

Advanced Topics in Micro- and Nanomanufacturing

MASSIMO MASTRANGELI, PHD

DEPARTMENT OF MICROELECTRONICS
DELFT UNIVERSITY OF TECHNOLOGY

DELFT, THE NETHERLANDS

M.MASTRANGELI@TUDELFT.NL

Part 4:

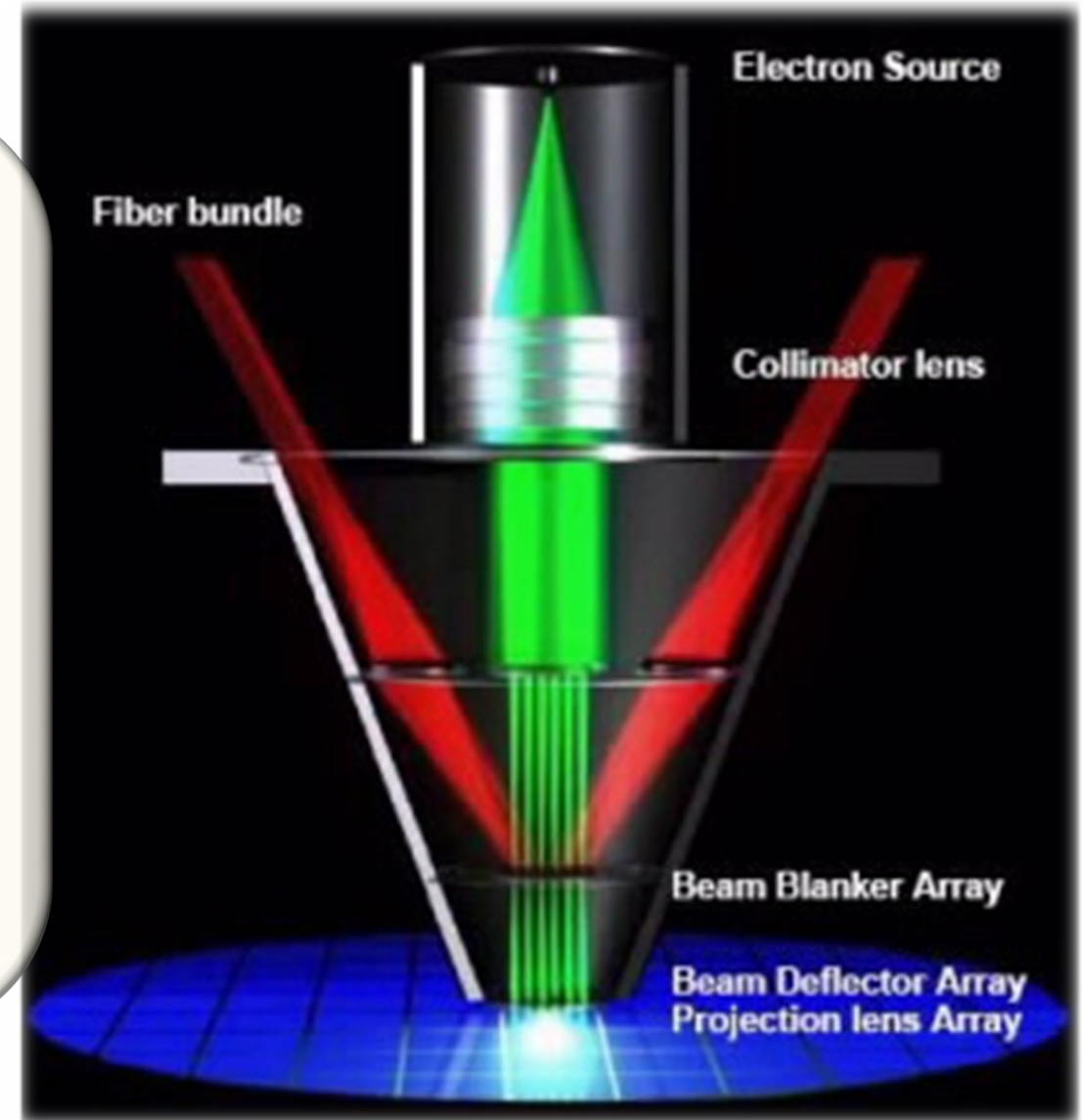
Single nanoparticle printing

TOP-DOWN MEETS BOTTOM-UP
ELECTRO-TOPOGRAPHICAL TRAPPING
COFFEE RING EFFECT
TOPOGRAPHICALLY-TEMPLATED FLOW COATING
APPLICATION TO MICRO/NANOSYSTEM ASSEMBLY

Beyond top-down micro/nanofabrication

Lithography

- ✓ Ultimate resolution
- ✓ Wafer-level
- ✗ Pre-formed layers have low crystalline quality
- ✗ Integration of high-quality materials limited



Source: Burn Lin

From nanoparticles to nanodevices

Unique properties of NPs

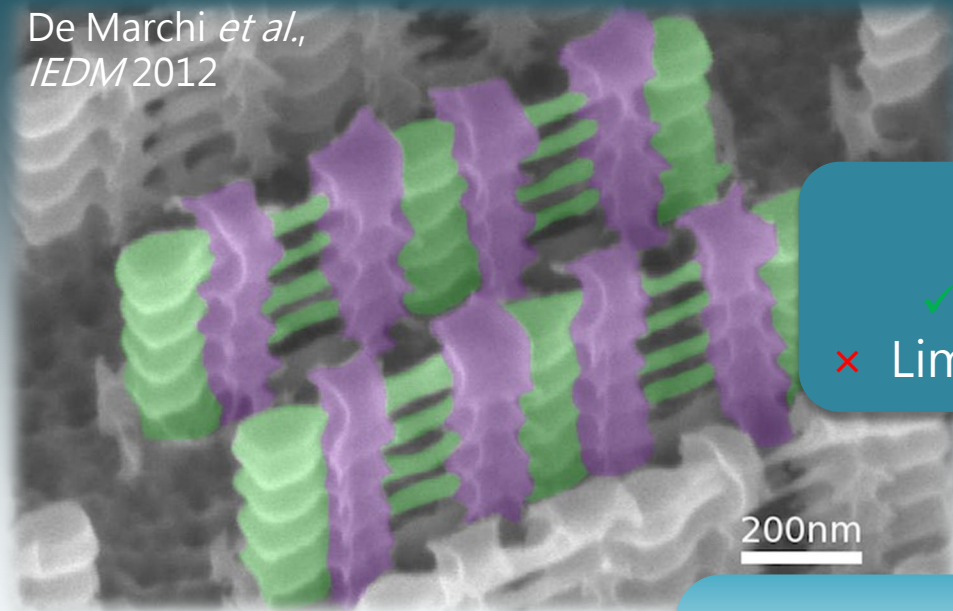
- Metal, oxide, core/shell
- Carbon-based
 - Graphene
 - Nanotubes
 - Nanodiamonds
- Quantum dots
- Nanowires



How to build
devices out of
nanoparticles?

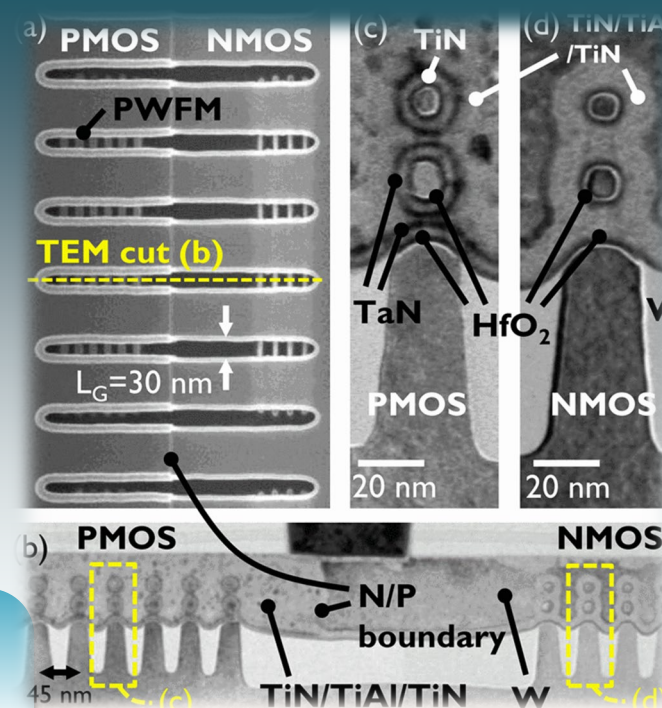
NP-based applications

- Surface-enhanced spectroscopy
 - CNT/NW-based FETs
 - Sensor decoration
 - Plasmonic devices
 - Quantum systems
 - Interconnections
 - Metamaterials



Top-down

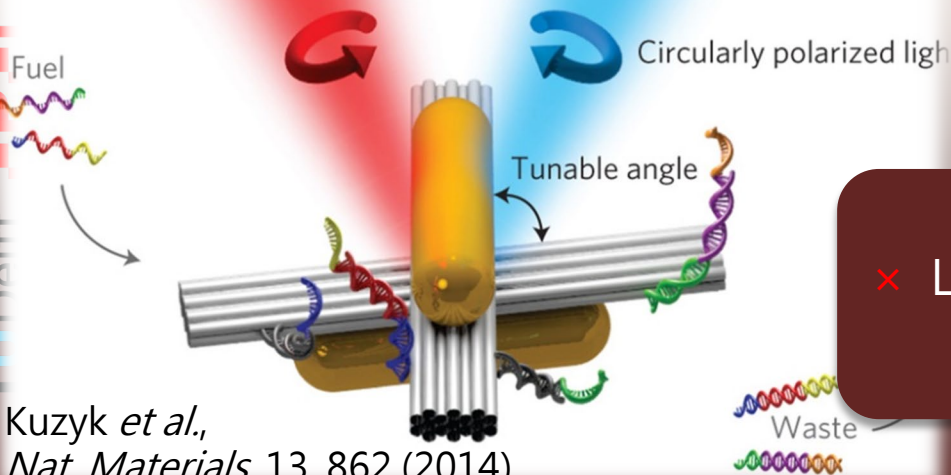
- ✓ Ultimate spatial accuracy
- ✗ Limited material choice & quality



Top-down & bottom-up?

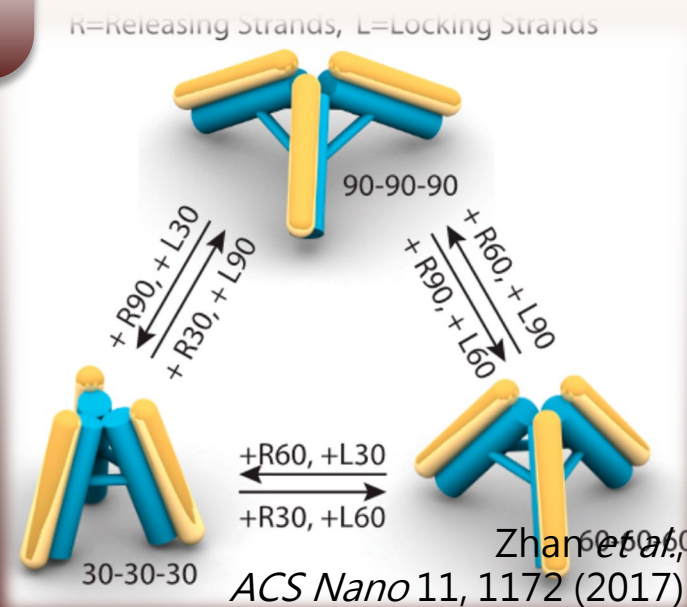
Assembly of chemically synthesized NPs over lithographically patterned substrates

3D plasmonic metamolecule



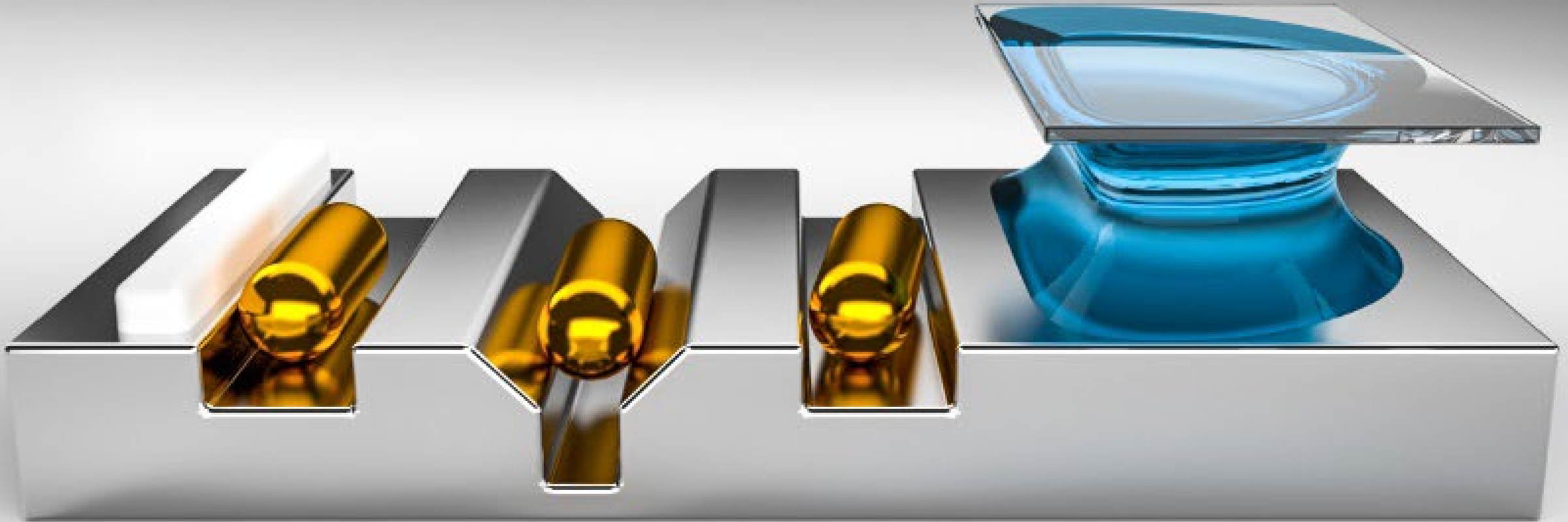
Kuzyk *et al.*,
Nat. Materials. 13, 862 (2014)

- ✓ Superior NP quality
 - ✗ Limited morphology control
- ## Bottom-up



Single nanoparticle printing

Assembly of chemically synthesized NPs
over lithographically patterned substrates



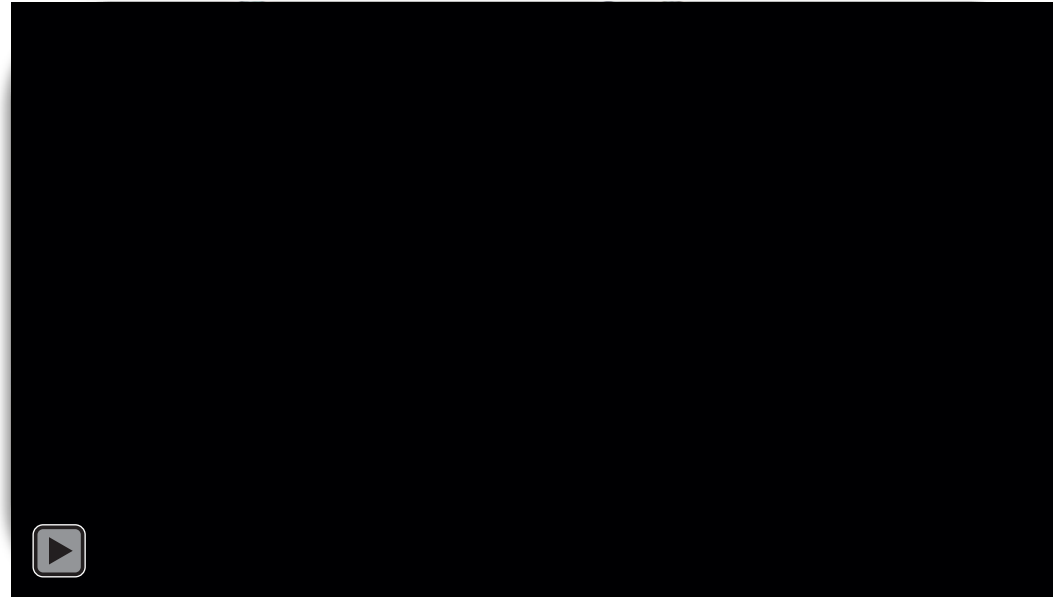
V. Flauraud, M. Mastrangeli, G. Bernasconi, J. Butet, D. Alexander, E. Sharabi, O. Martin, J. Brügger,
Nanoscale Topographical Control of Capillary Assembly of Nanoparticles
Nature Nanotechnology 12, 73-80 (2017)

Eppur si muove: Diffusion



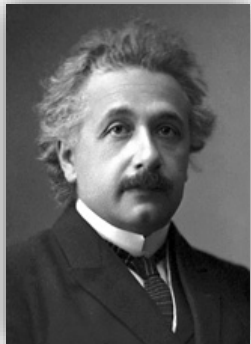
Robert Brown
(1828)

$$\frac{\langle x^2 \rangle}{2t} = D = \mu k_B T = \frac{\mu R T}{6\pi\eta r N}$$



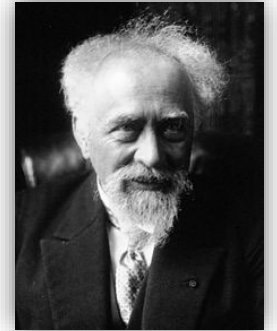
[source: Y. Y. Kiat's YouTube channel]

Pollen grains in water



Albert Einstein
(1905)

$$J = -D \nabla c \quad \text{Fick's 1st law}$$
$$\frac{\partial c}{\partial t} = D \nabla^2 c \quad \text{Fick's 2nd law}$$



Jean Perrin
(1908)



Paul Langevin
(1908)

Diffusivity

Confinement & templating

- Dimensionality

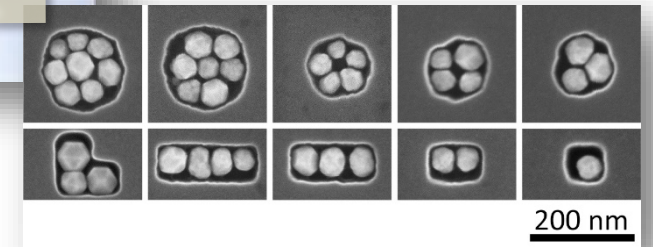
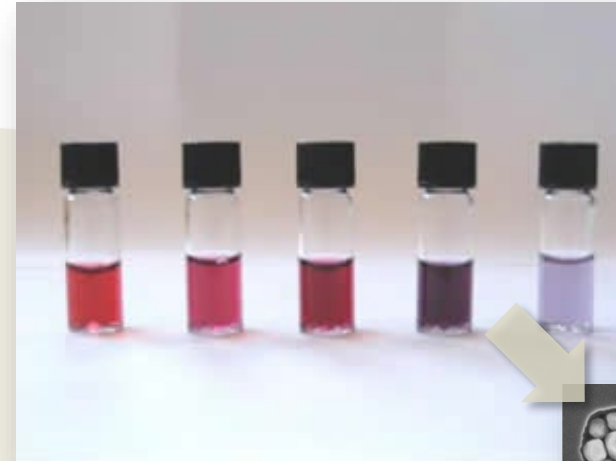
- From suspension... (3D)
to substrate... (2D)
to trap... (1D→0D)

- Degrees of freedom

- From 6 to 0

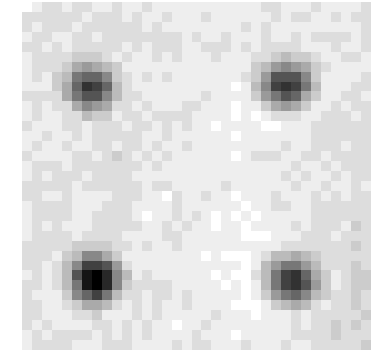
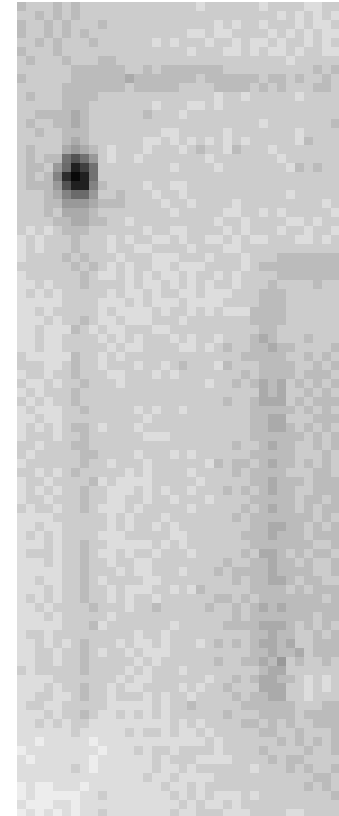
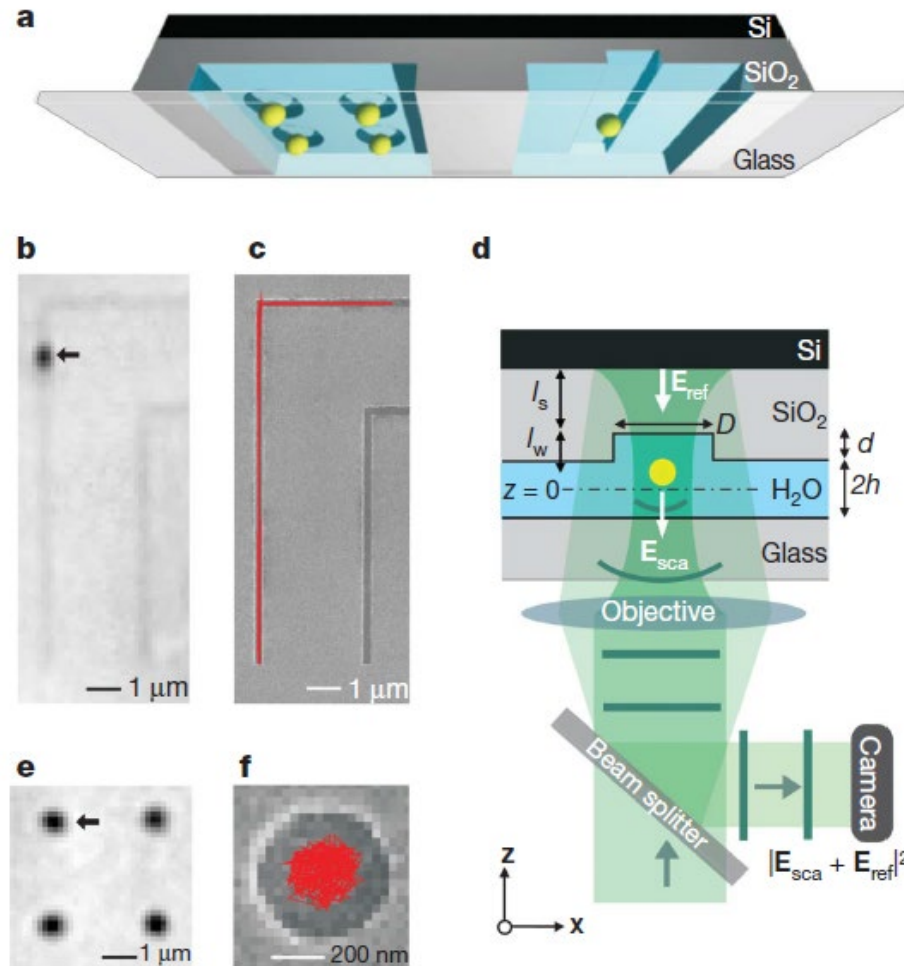
- Non-interacting particles

