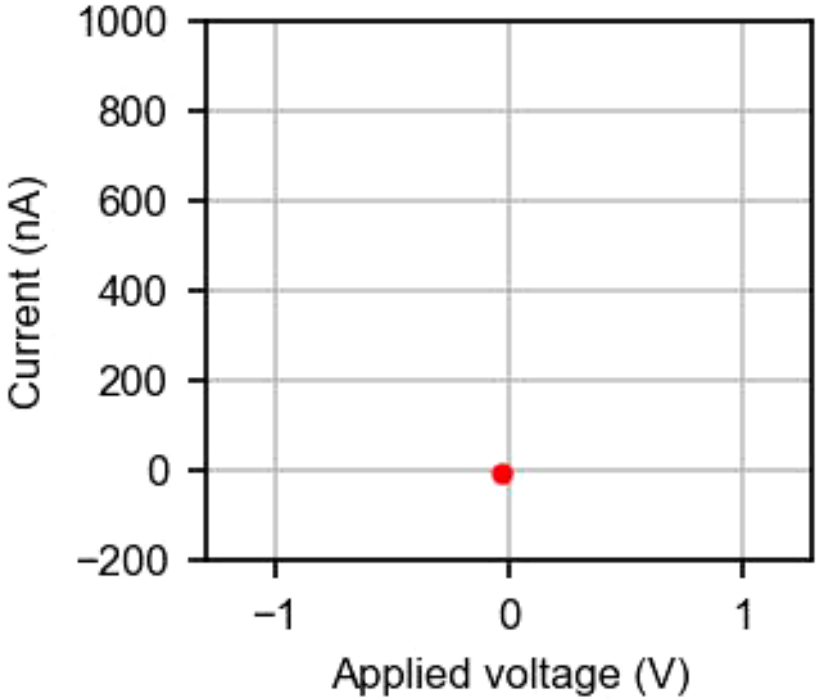


Endurance measurements

300 mHz –Voltage ramp from 0.2 to 1.2 V
blister bridges the pore to somewhere closer to the outer region





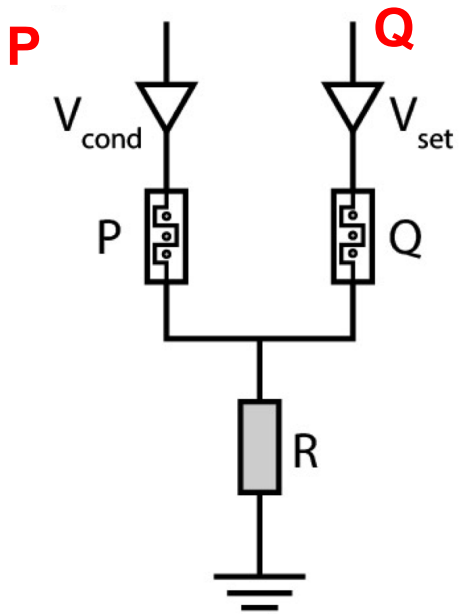
Material implication (IMP) logic

Vol 464|8 April 2010|doi:10.1038/nature08940 nature

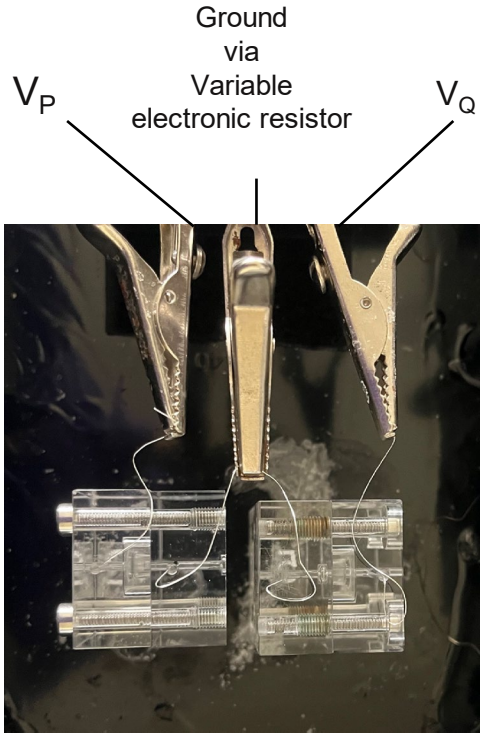
LETTERS

'Memristive' switches enable 'stateful' logic operations via material implication