- $\Delta H \approx 0 \rightarrow \Delta G \approx -T\Delta S$



Assembly as
spatial search
problem

Electro-topographical trapping

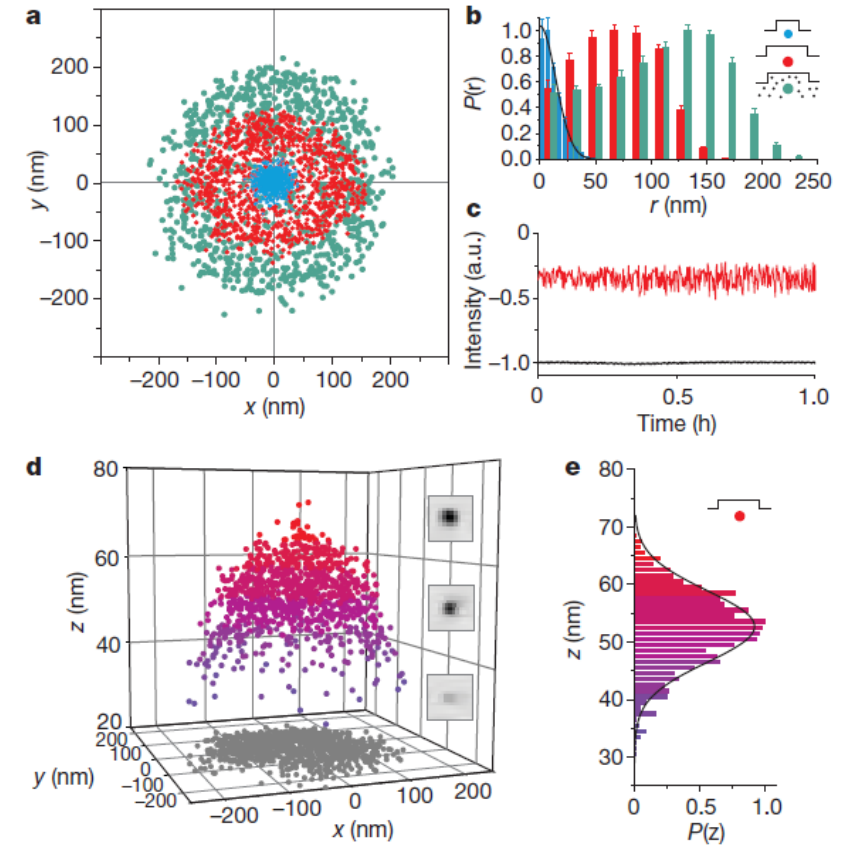
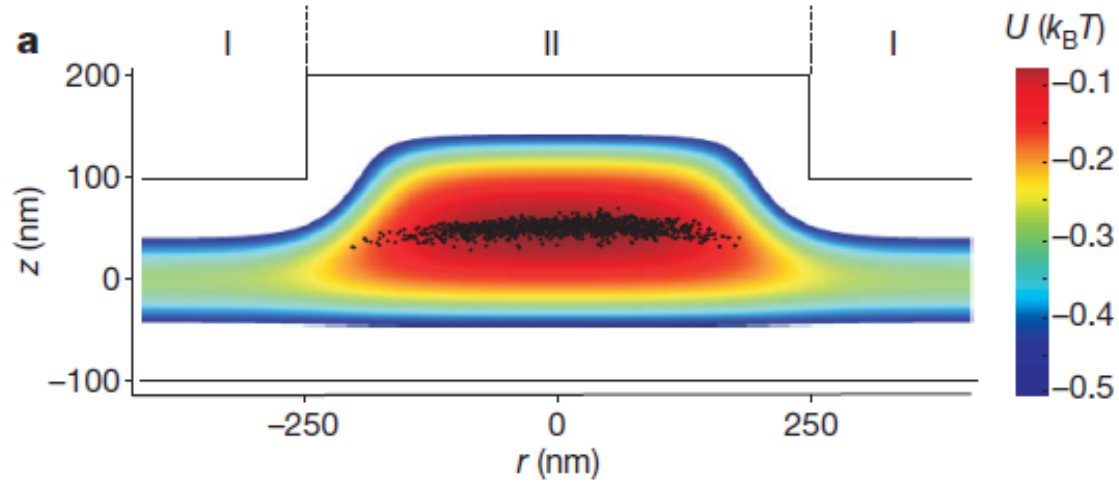


Krishnan *et al.*, *Nature* 467, 692 (2010)

Localization

Single nanoparticle printing | M. Mastrangeli, PhD – EPFL, 26 August 2022

Electro-topographical trapping

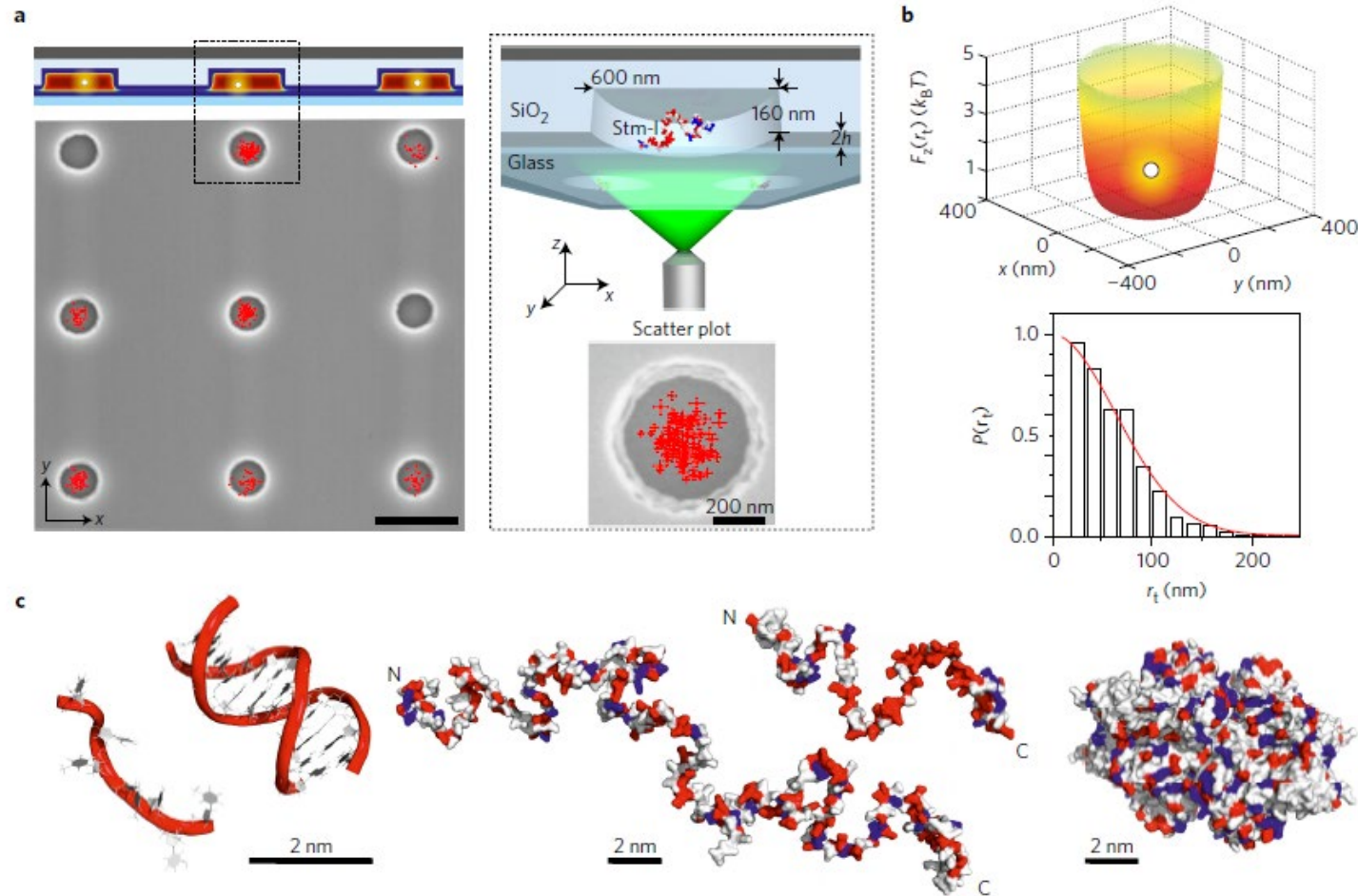


Krishnan *et al.*, *Nature* 467, 692 (2010)

Localization

Single nanoparticle printing | M. Mastrangeli, PhD – EPFL, 26 August 2022

Electro-topographical trapping

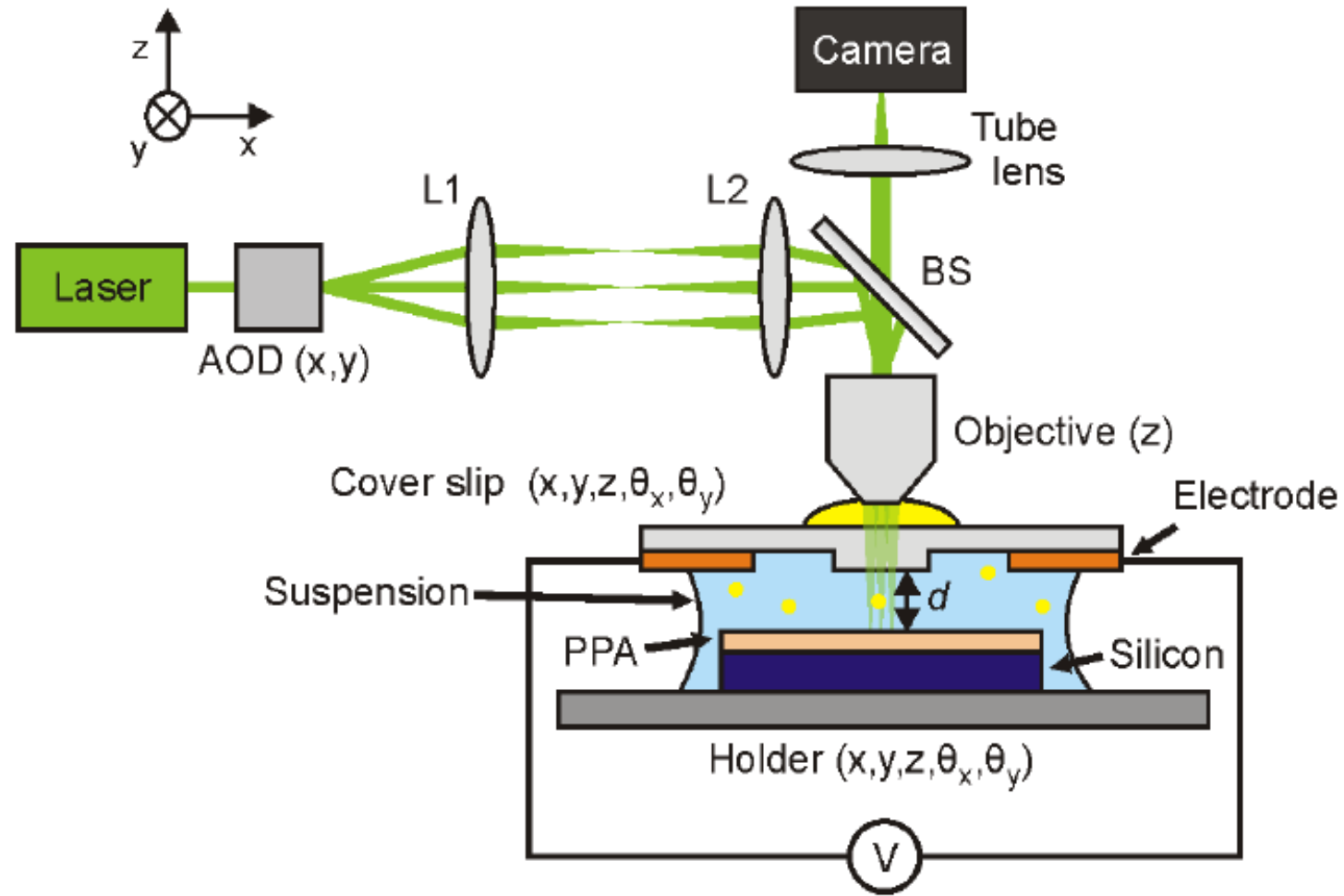


Ruggeri *et al.*, *Nat. Nanotechnol.* 12, 488 (2017)

Localization

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Rocking Brownian motors



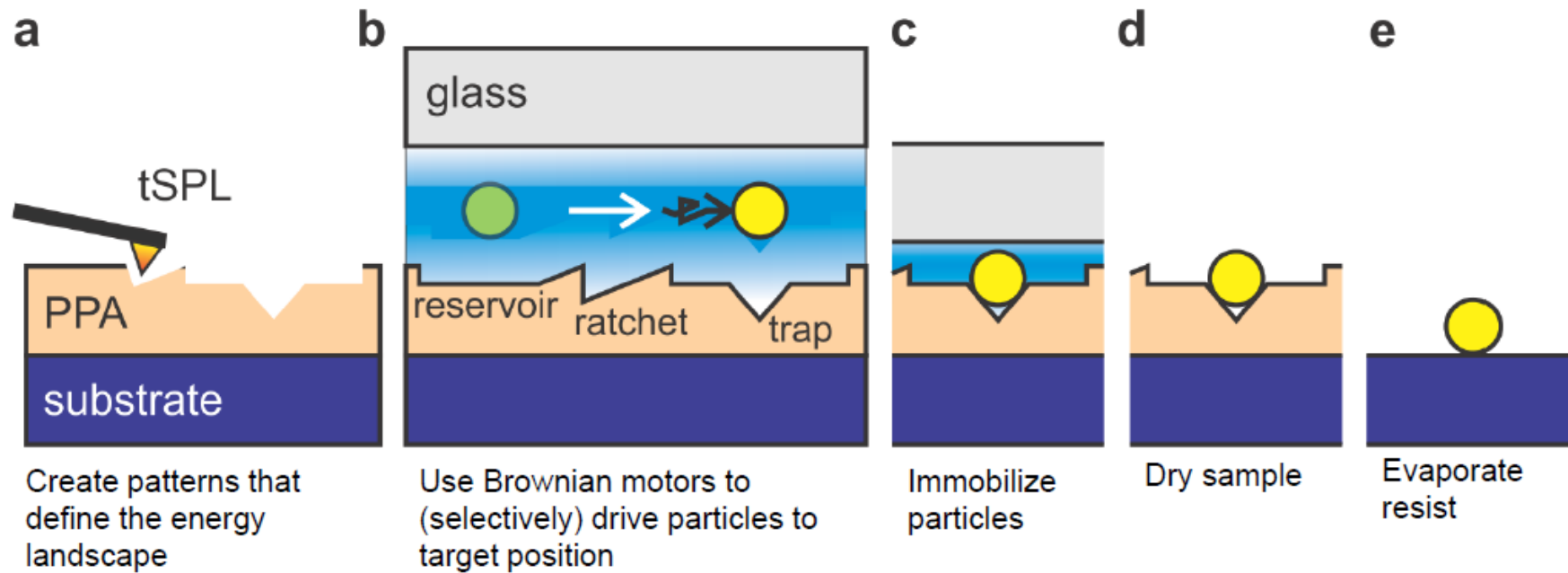
Fringes *et al.*, *J. Appl. Phys.* 2016

Fringes *et al.*, *Bellstein J. Nanotech.* 2018

Interferometric scattering detection (iSCAT)

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Rocking Brownian motors

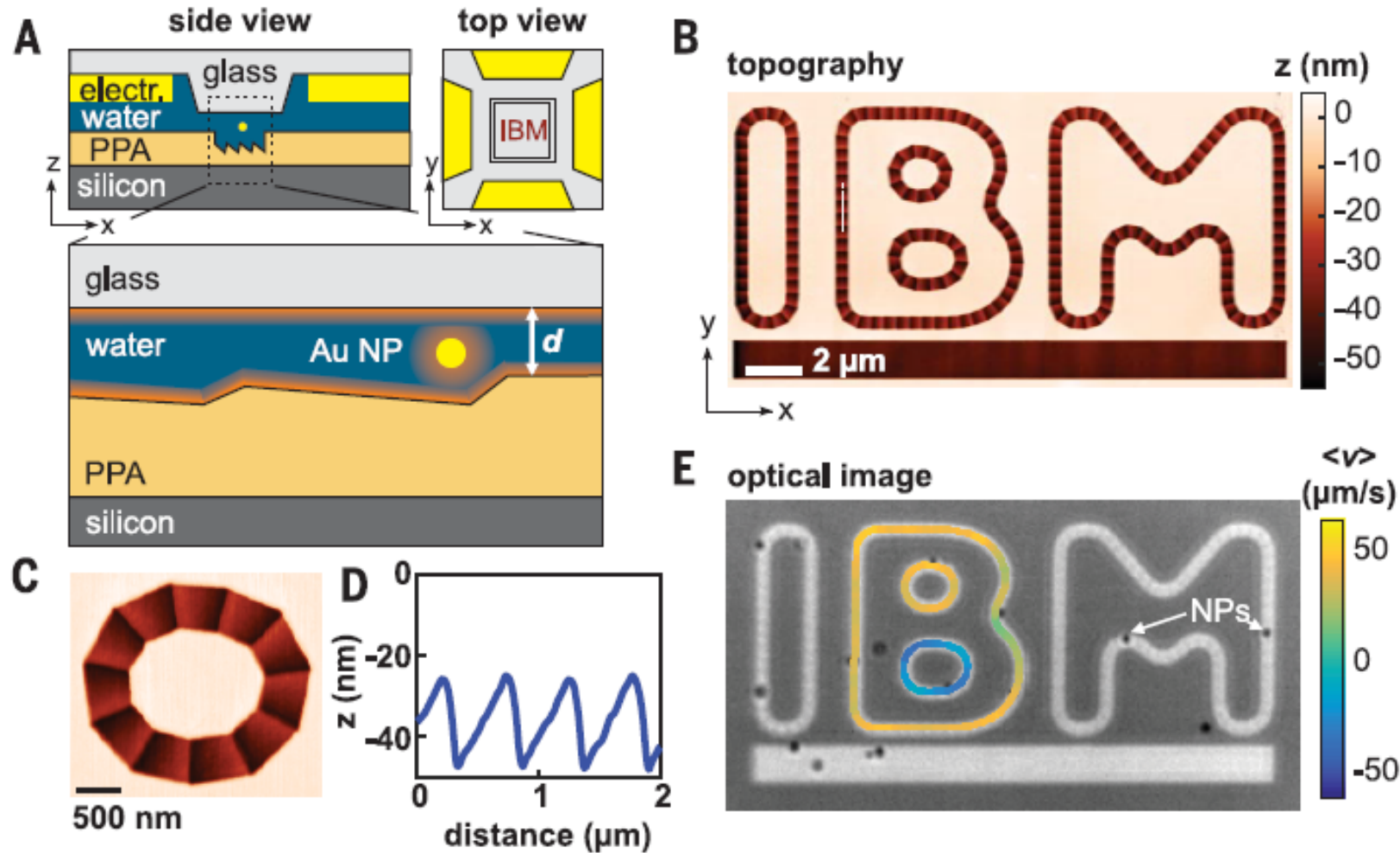


Knoll *et al.*, *TRANSDUCERS 2019*

tSPL-based topography

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Rocking Brownian motors

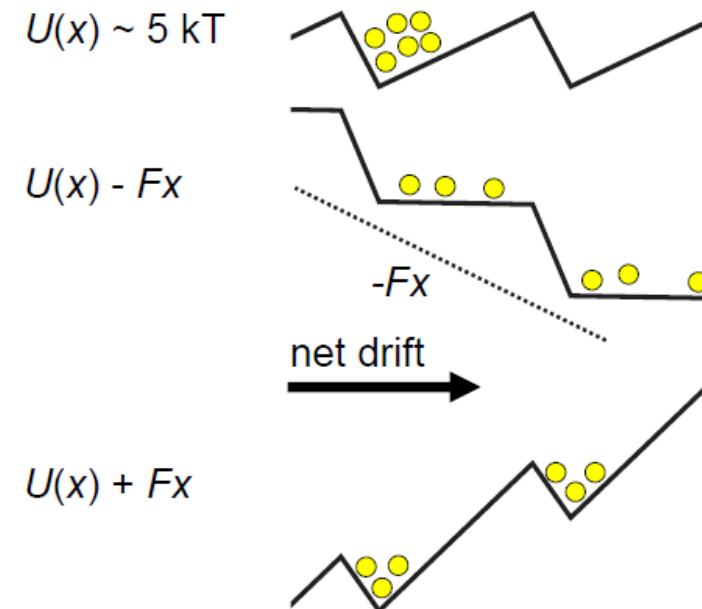
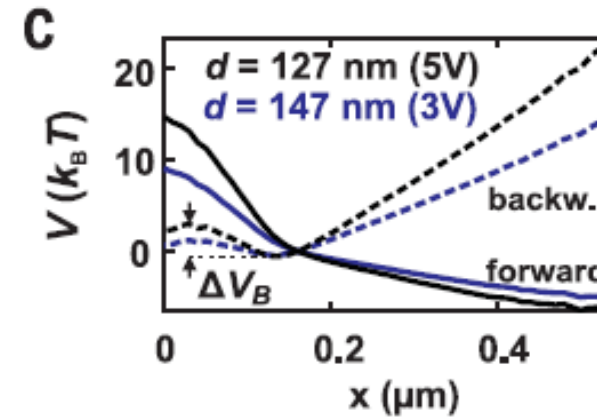
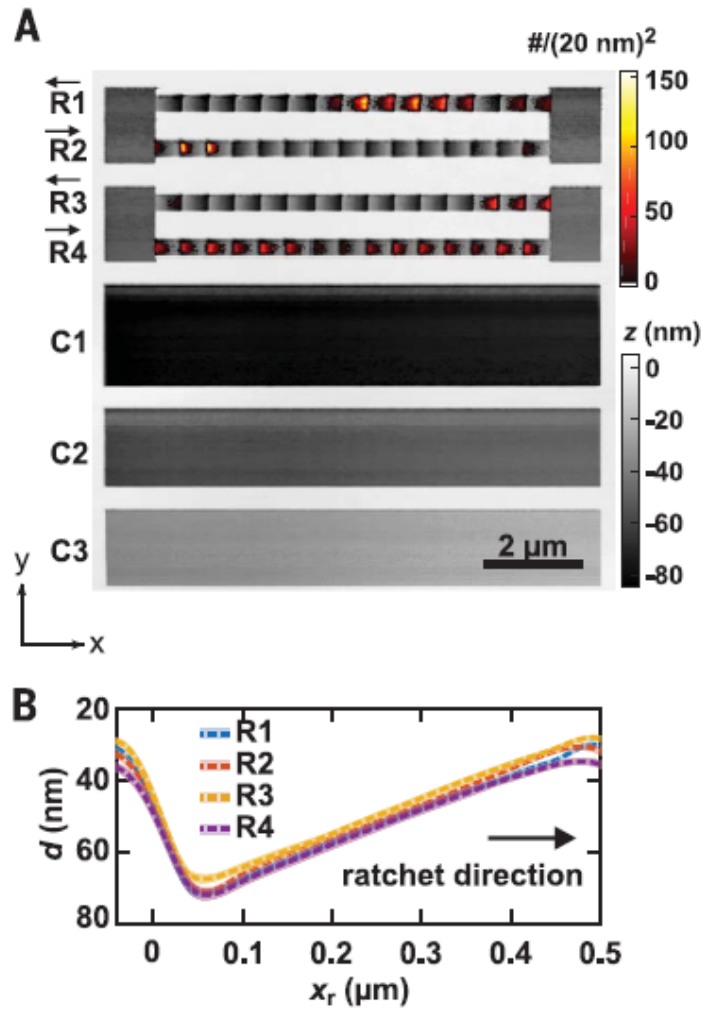


Skaug *et al.*, *Science* 359, 1505 (2018)

3D asymmetric ratchet topography

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Rocking Brownian motors

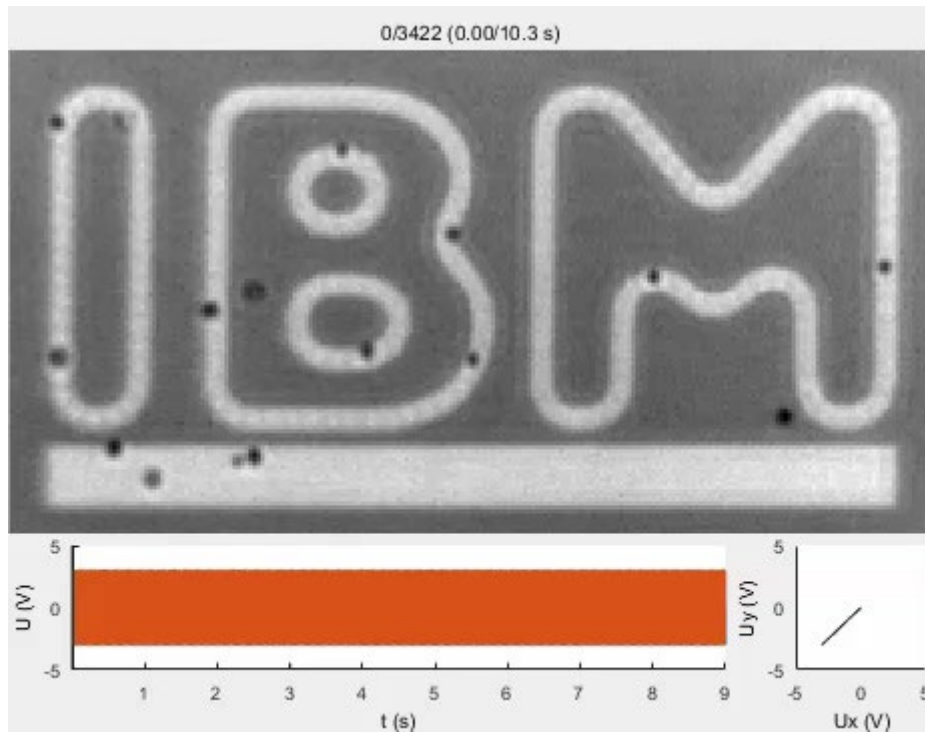
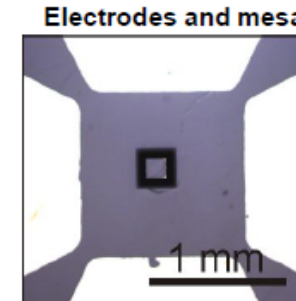
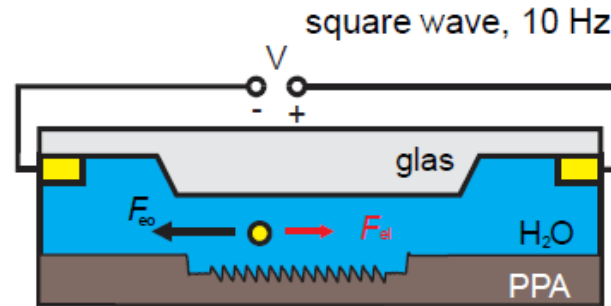


Skaug *et al.*, *Science* 359, 1505 (2018)

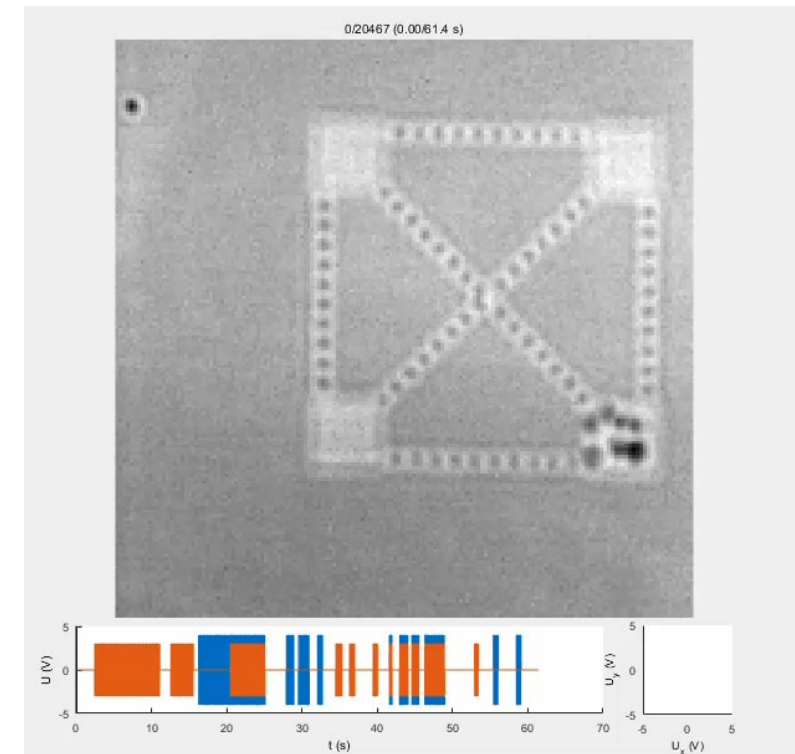
Working principle

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Rocking Brownian motors



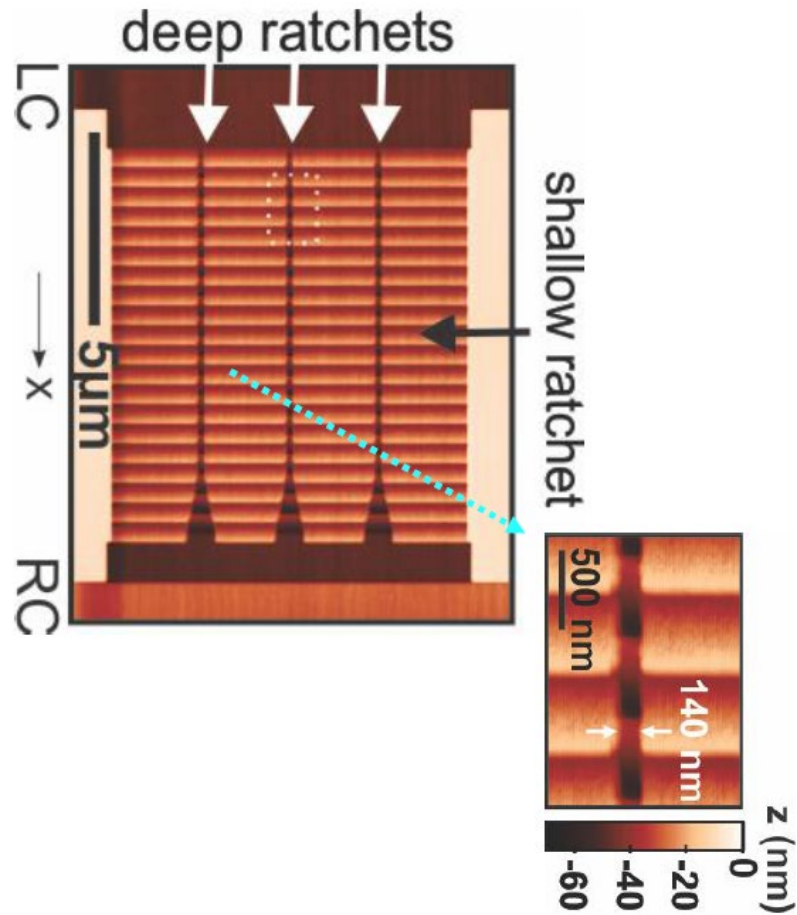
Skaug *et al.*, *Science* 359, 1505 (2018)



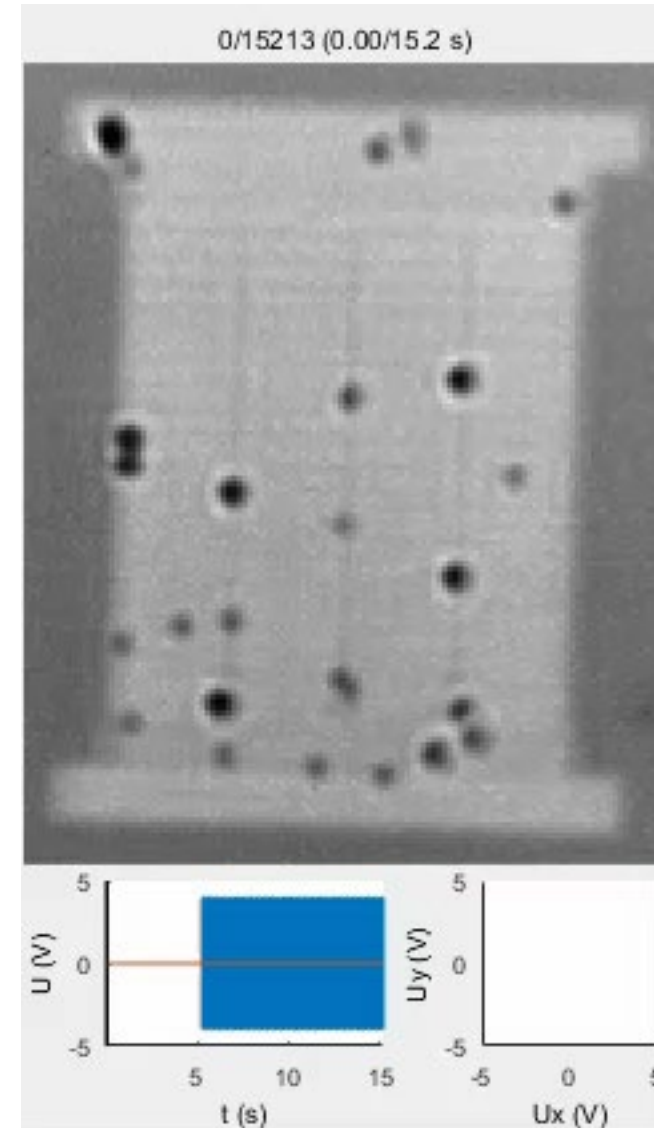
Demonstrations

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Rocking Brownian motors



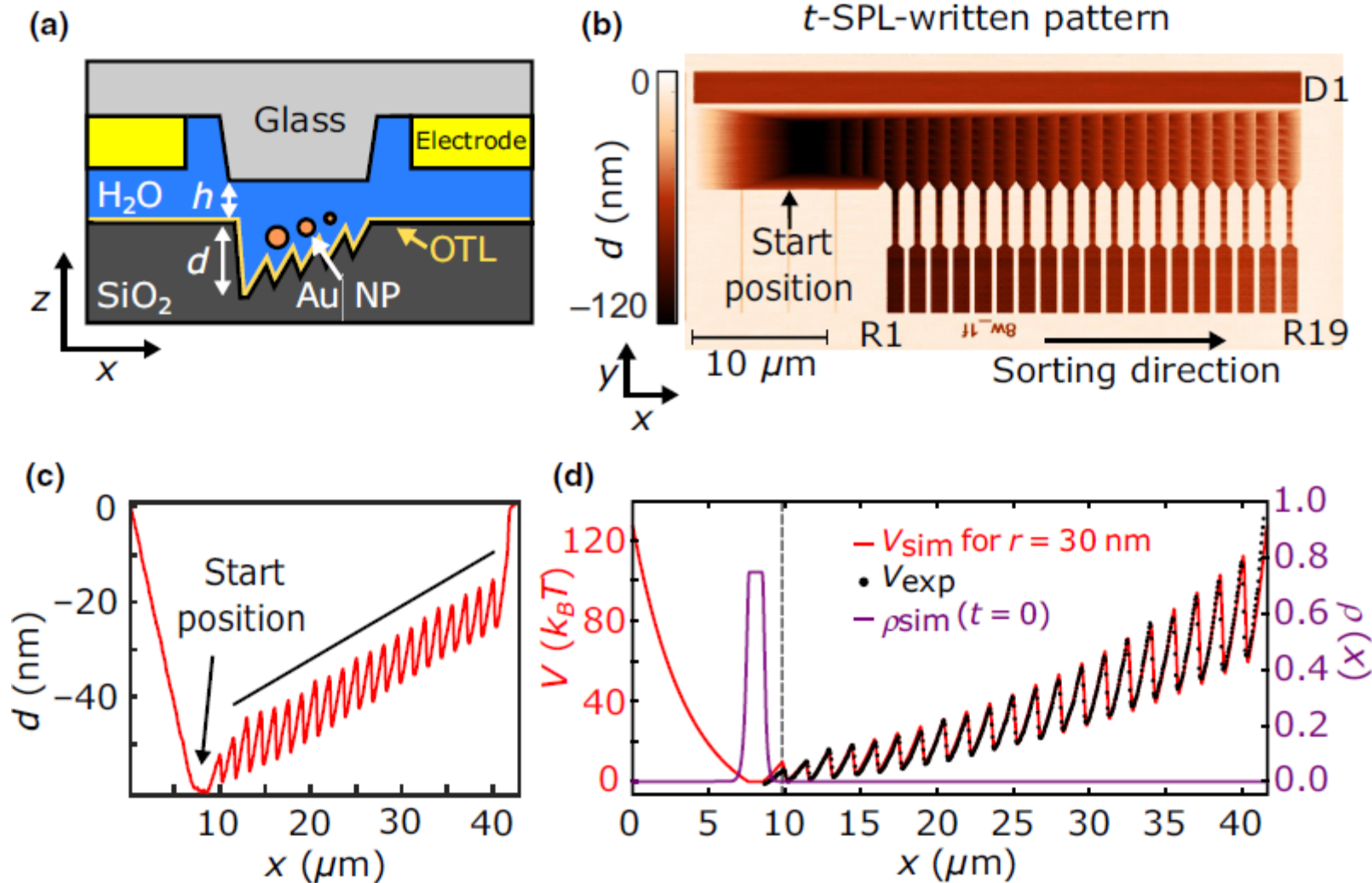
Skaug *et al.*, *Science* 359, 1505 (2018)



Particle sorting

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Rocking Brownian motors

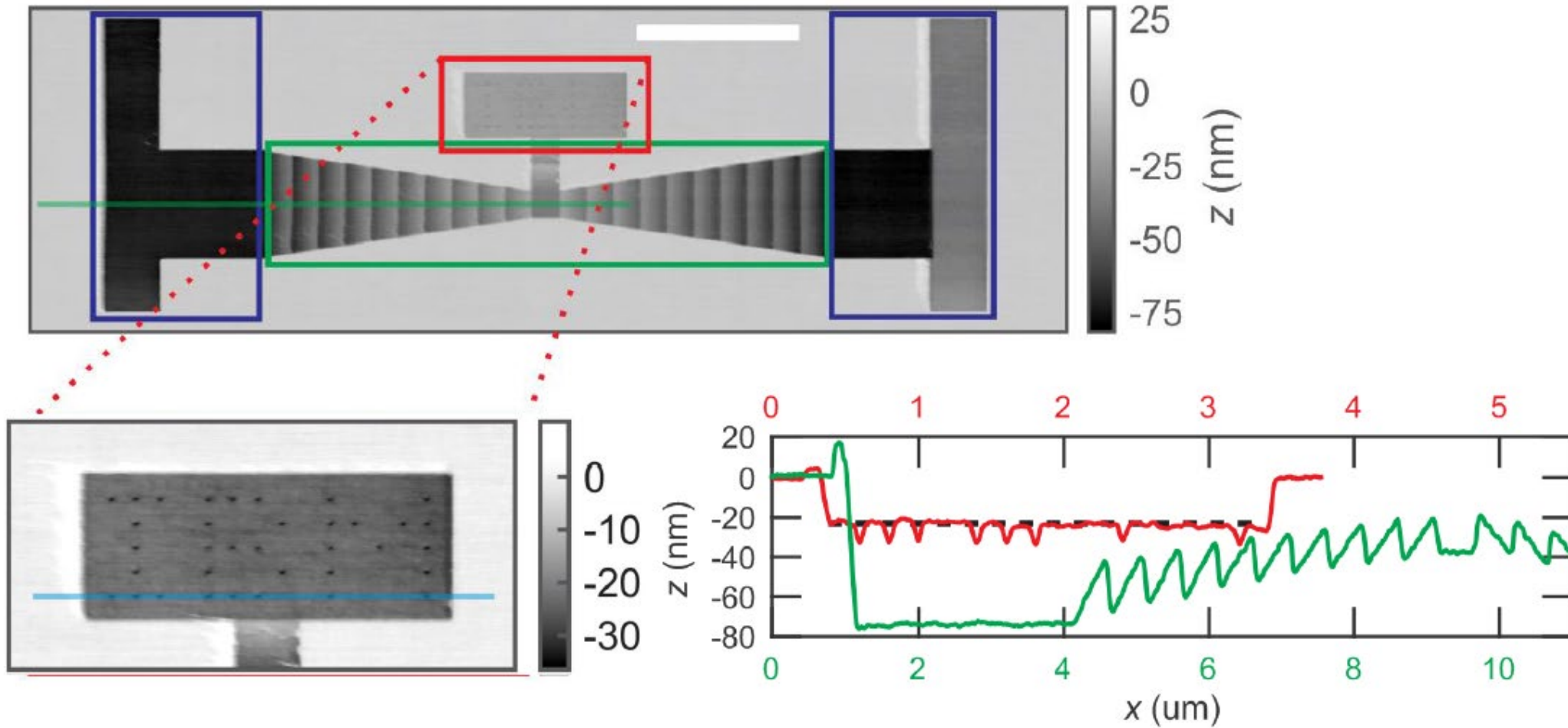


Nicollier *et al.*, *Phys. Rev. Appl.* 15, 034006 (2021)

Particle sorting

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Rocking Brownian motors

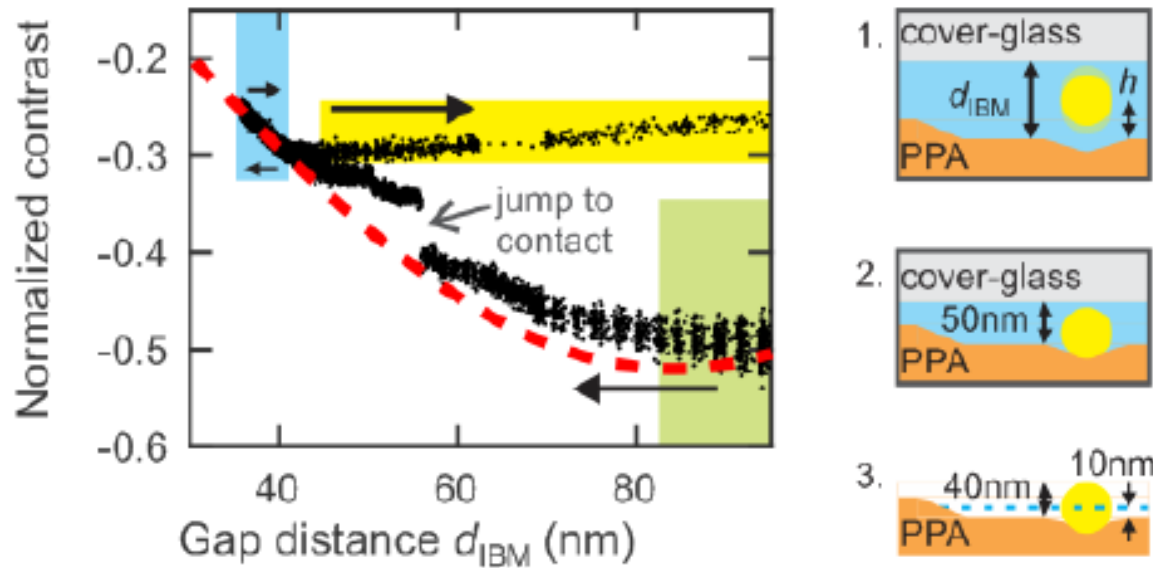


Knollet al., *TRANSDUCERS 2019*
Fringes et al., *Nano Letters* 19, 8855 (2020)

Particle placement

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Rocking Brownian motors



• Deposition pathway

- First contact with glass (still mobile)
- Jump into contact with polymer (5 to 9 nm)
- Elastically compressed system
- Final particle height: 10 nm above substrate surface

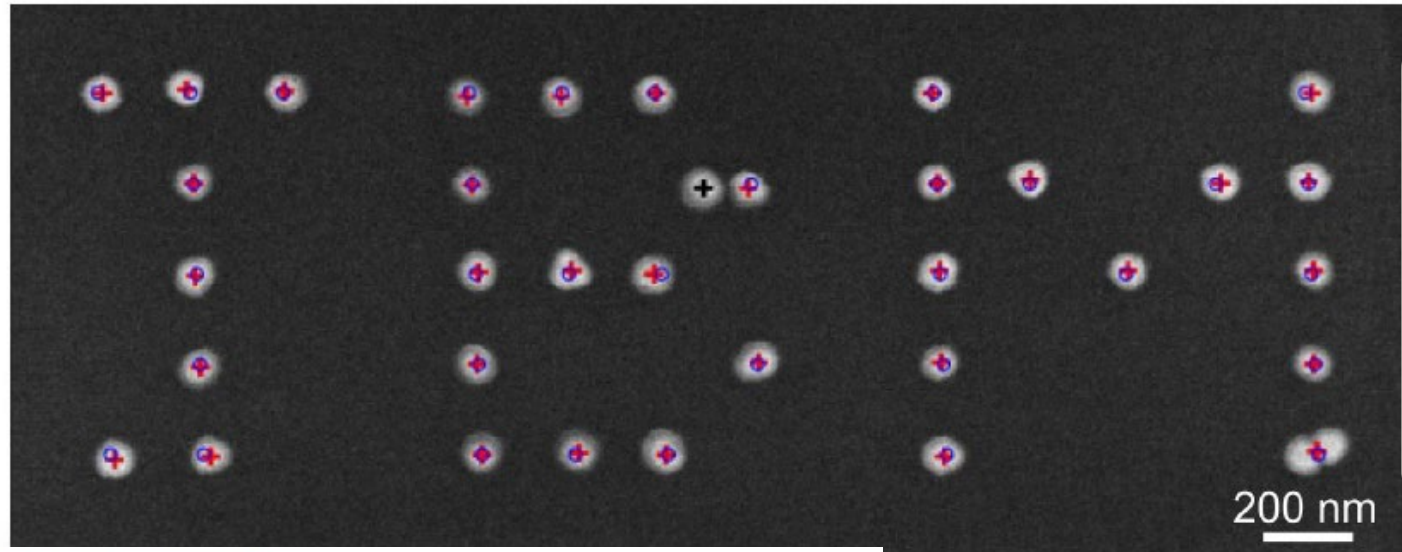
Knollet al., *TRANSDUCERS 2019*

Fringes et al., *Nano Letters* 19, 8855 (2020)

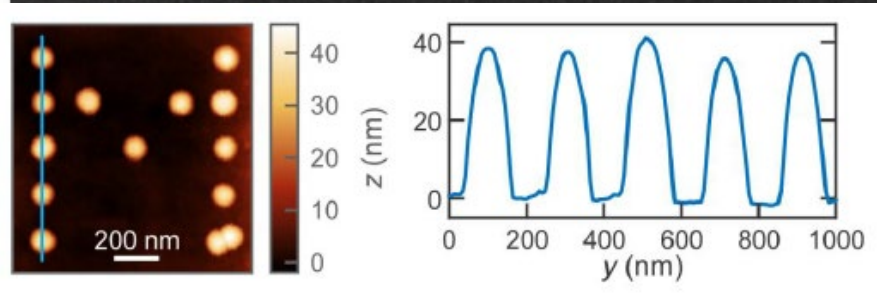
Particle placement

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Rocking Brownian motors



- Error sources
 - Placement
 - Lithography accuracy
 - Particle motion upon resist evaporation
 - SEM distortions (non-linear drift)