Julien Borghetti[1], Gregory S. Snider[1], Philip J. Kuekes[1], J. Joshua Yang[1], Duncan R. Stewart[1]† & R. Stanley Williams[1]



First ionic computing experiment with channels communicating with each other the material implication (IMP) logic gate



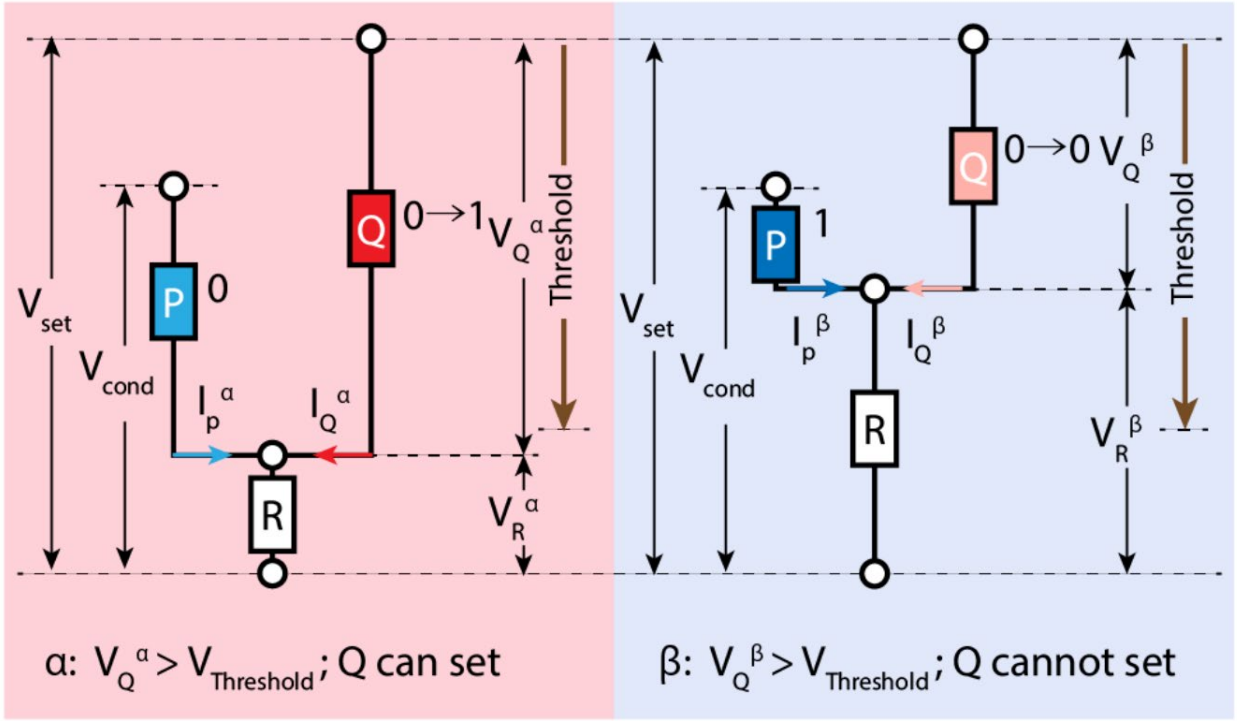
- Ionic logic with highly asymmetric nanofluidic memristive switches
  Emmerich*, Teng*, …….Radenovic* https://arxiv.org/abs/2306.07617