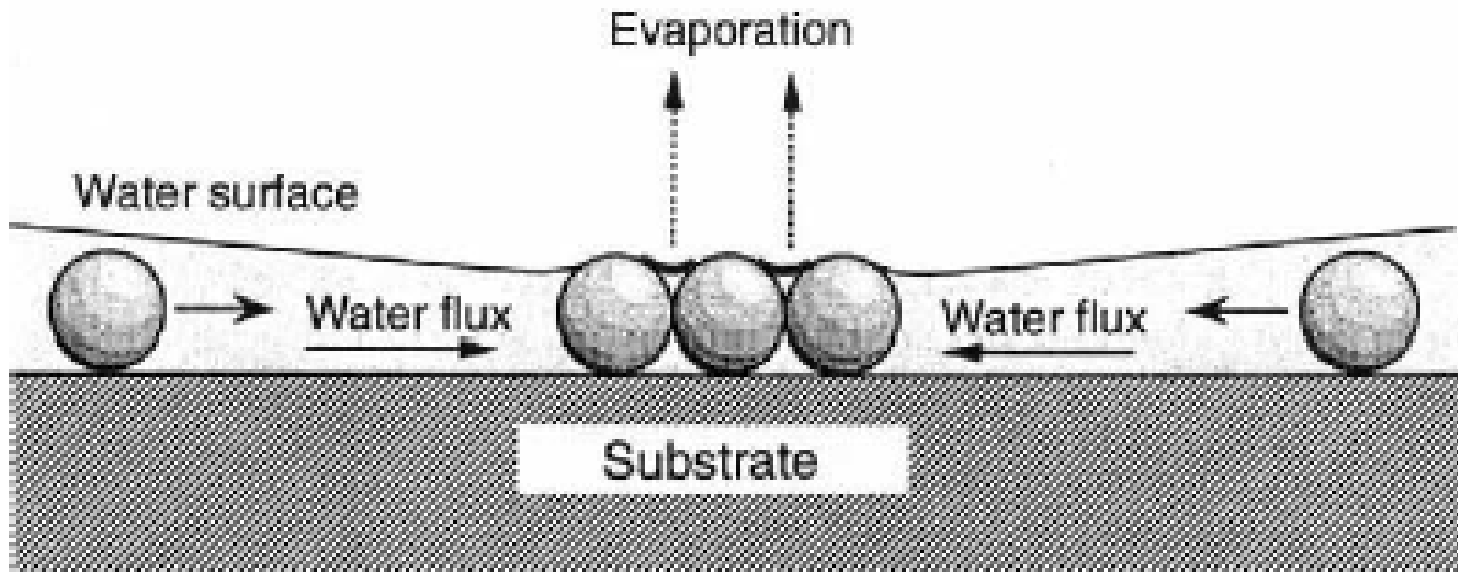


Knoll *et al.*, *TRANSDUCERS 2019*

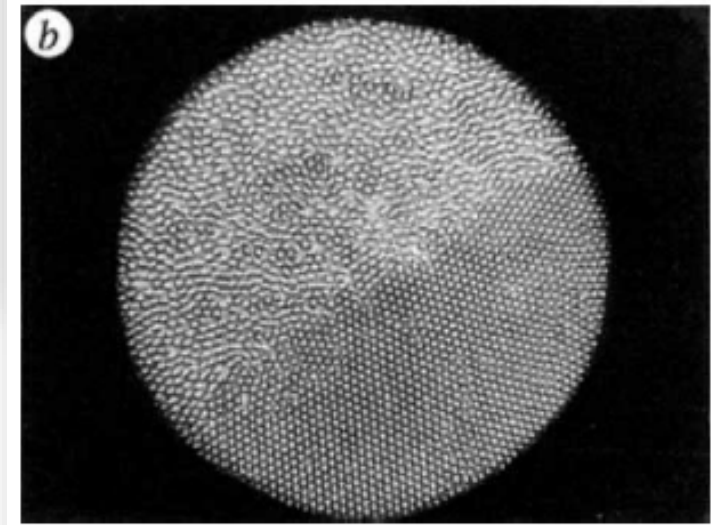
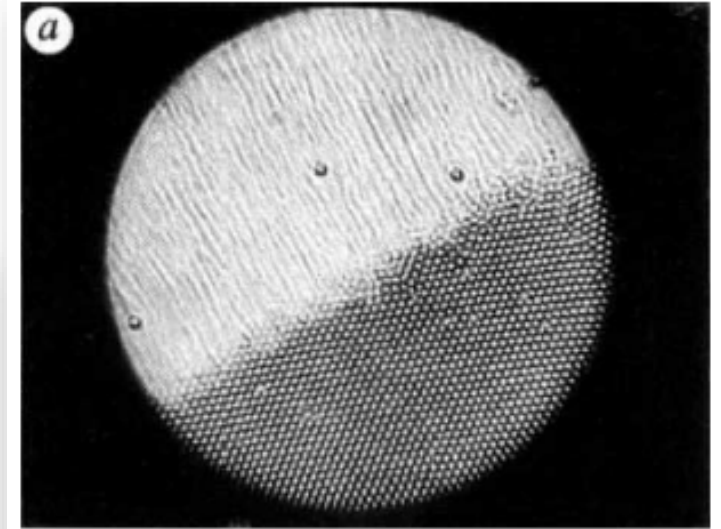
Fringes *et al.*, *Nano Letters* 19, 8855 (2020)

Localization

From 3D to 2D: Capillary crystallization



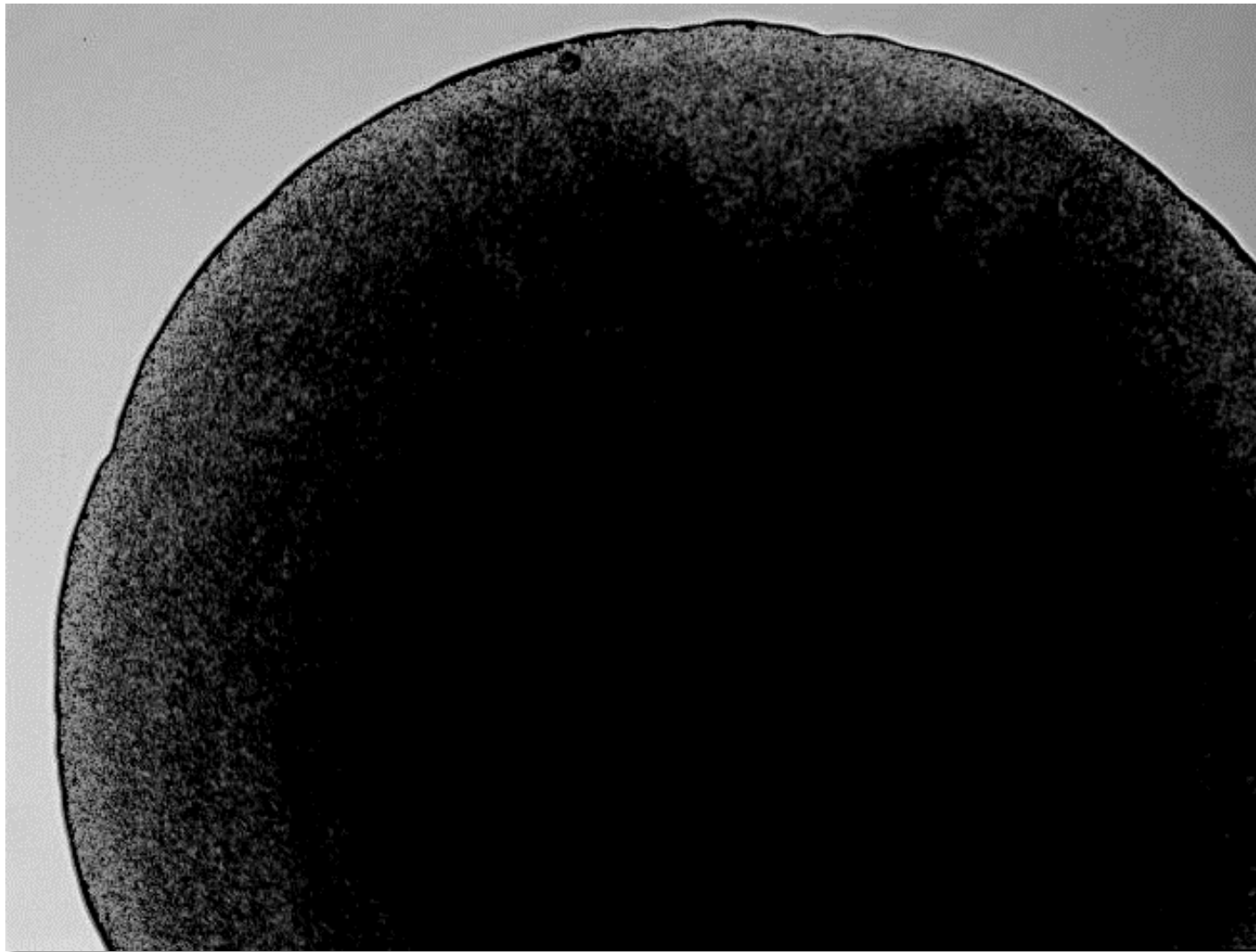
Denkov *et al.*, *Nature* 361, 26 (1993)



From suspension to substrate

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From 3D to 1D: Coffee ring effect

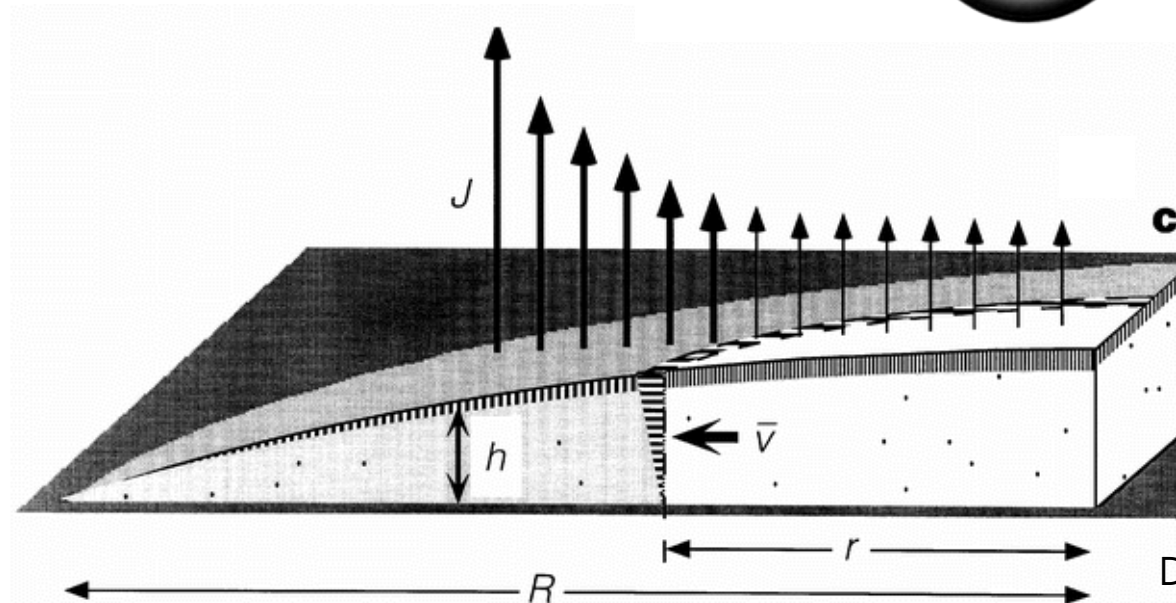
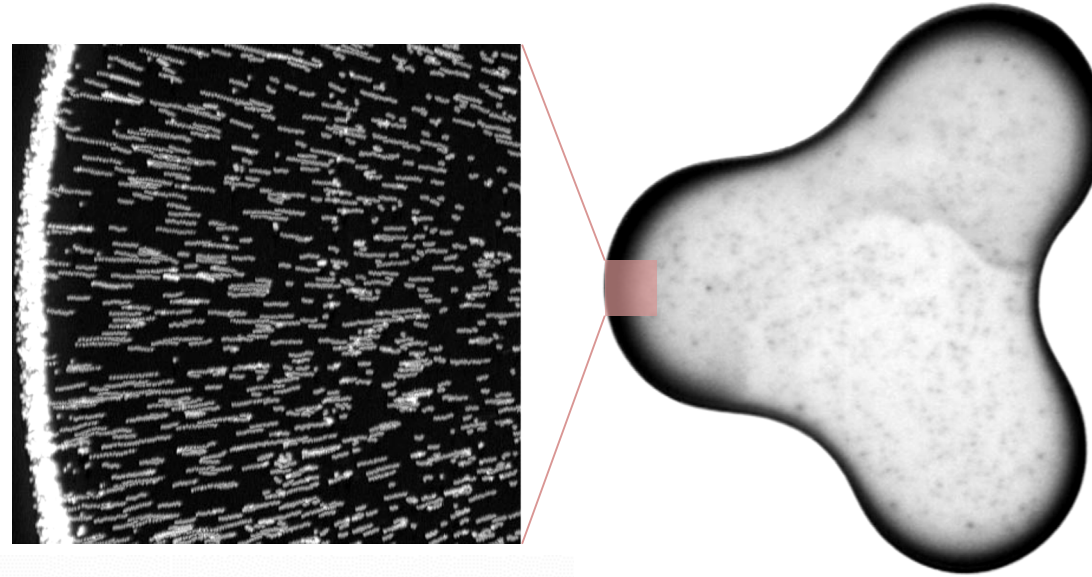


Still *et al.*, *Langmuir* 28, 4984 (2012)

From suspension to substrate

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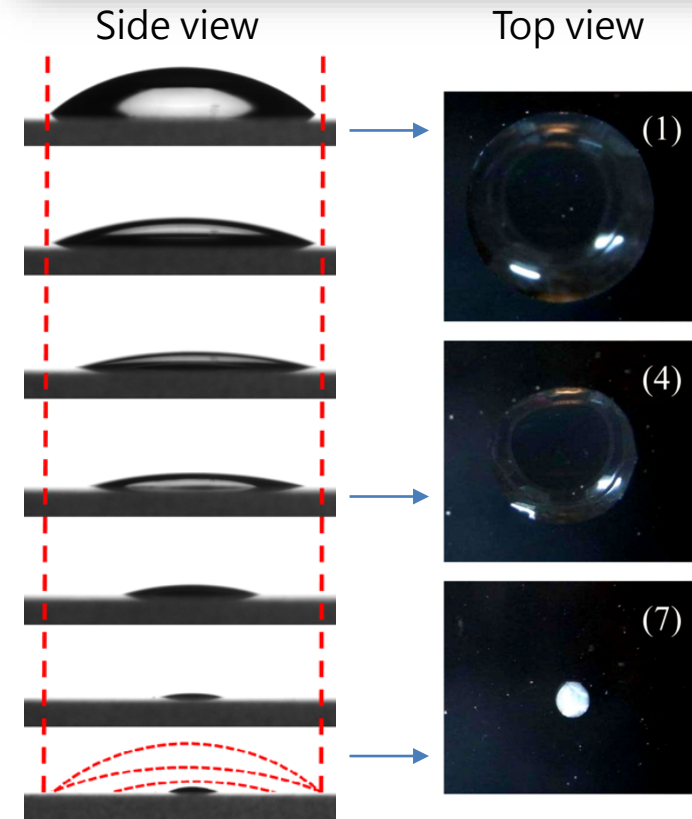
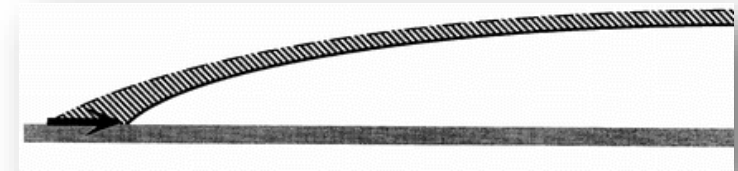
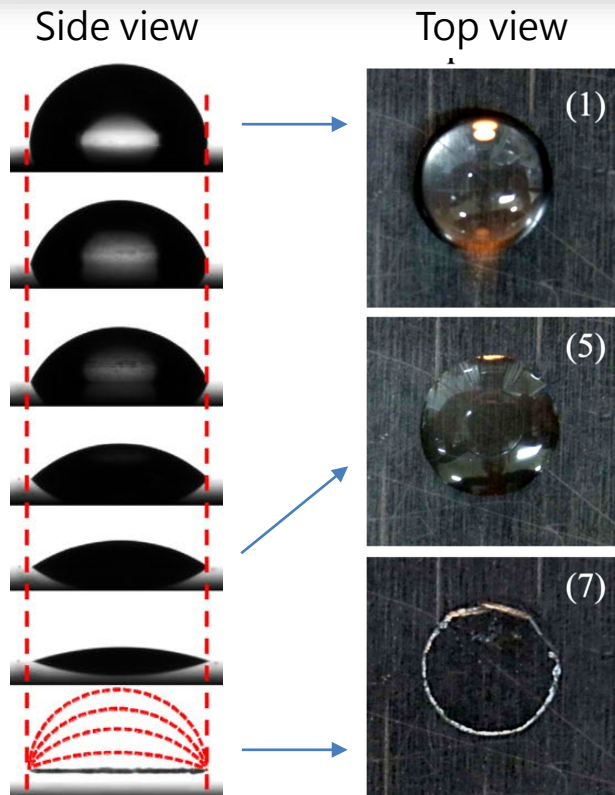
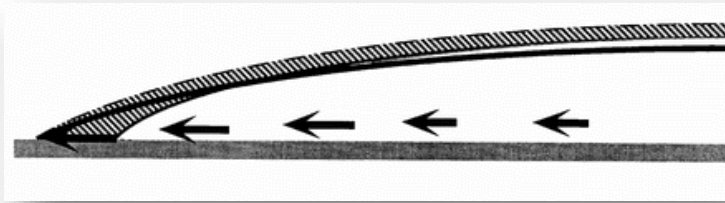
From 3D to 1D: Coffee ring effect



Deegan *et al.*, *Nature* 389, 6653 (1997)

From suspension to substrate

Drop casting

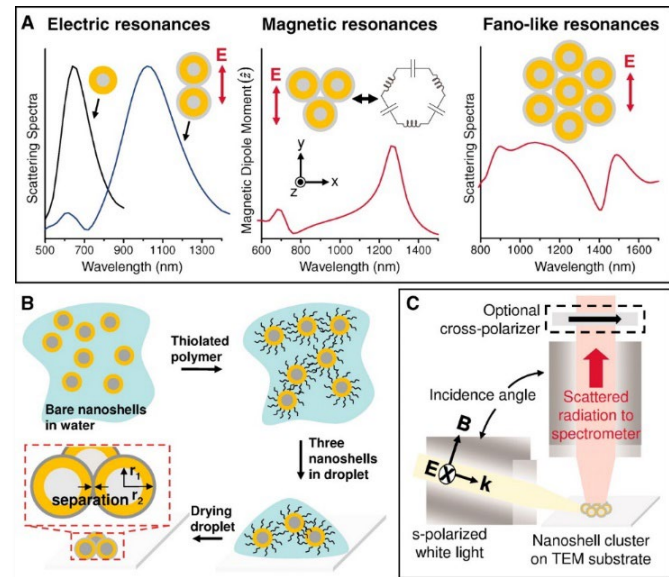


Li *et al.*, *Langmuir* 29, 782 (2013)

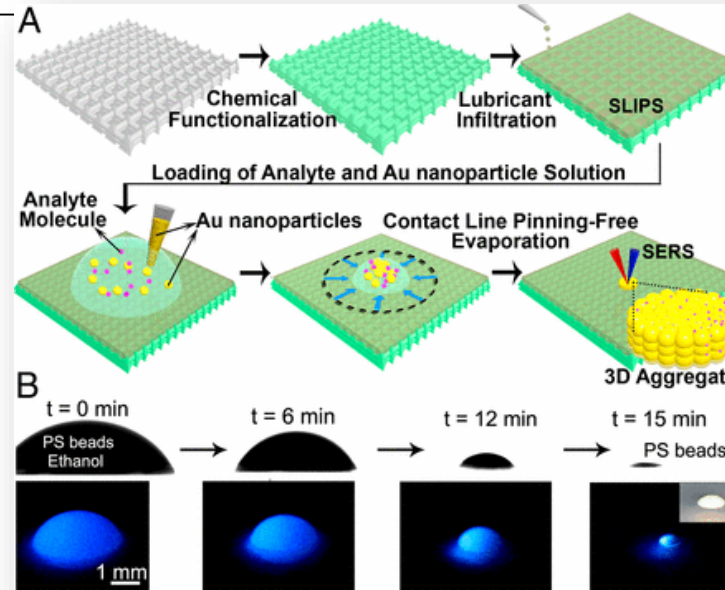
From suspension to substrate

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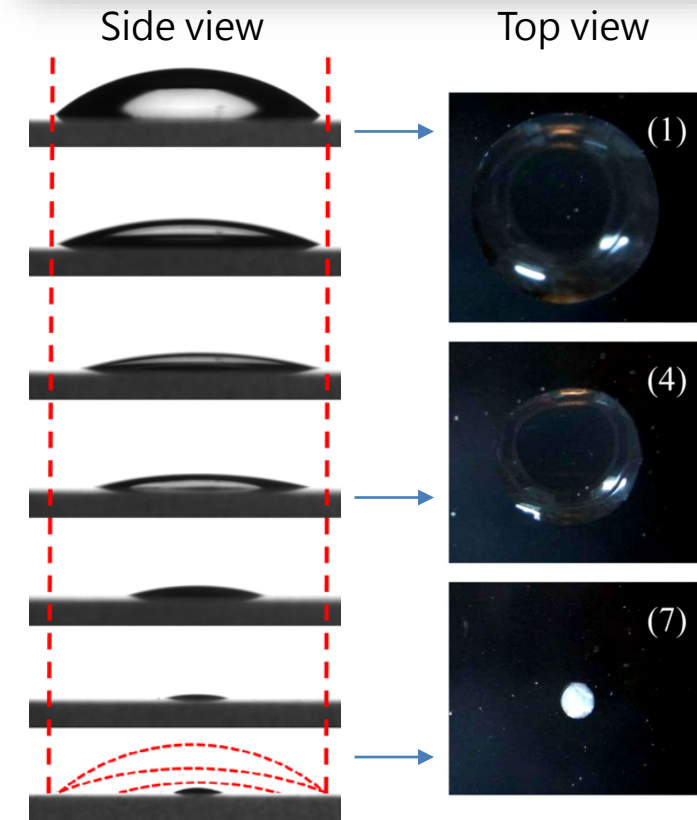
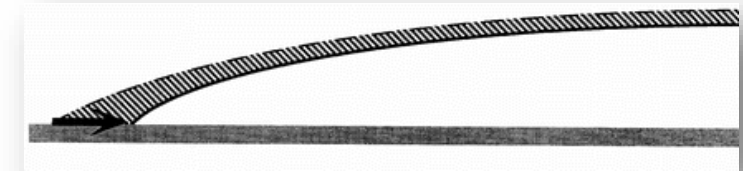
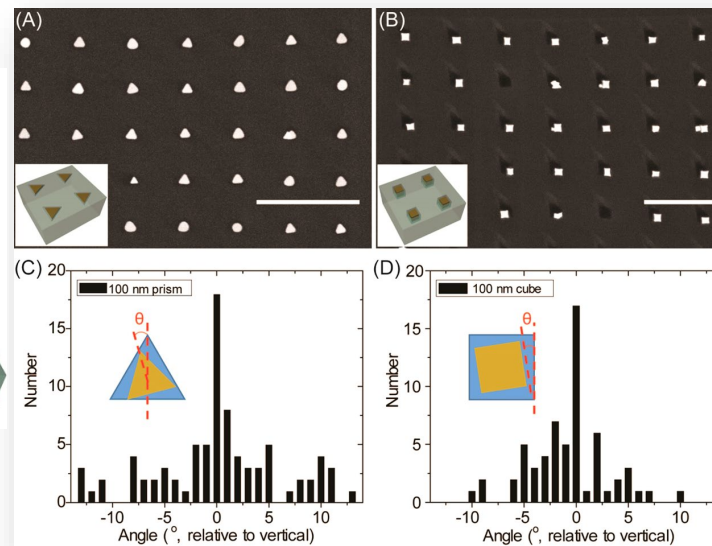
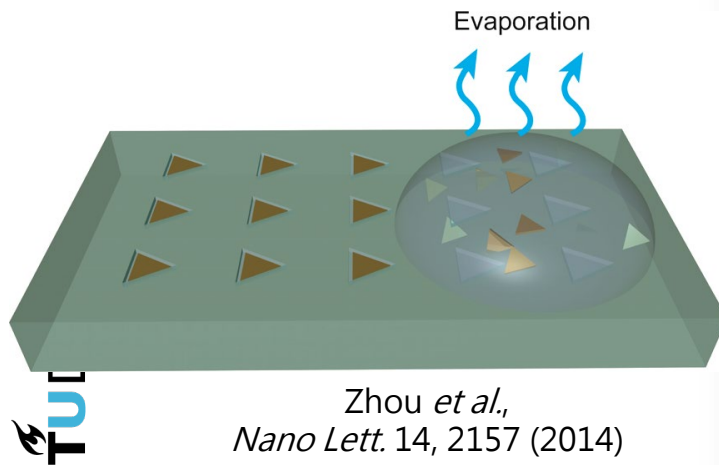
Drop casting