Logic: IMP gate

IMP gate can be implemented with two memristive switches

Set Q only if P is off

Case	P	Q	P′	Q′
α	0	0	0	1
β	1	0	1	0
γ	0	1	0	1
δ	1	1	1	1



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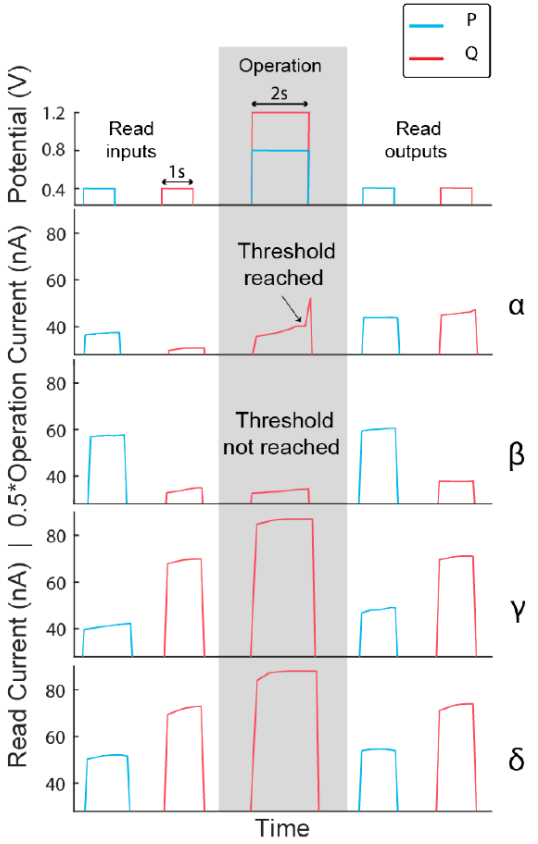
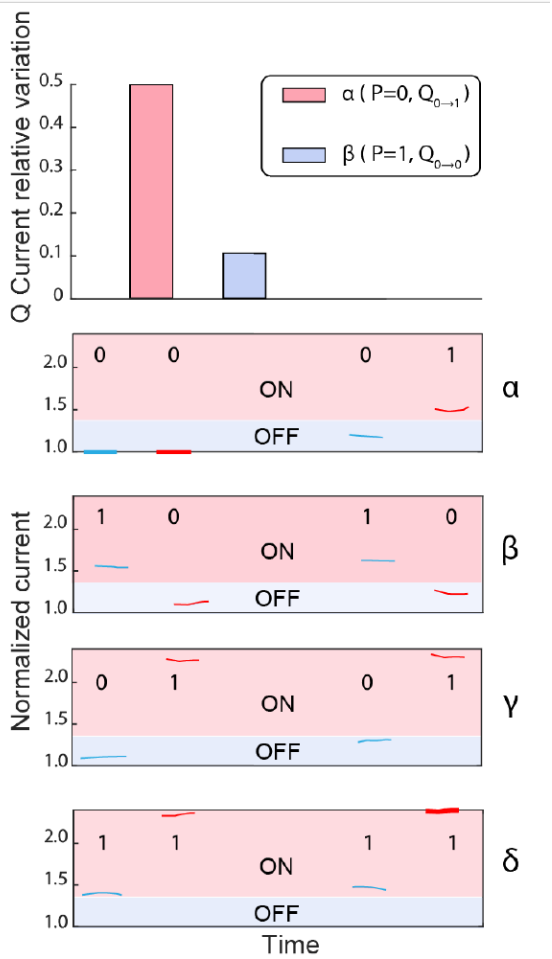
Logic: Results





1M KCl

IMP logic successfully implemented with two HACs

$$V_R^{\alpha} = R(I_P^{\alpha} + I_Q^{\alpha})$$

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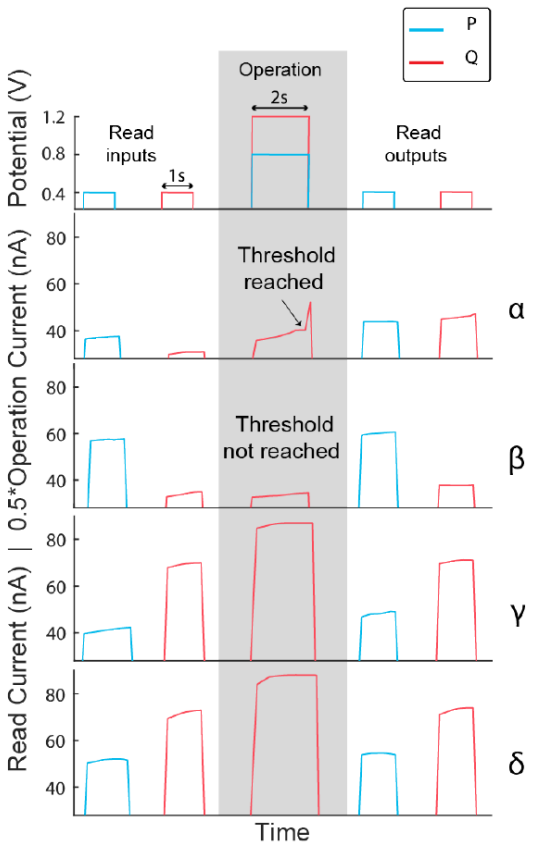
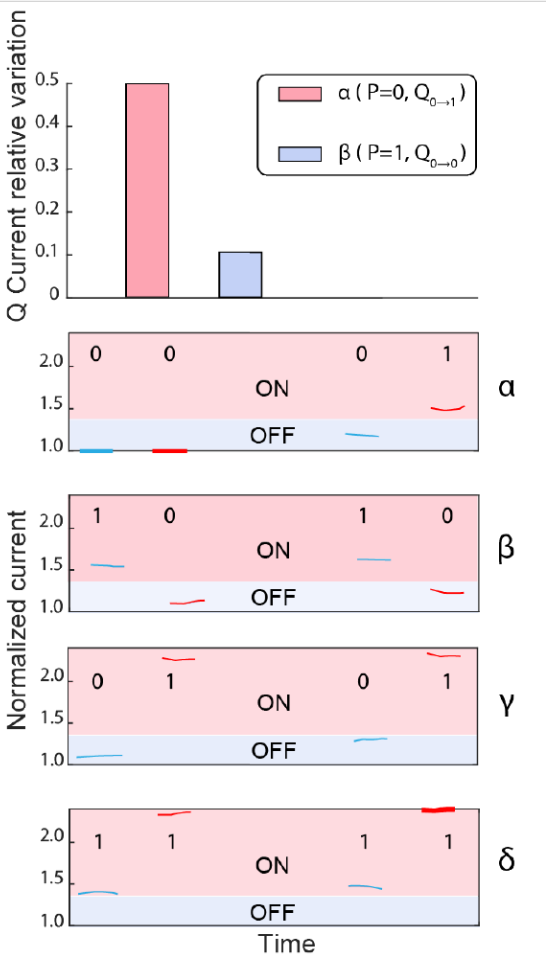
Logic: Results





1M KCl

IMP logic successfully implemented with two HACs

$$V_R^{\alpha} = R(I_P^{\alpha} + I_Q^{\alpha})$$

$$R_{set} < R < R_{cond}$$

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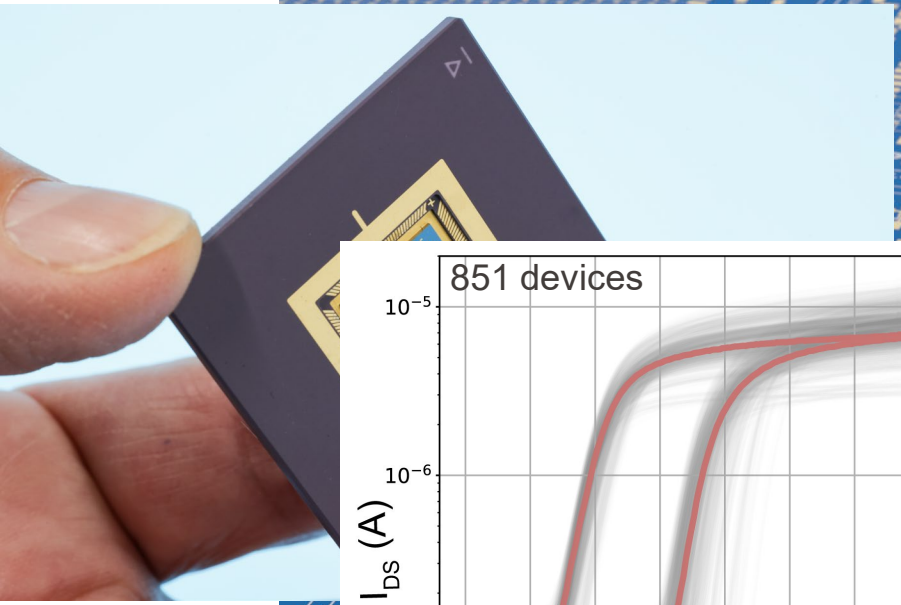