Fan *et al.*, *Science* 328, 1135 (2010)



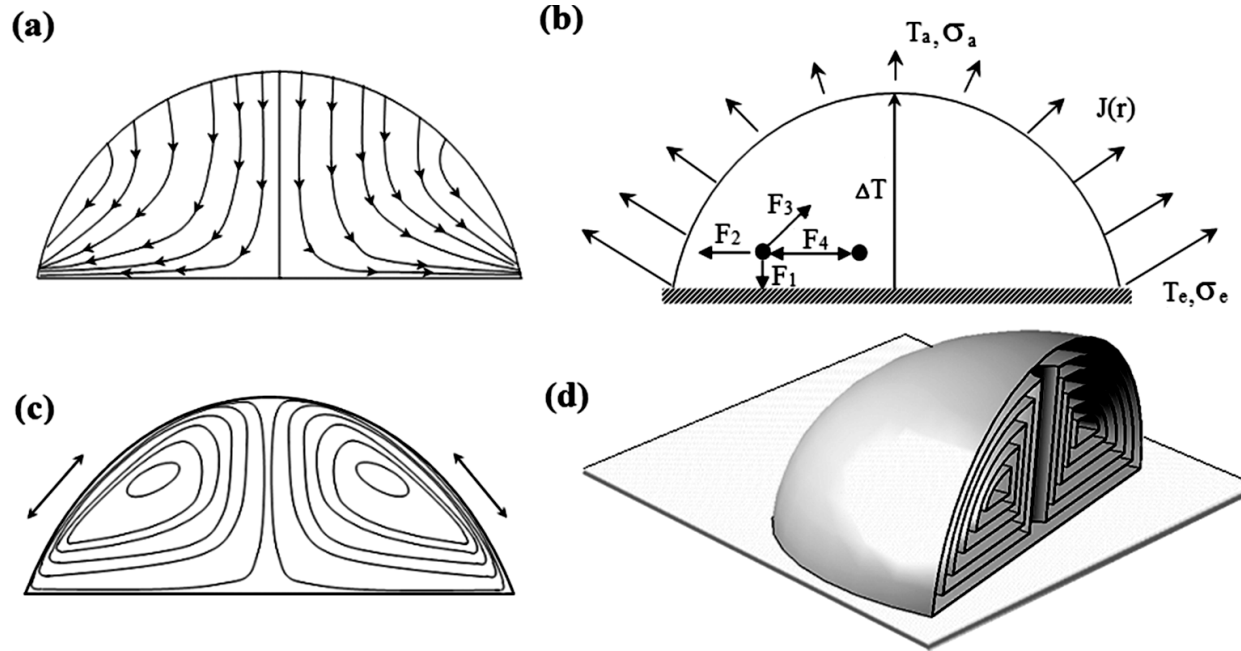
Yang *et al.*, *PNAS* 113, 268 (2016)



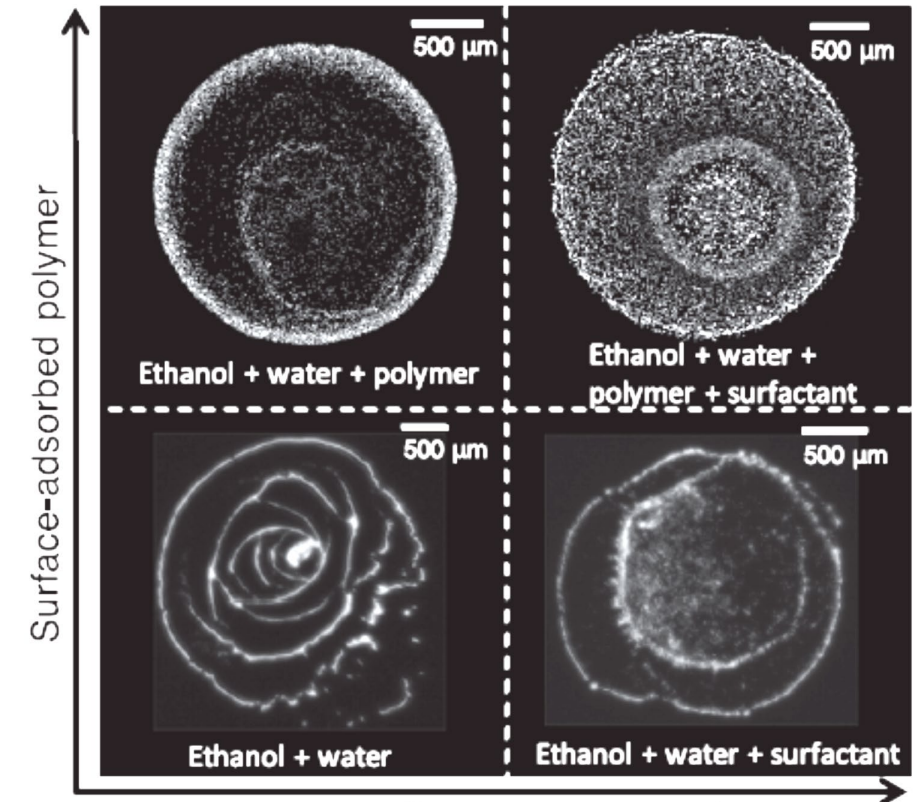
Li *et al.*, *Langmuir* 29, 782 (2013)

From suspension to substrate

Drop casting



Thokchom *et al.*, *Langmuir* 30, 12144 (2014)



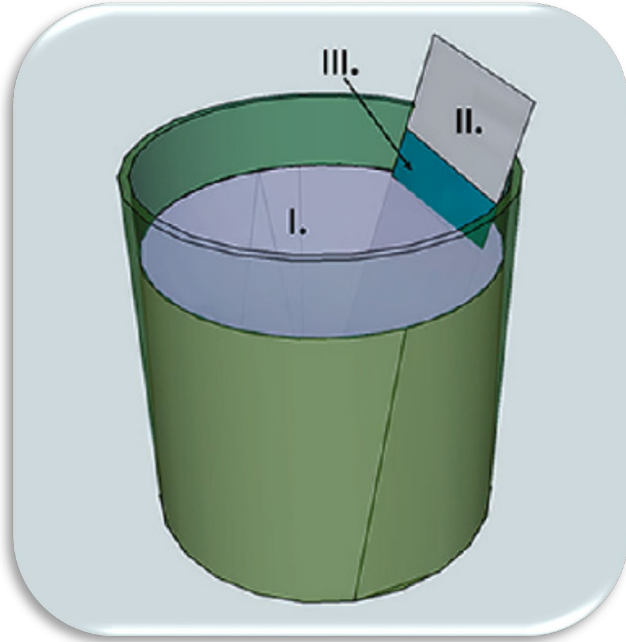
Kim *et al.*, *PRL* 116, 124501 (2016)

- ✓ Resource efficient
- ✗ Limited control

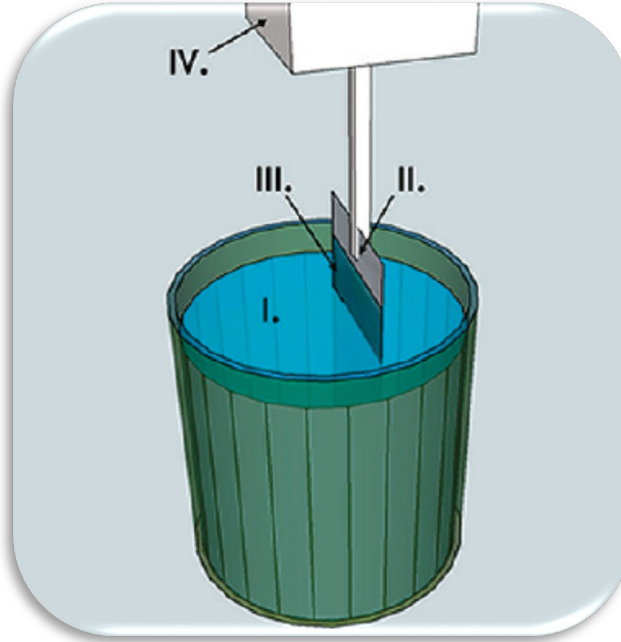
From suspension to substrate

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Dip coating

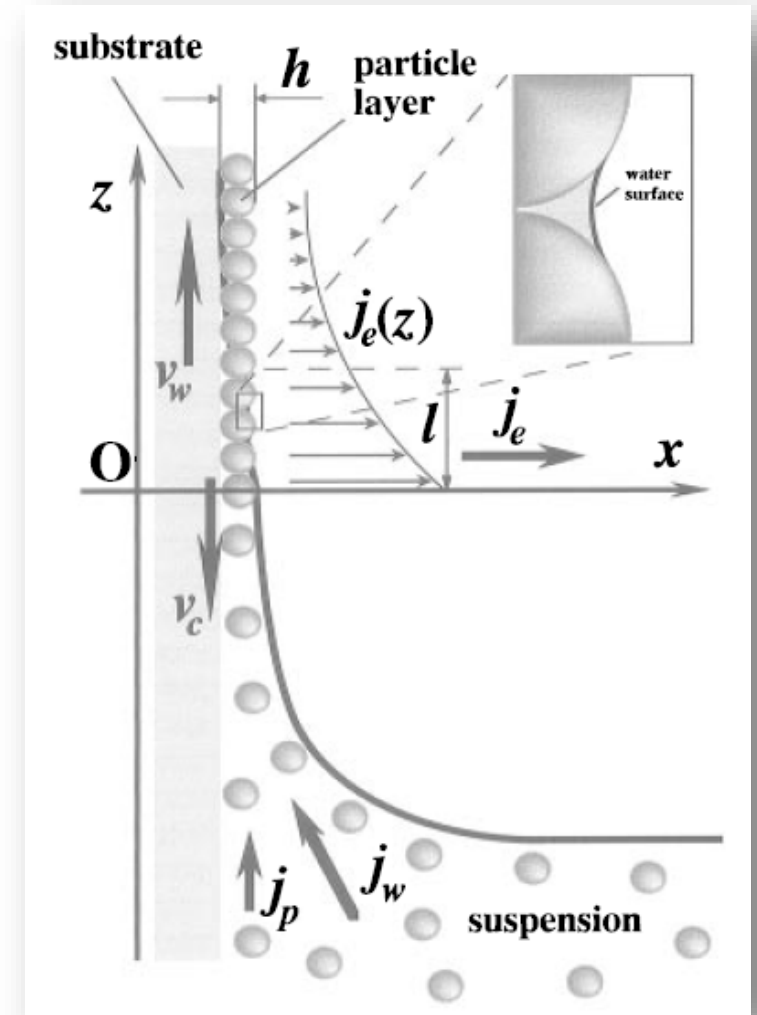


Evaporation-driven



Moving substrate

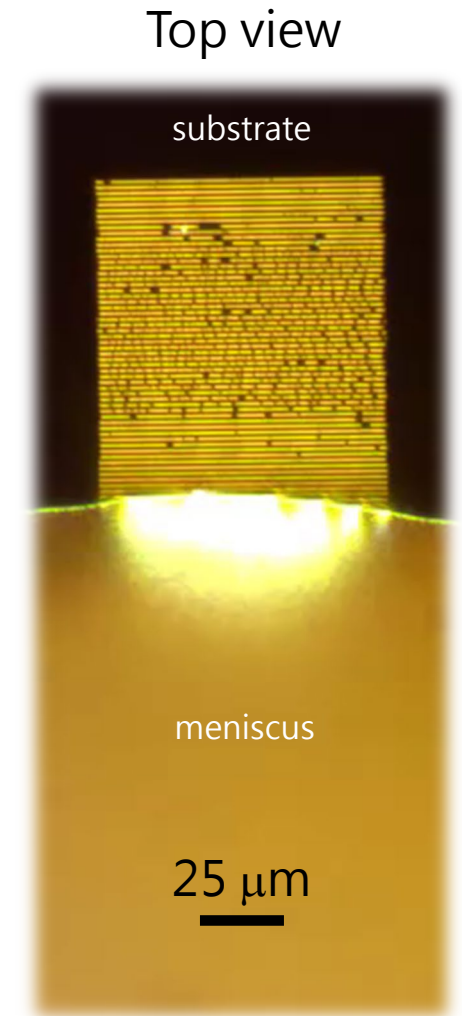
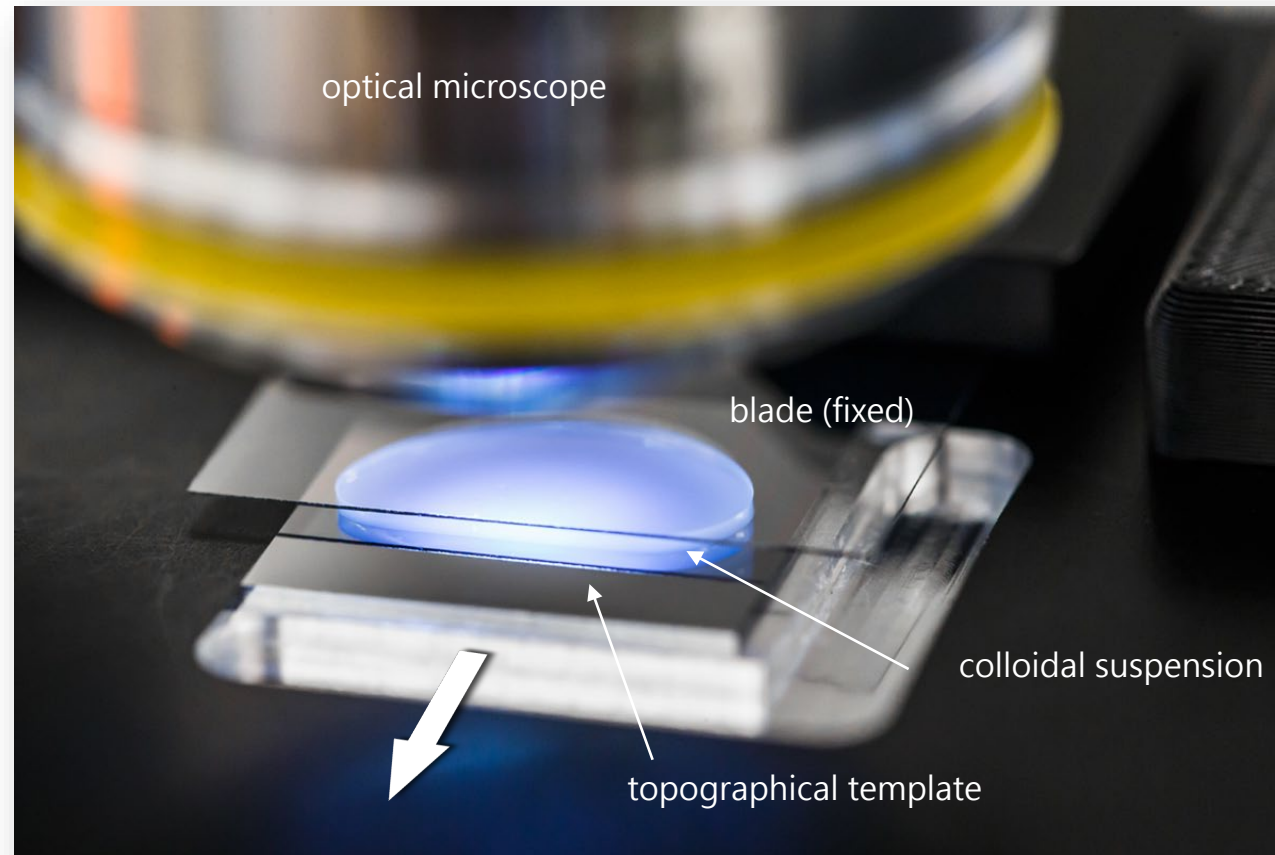
- ✓ Uniform deposition over large areas
- ✗ Limited control on meniscus geometry



Dimitrov & Nagayama,
Langmuir 12, 1303 (1996)

From suspension to substrate

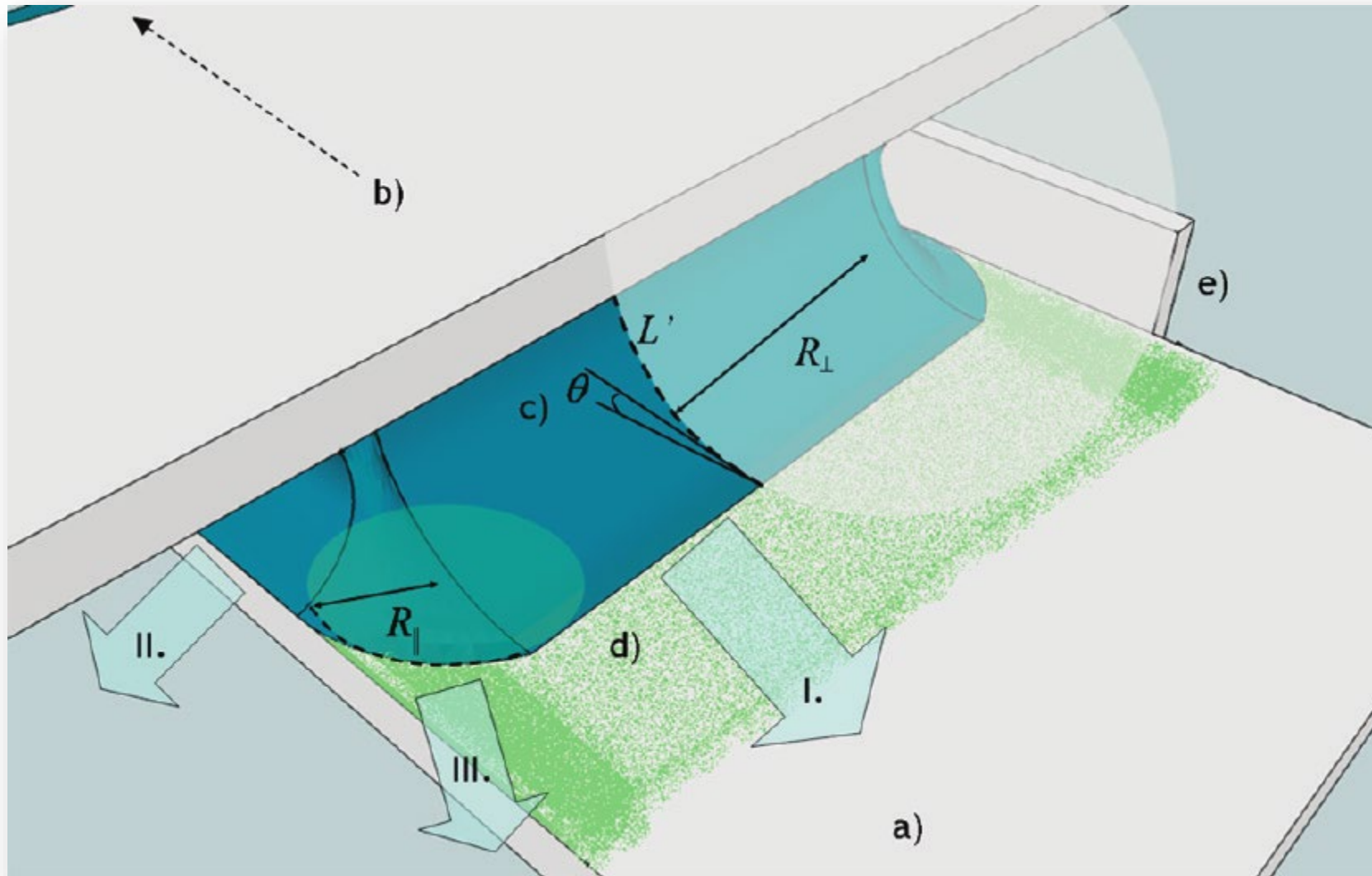
Blade coating



- ✓ Control of meniscus
- ✓ Resource efficient

From suspension to substrate

Blade coating

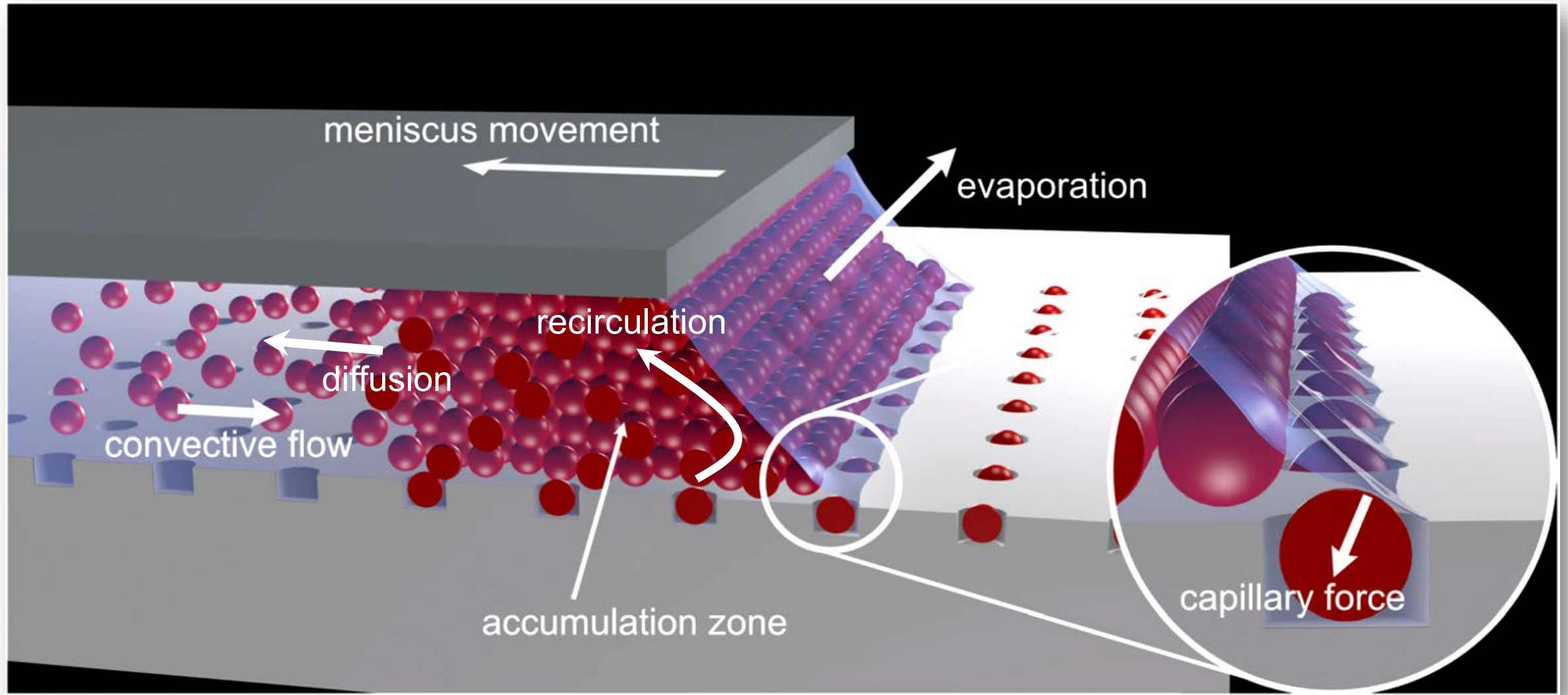


Born *et al.*, *Langmuir* 27, 8621 (2011)

From suspension to substrate to traps

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Blade coating

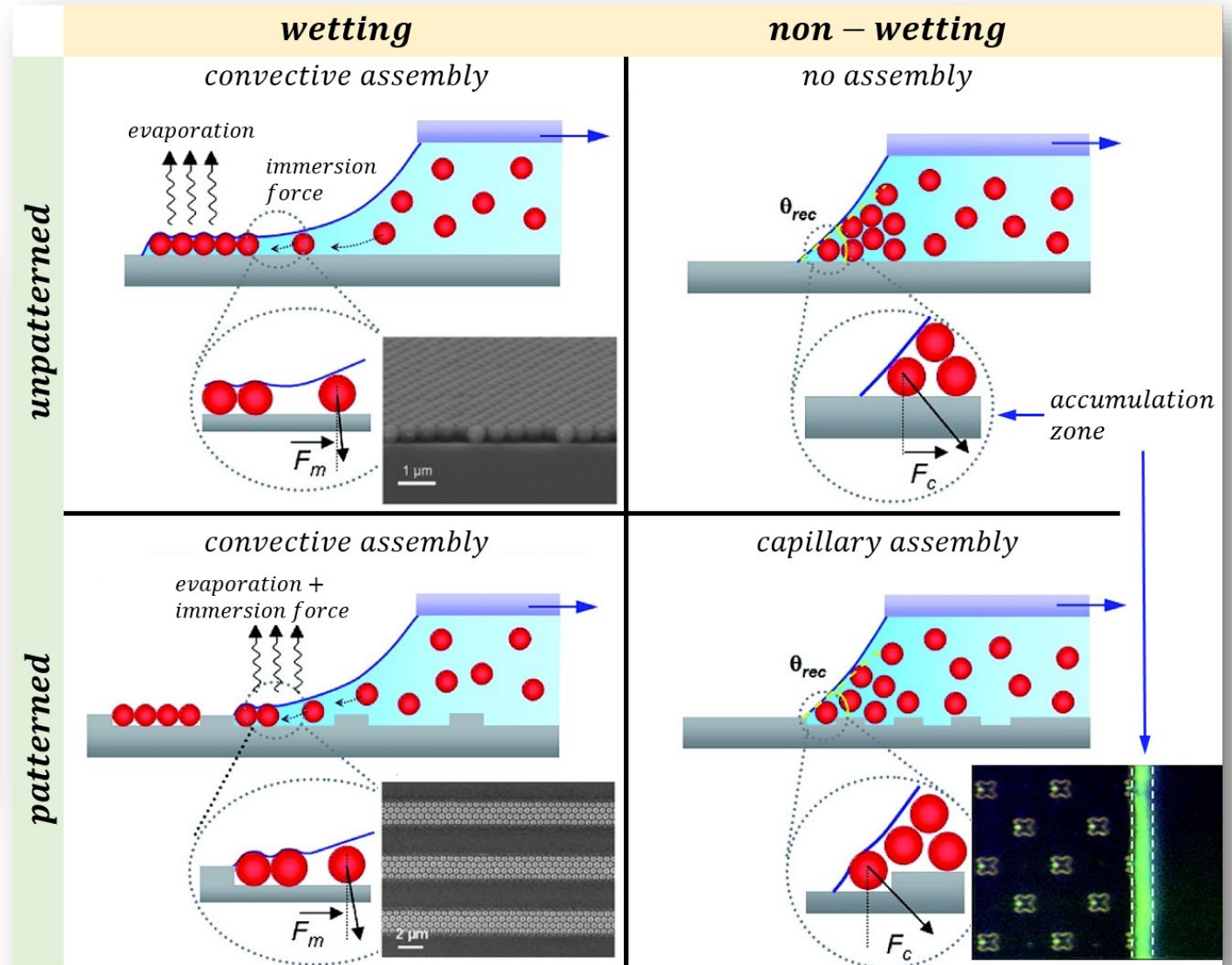
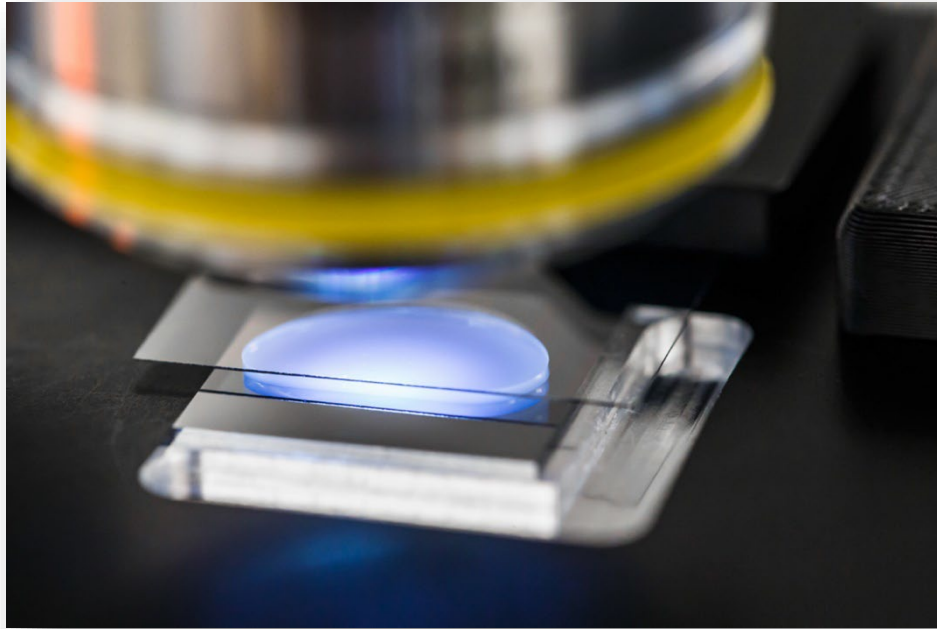


Ni et al., *Soft Matter* 2018

From suspension to substrate to traps

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Blade coating

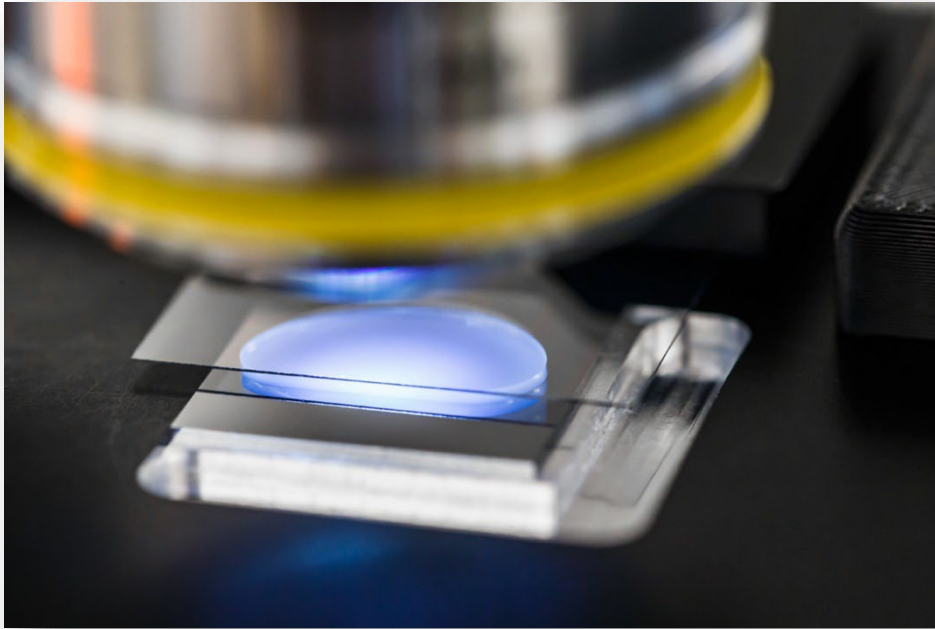


Mastrangeli, *Adv. Mater.* 2015

From suspension to substrate to traps

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Top-down meets bottom-up



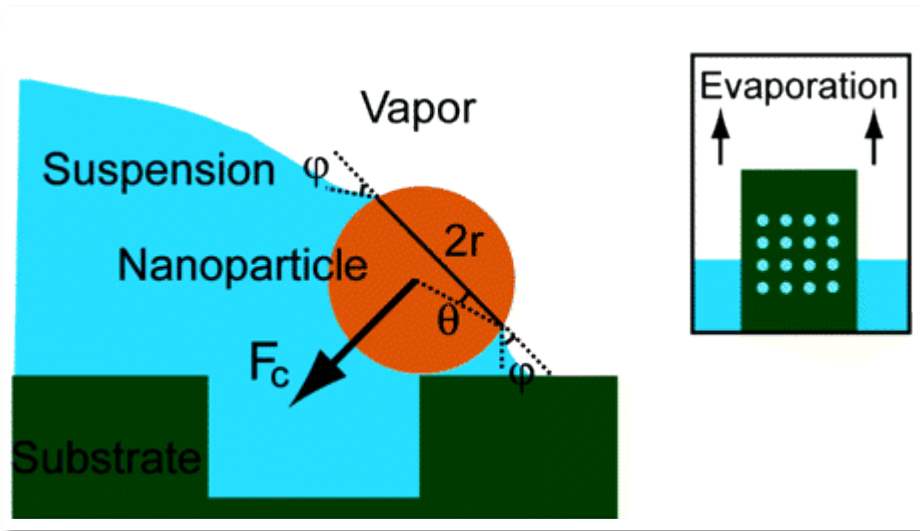
iff

- ✓ Stable colloidal suspension
- ✓ Control of substrate wetting & patterning

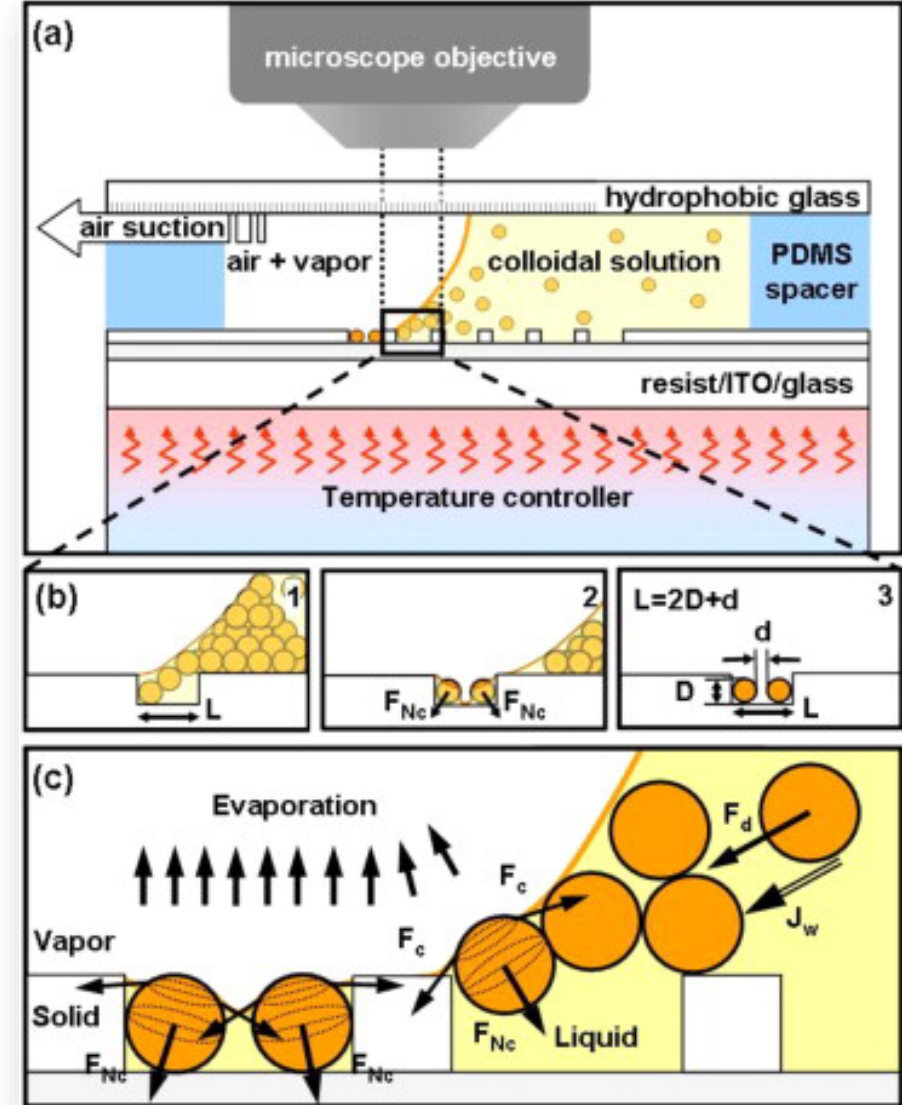
then

Capillary assembly
does not depend
on specificities
of particles or substrates

Capillary push



Cui *et al.*, *Nano Lett.* 4, 1093 (2004)

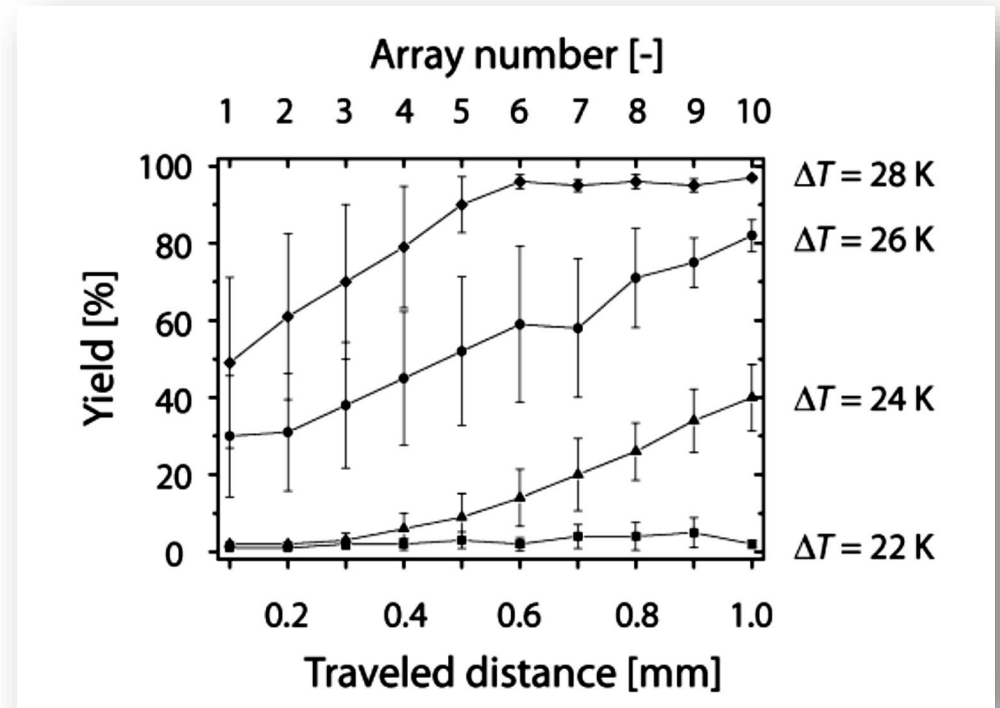
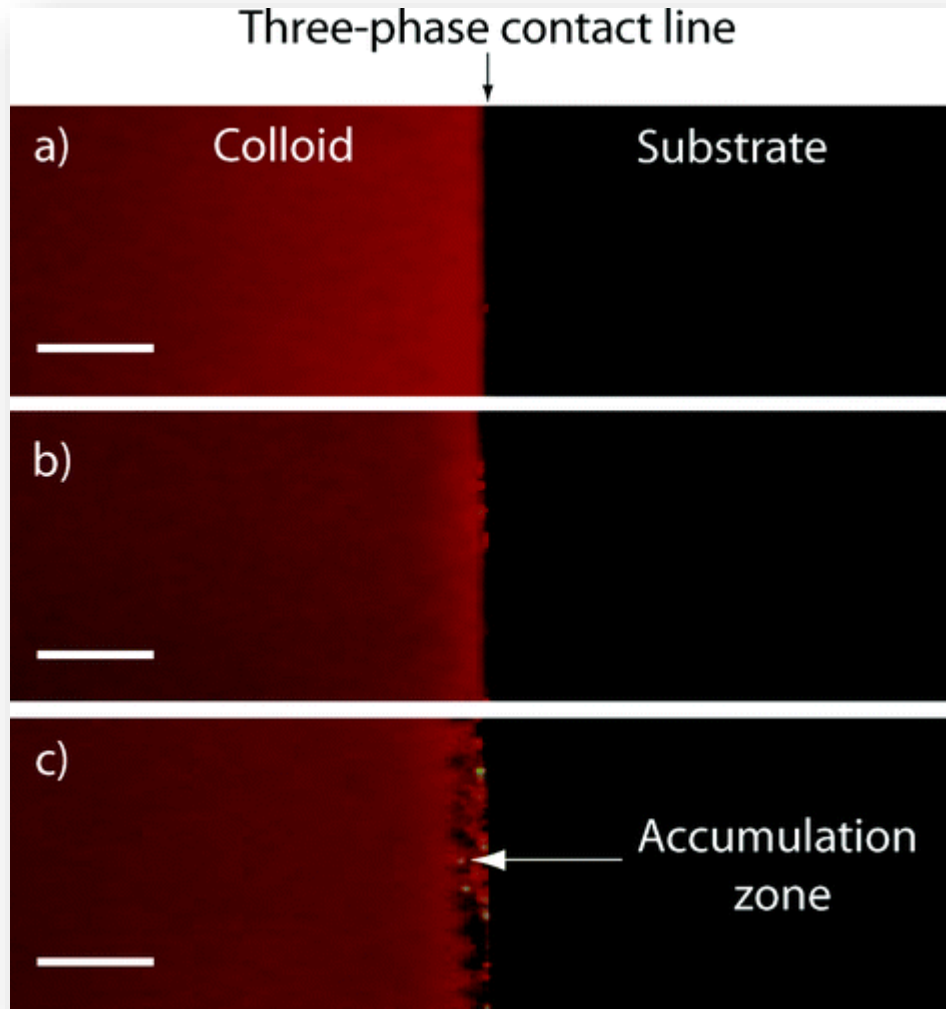


Pinedo Rivera *et al.*, *Microelectron. Eng.* 86, 1089 (2009)

Prior wisdom

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Size of accumulation zone

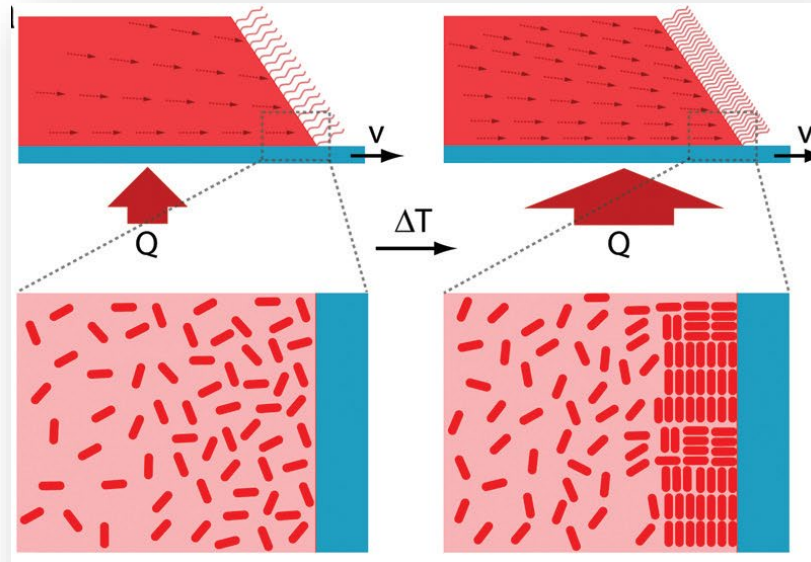


Kuemin *et al.*, *Langmuir* 27, 6305 (2011)

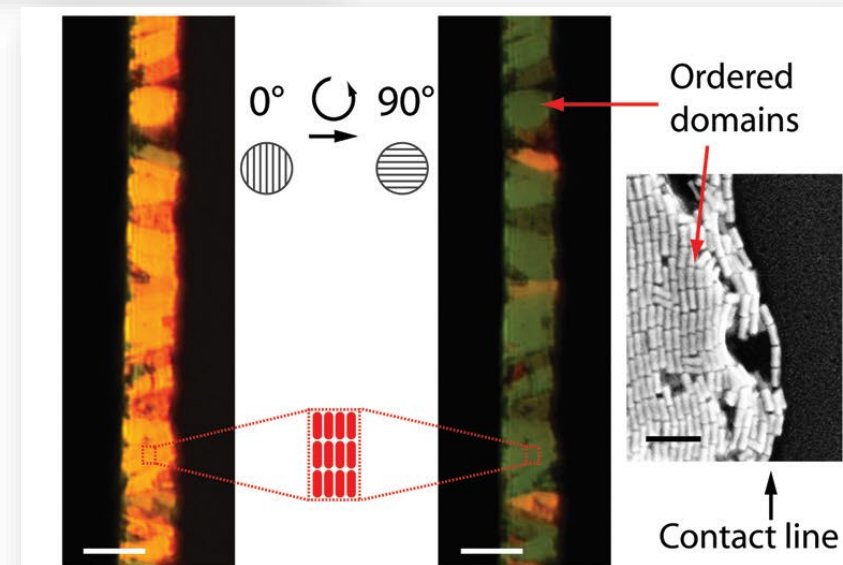
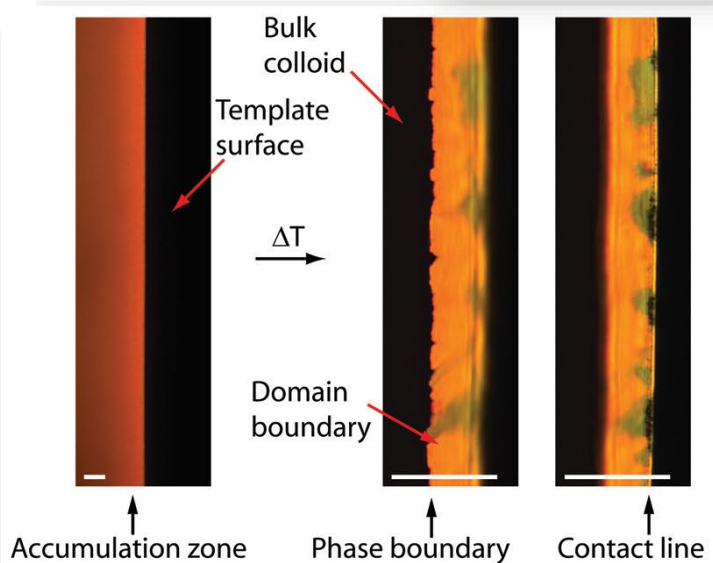
Prior wisdom