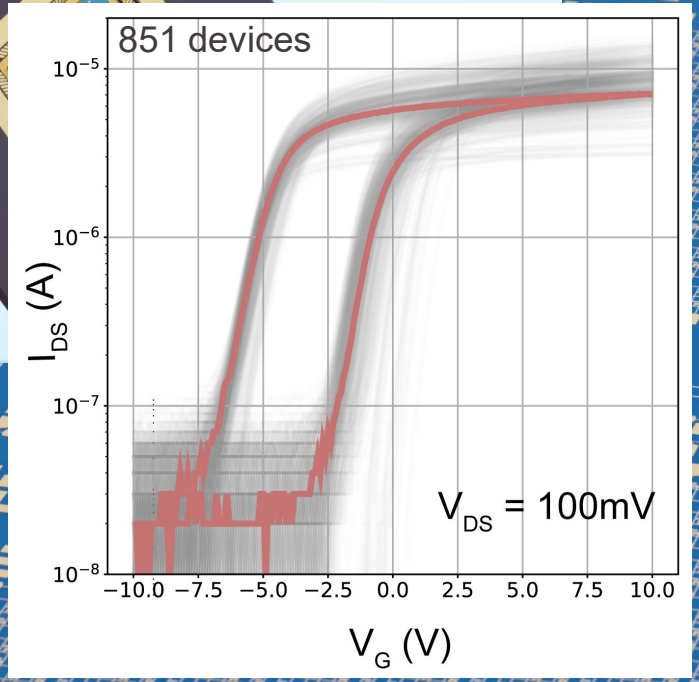

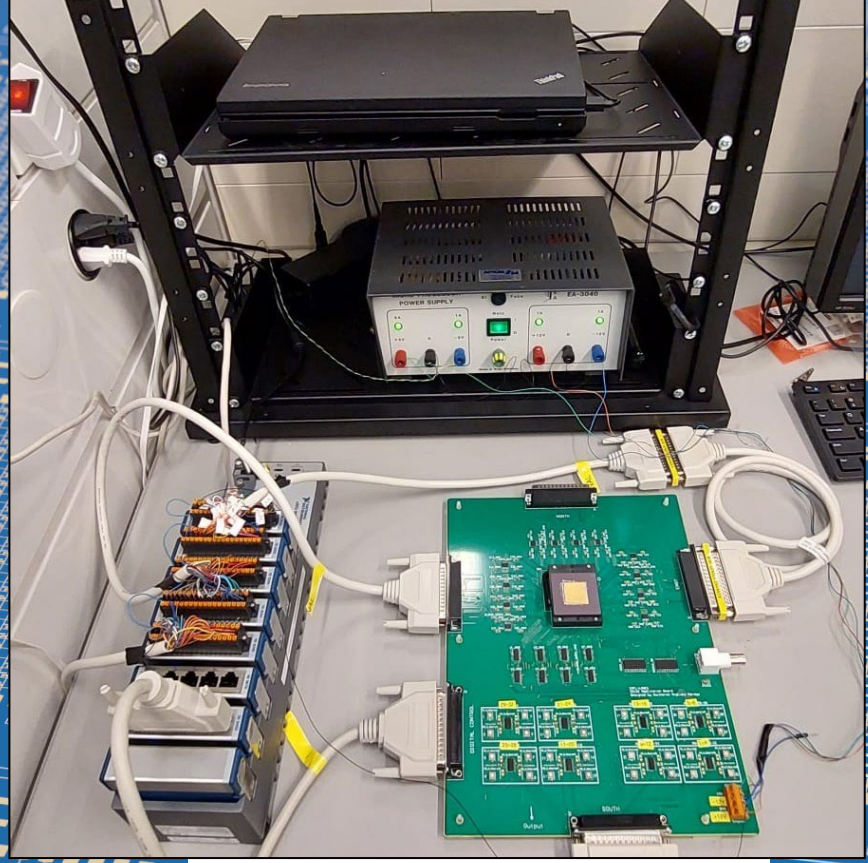

Marega…Kis;
arXiv:2303.07183

Working conclusions

- HACs exhibit excellent scalability, speed, and conductance ratio
- Direct imaging of the mechano-ionic memory mechanism with in-operando optical observation –will guide the device optimization
- Logic circuits with two HACs working simultaneously have been successfully assembled
- Future efforts should focus on connecting HACs with water channels to create fully liquid circuits.

Ionic logic with highly asymmetric nanofluidic memristive switches
Emmerich*, Teng*, …….Radenovic* https://arxiv.org/abs/2306.07617