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Density-driven phase transition



Kuemin *et al.*,
Adv. Funct. Mater. 22, 702 (2012)



Prior wisdom

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Topographical template fabrication

EBL / RIE / Silanisation

- Features $30\text{nm} \pm 5\text{nm}$
- Multilayer processes
- Chlorine-based RIE
- water CA: $\sim 110^\circ$



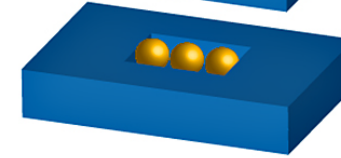
E-Beam lithography



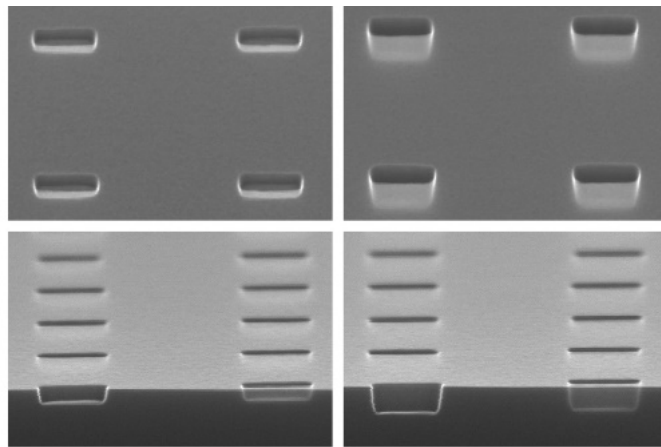
Silicon/Glass RIE



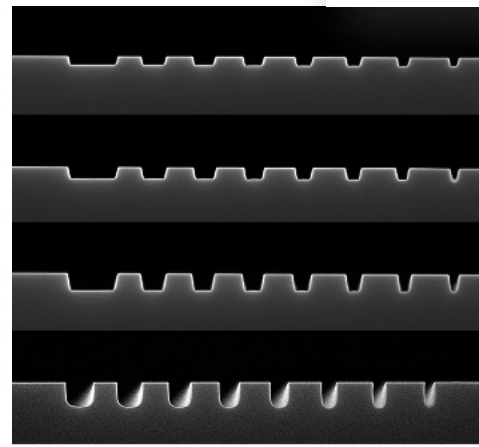
Surface silanization



Gold NP assembly



200 nm



200 nm



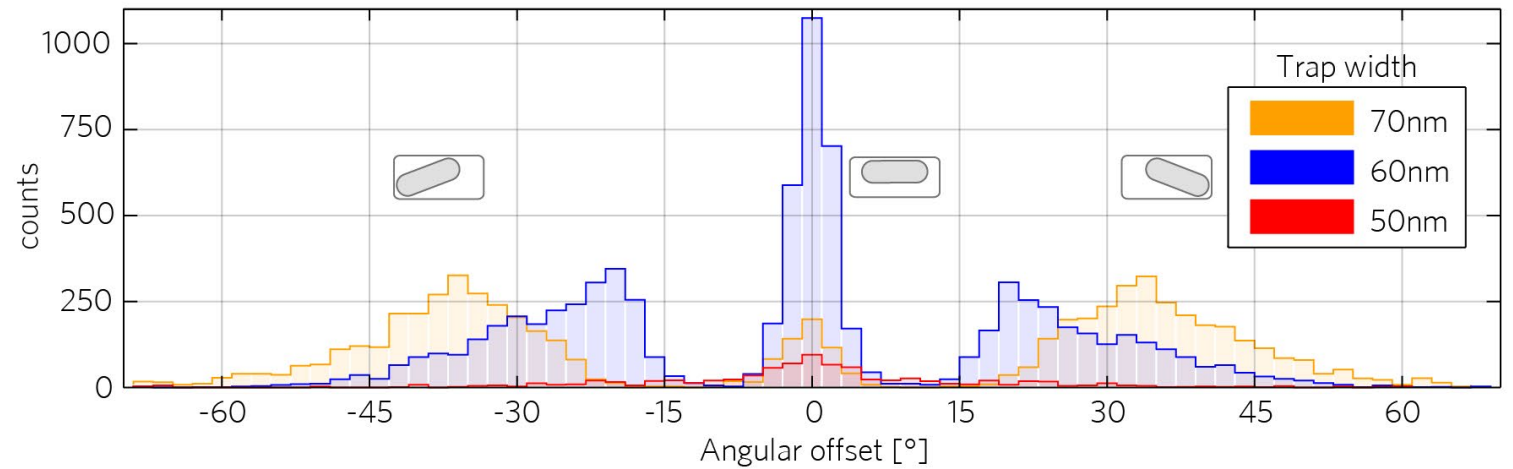
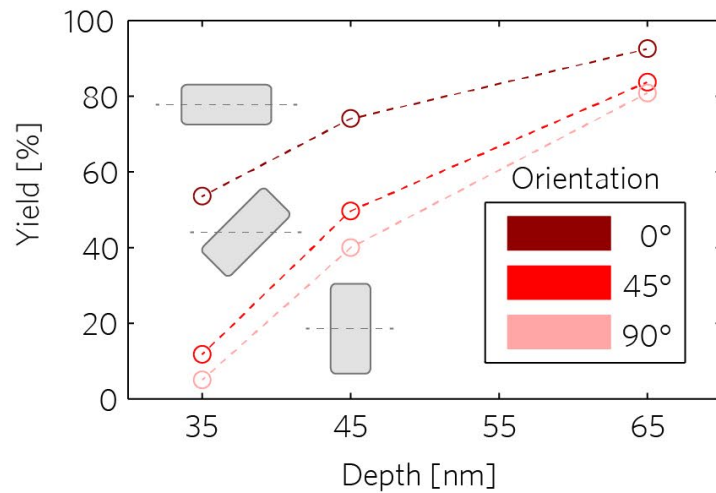
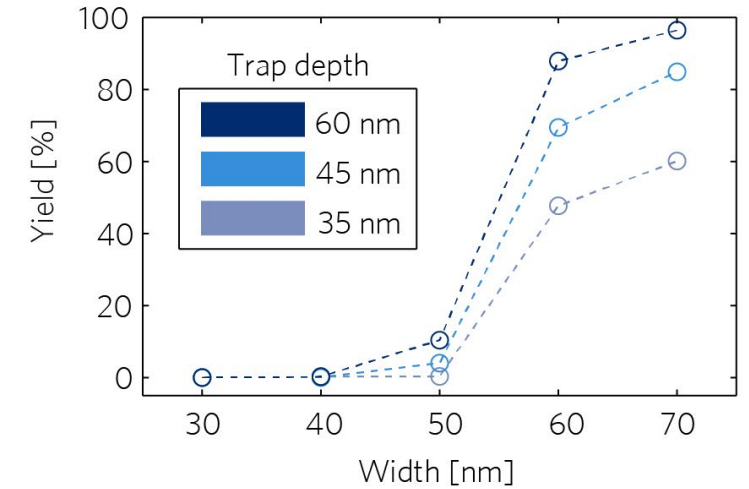
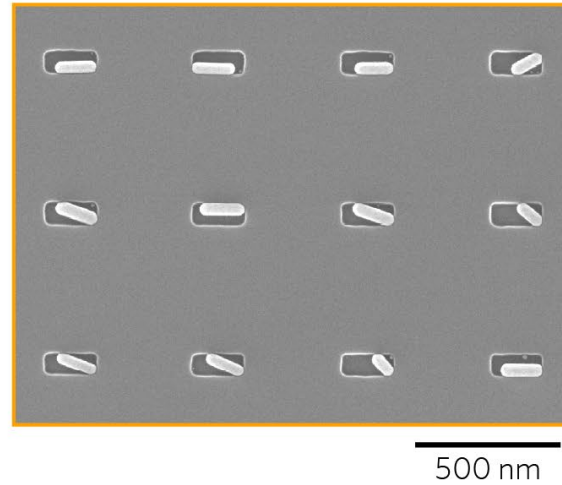
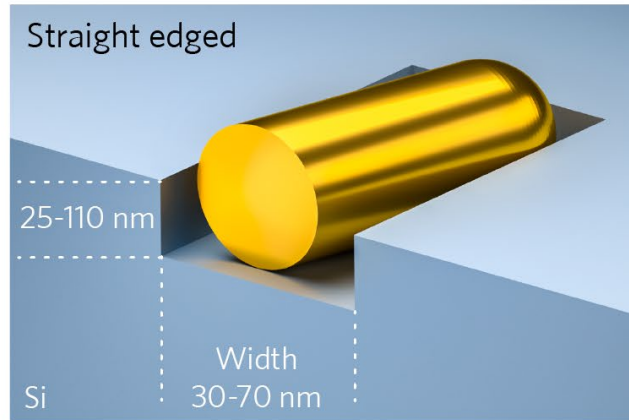
20 μm

Flauraud *et al.*, *Nat. Nanotechnol.* 2016

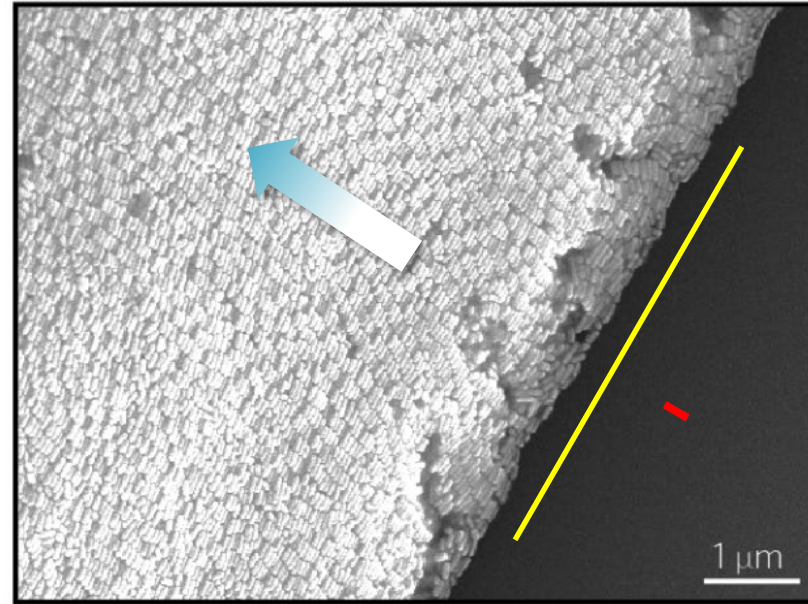
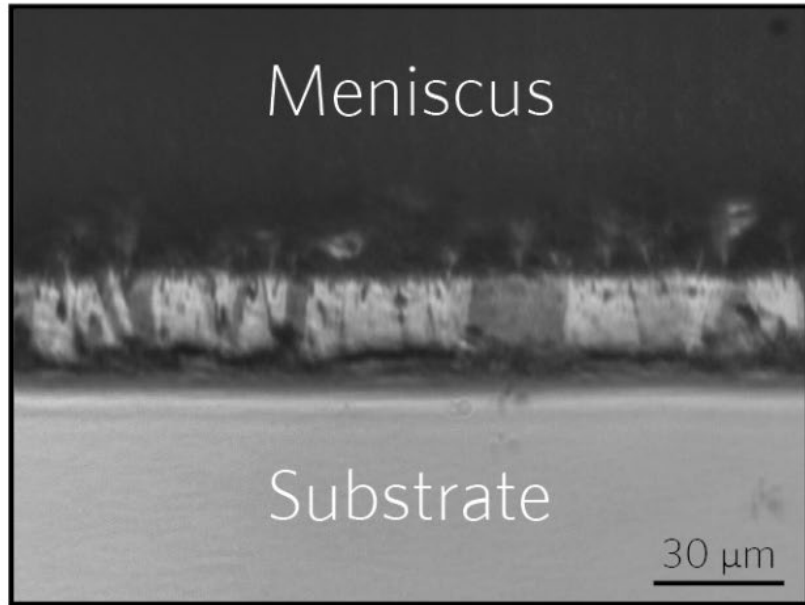
Fabrication

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Straight-edged traps



Particle accumulation and entropy



L. Onsager

Density-induced phase transition

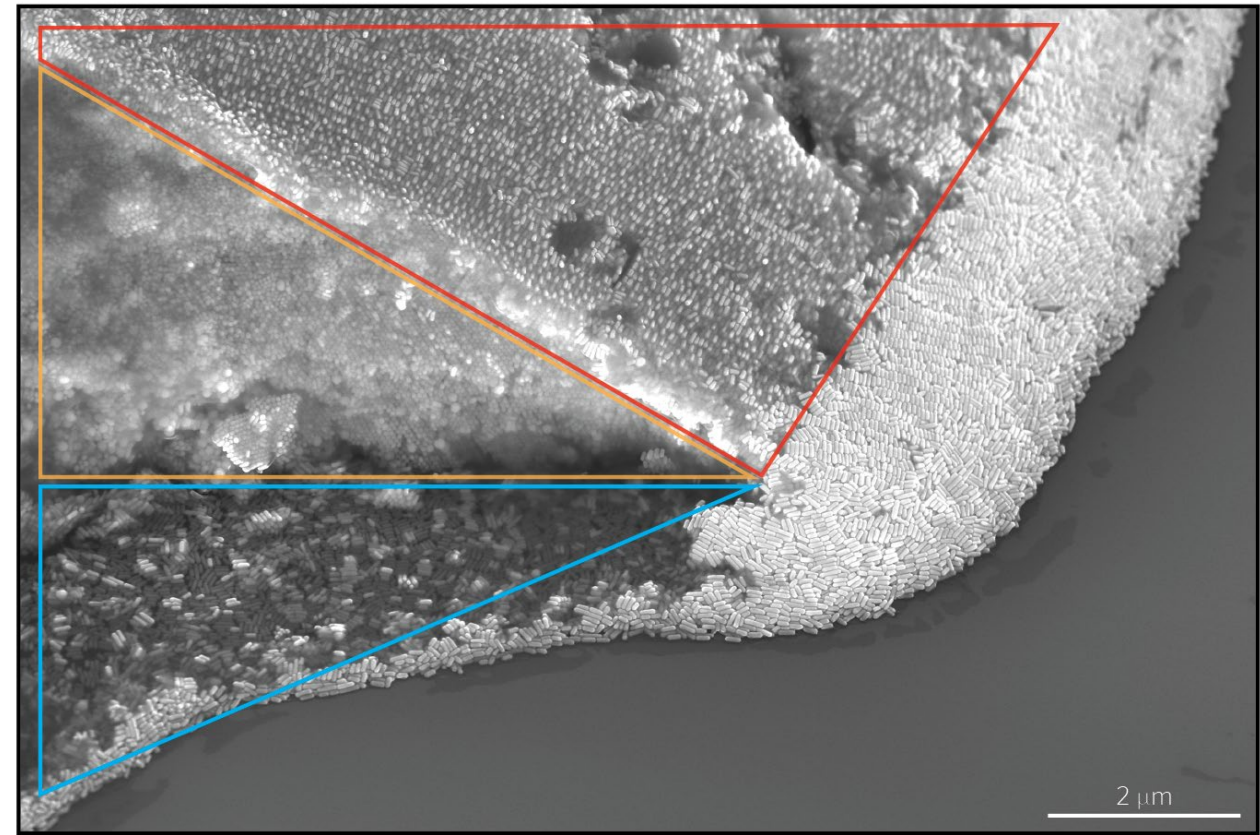
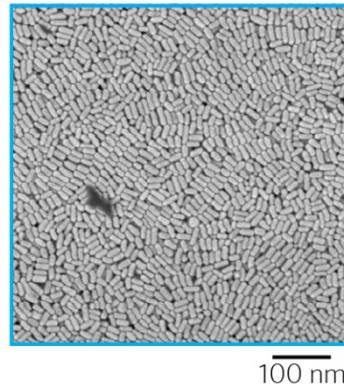
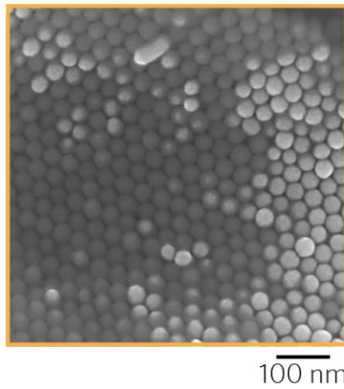
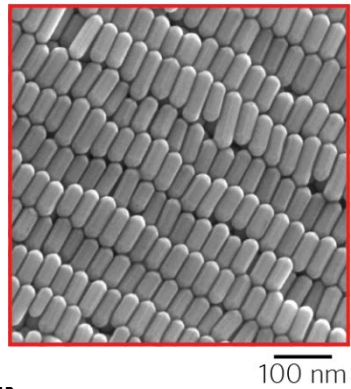
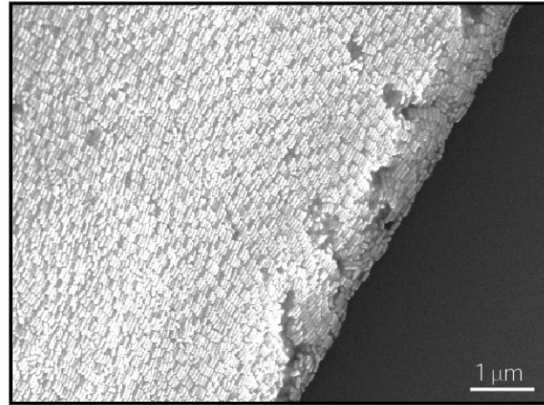
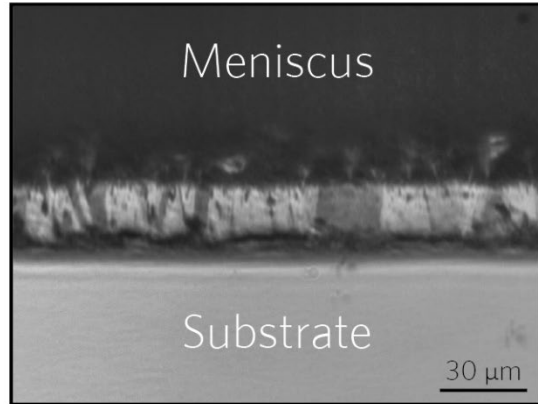
- Rotational entropy traded for translational entropy
- Net entropy gain

Flauraud *et al.*, *Nat. Nanotechnol.* 12, 73 (2017)

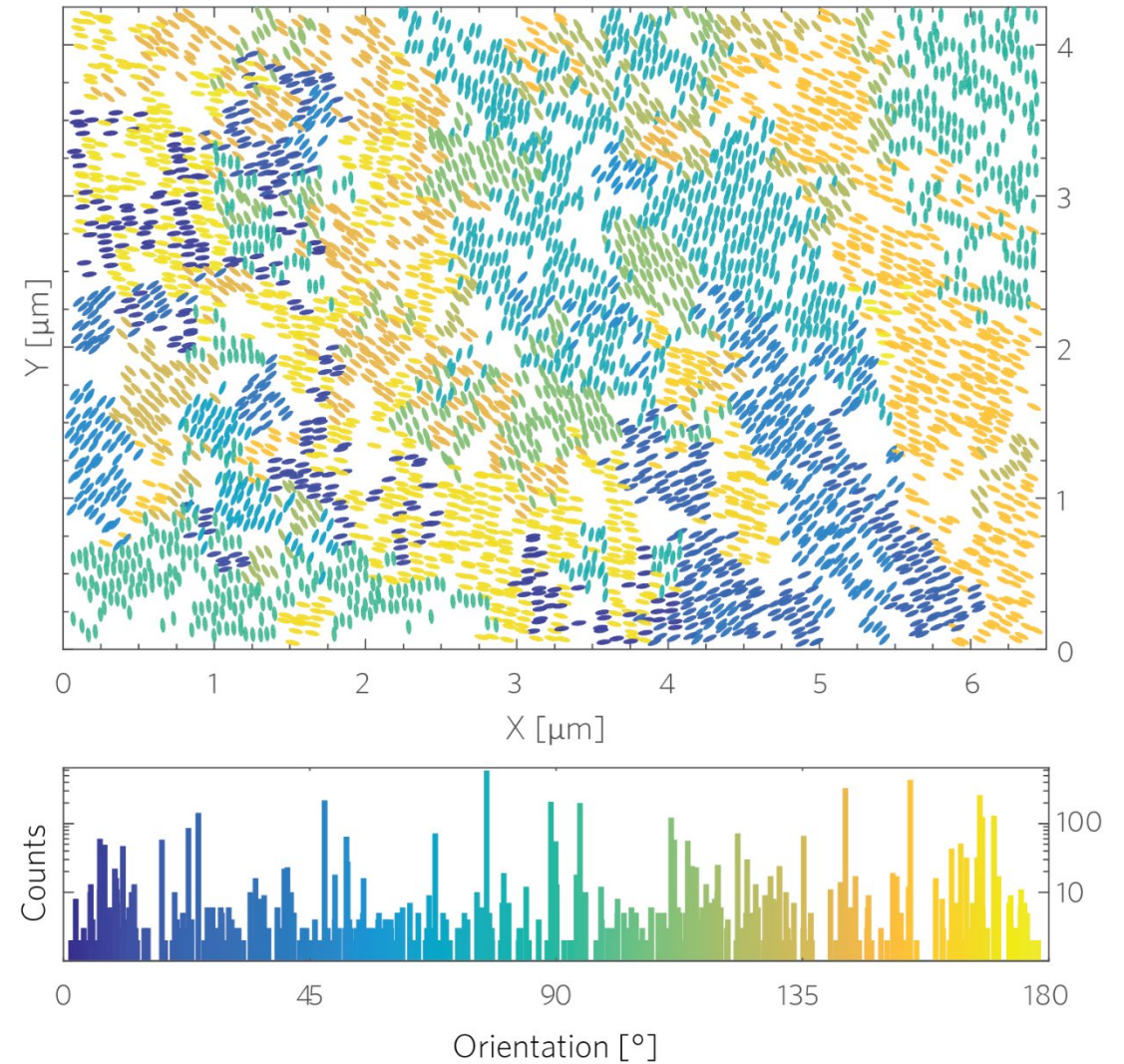
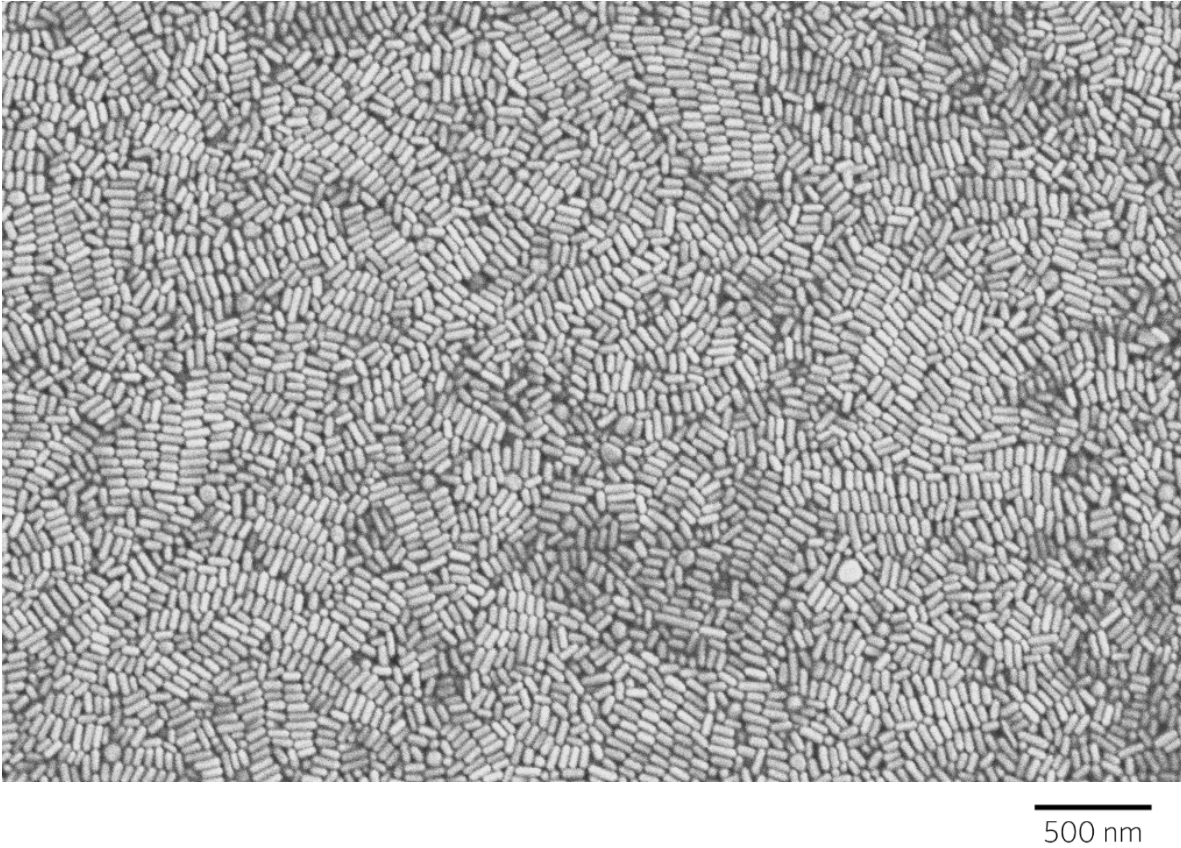
What would Lars think?

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Insight 1: Non-uniform accumulation



Insight 1: Non-uniform accumulation

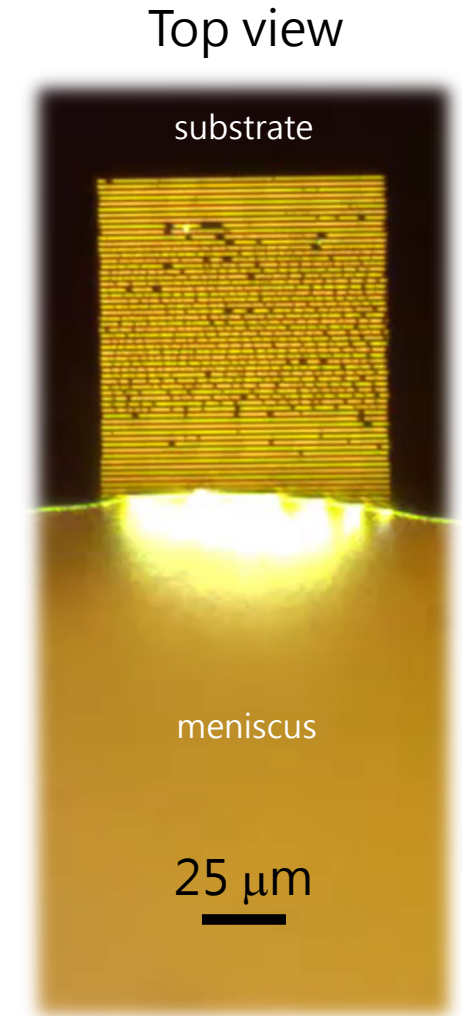
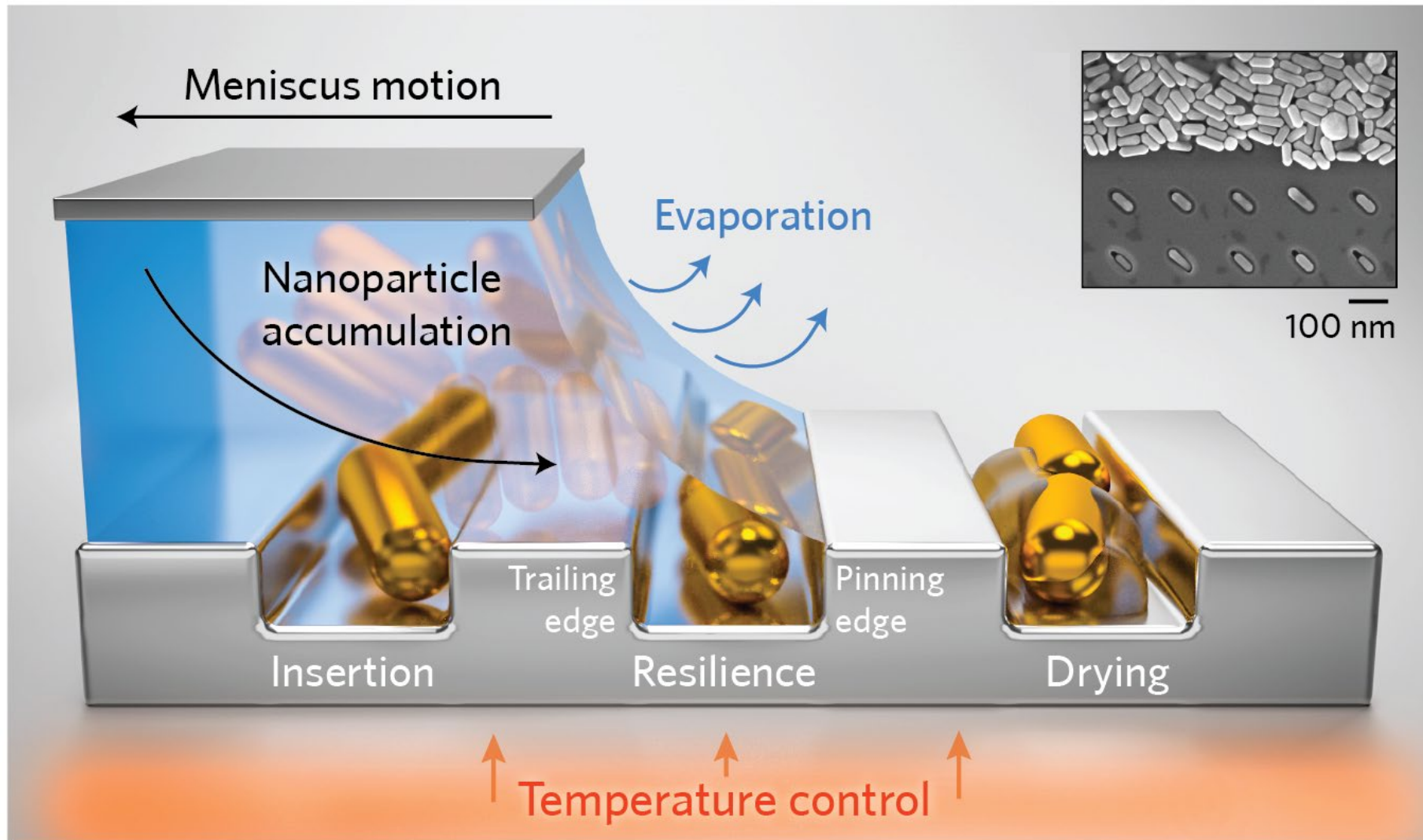


Flauraud *et al.*, *Nat. Nanotechnol.* 12, 73 (2017)

New insights

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Insight 2: Stages of capillary assembly



Flauraud *et al.*, *Nat. Nanotechnol.* 12, 73 (2017)

New insights

Single nanoparticle printing | M. Mastrangeli, PhD – EPFL, 26 August 2022

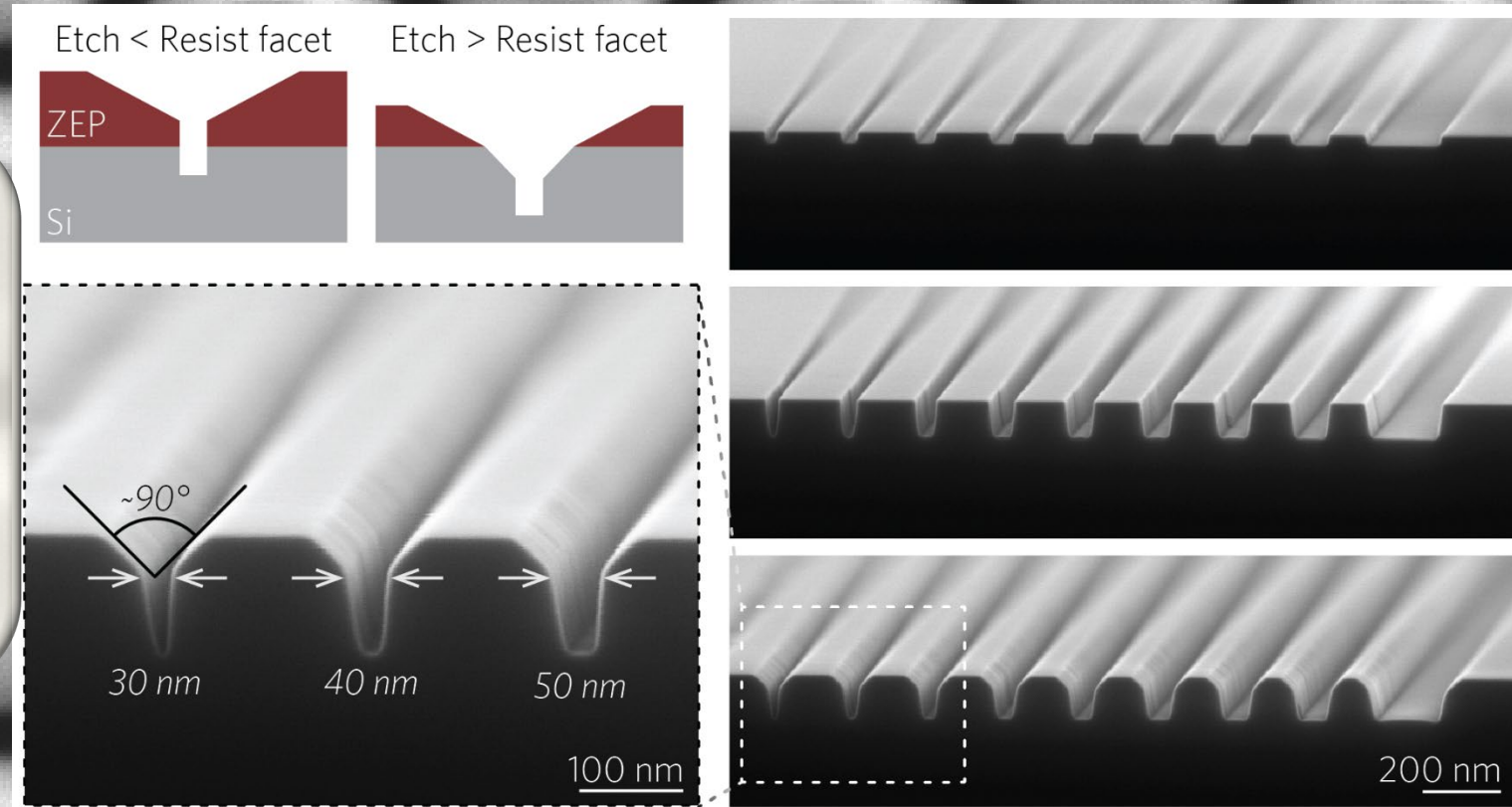
Limitations of straight-edged traps

- Trap cross-section needs to accommodate effective NPs size...
- ...But dry NPs are smaller
- Loose positional control

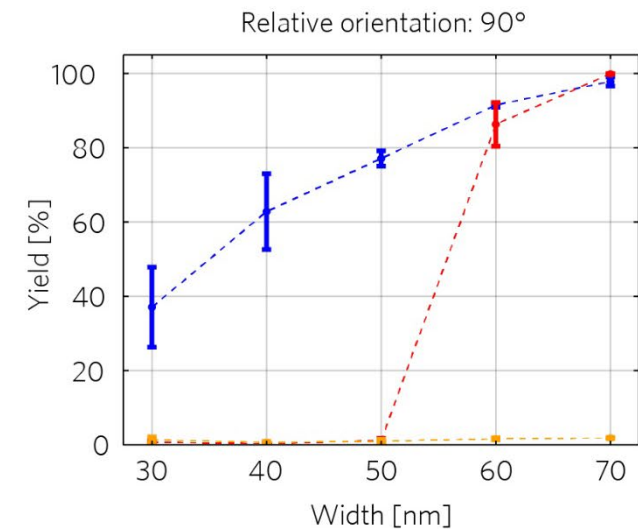
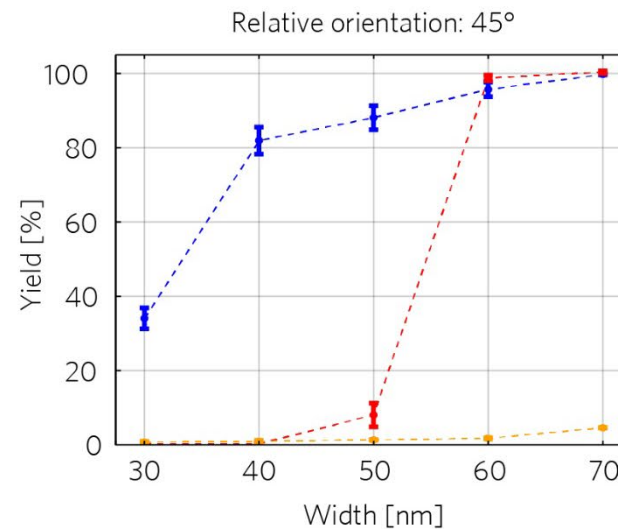
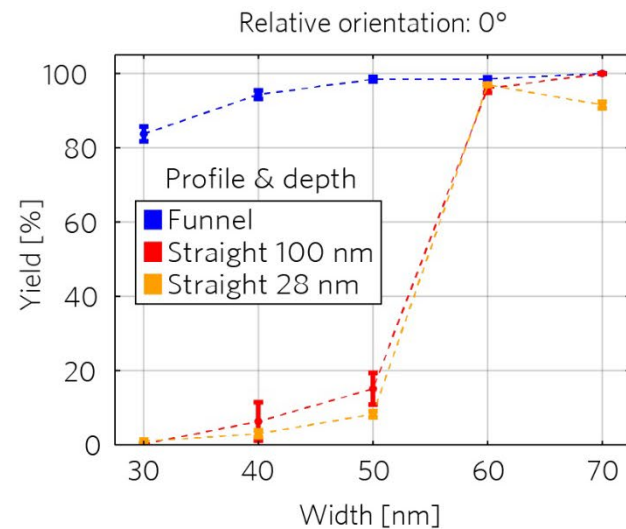
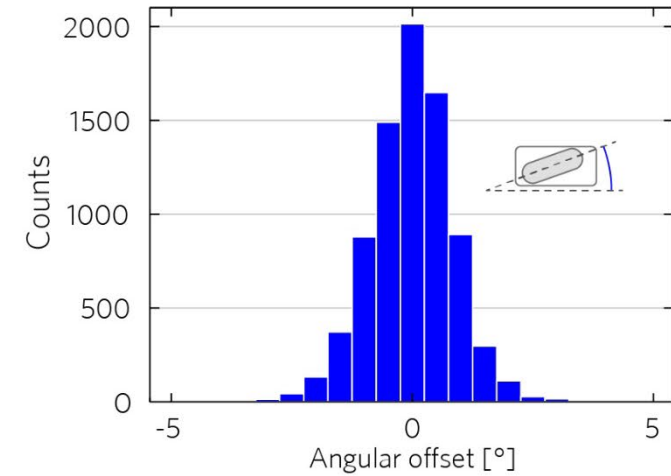
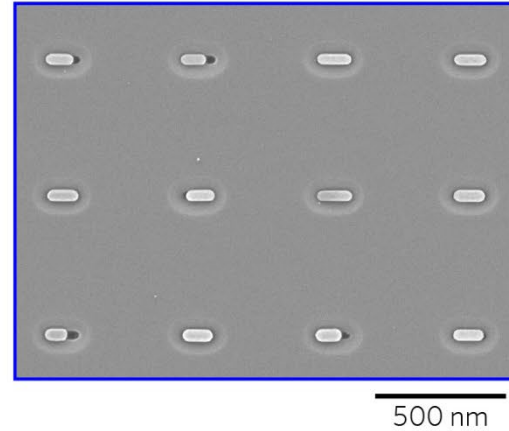
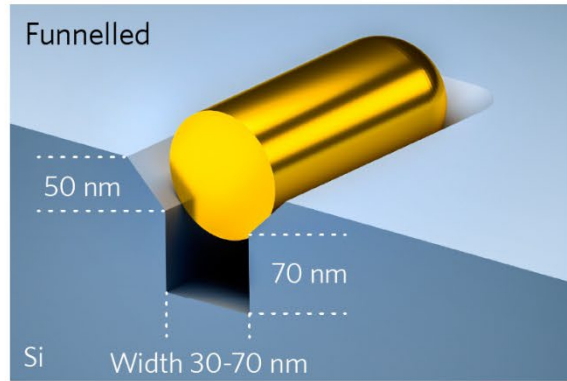
Insight 3: 3D trap geometry

EBL – RIE – Silanisation

- Cl_2/BCl_3 chemistry
- Progressive resist faceting
- Low roughness transfer to Si
- Compatible with SiO_2 & Si_3N_4



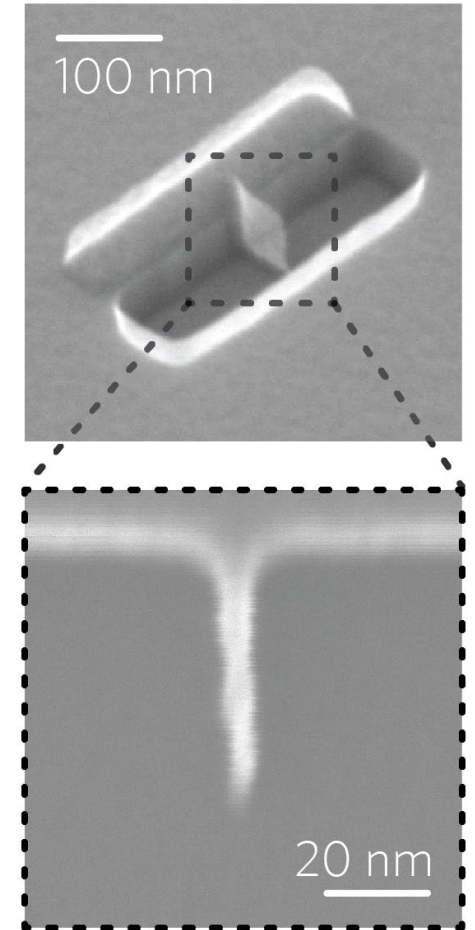
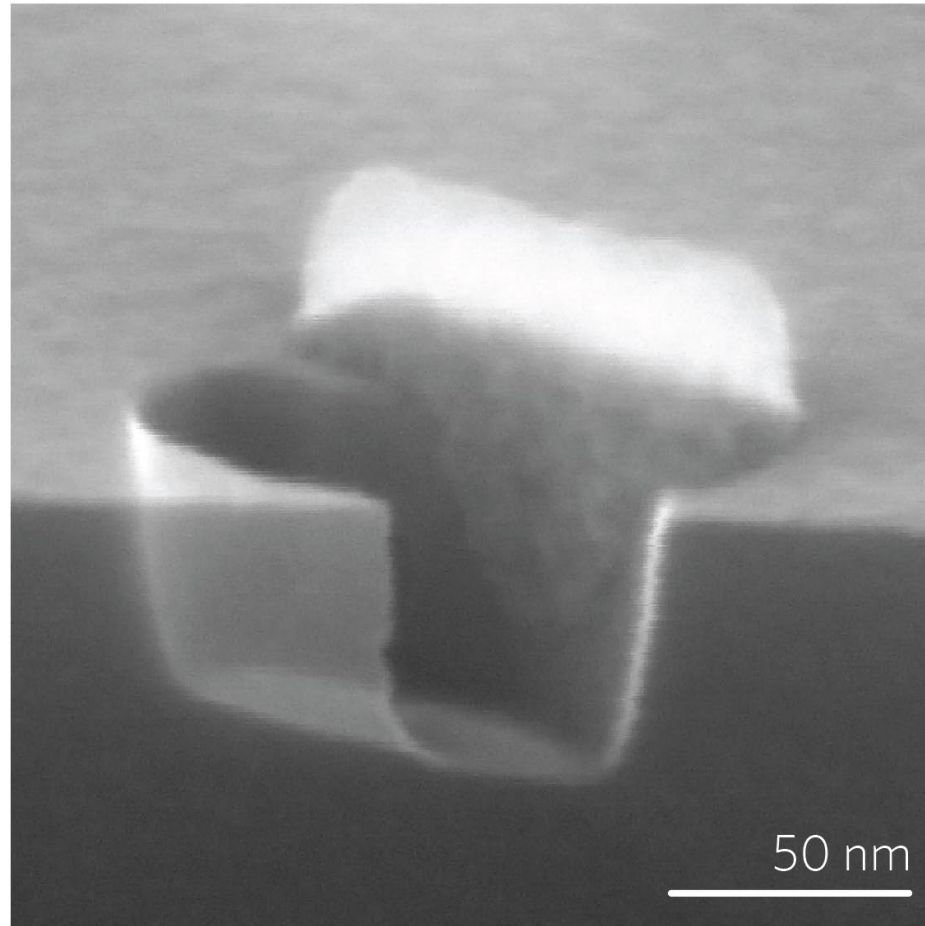
Assembly on funneled traps



Single auxiliary sidewall

Auxiliary sidewall

- HSQ (SiO_x) EBL resist
- Alignment < 10 nm
- Counteract NP removal
- Symmetry breaking



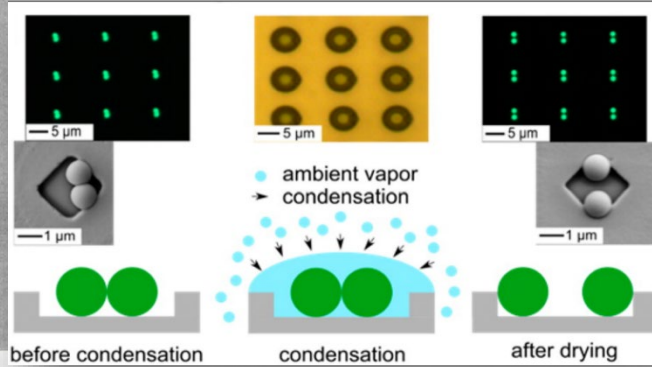
Flauraud *et al.*, *Nat. Nanotechnol.* 12, 73 (2017)

Capillary assembly of nanoparticles

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Straight-edged traps with auxiliary sidewall

Ni *et al.*, Langmuir 2014

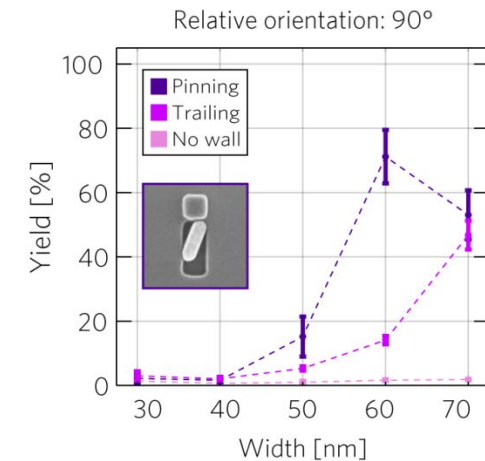
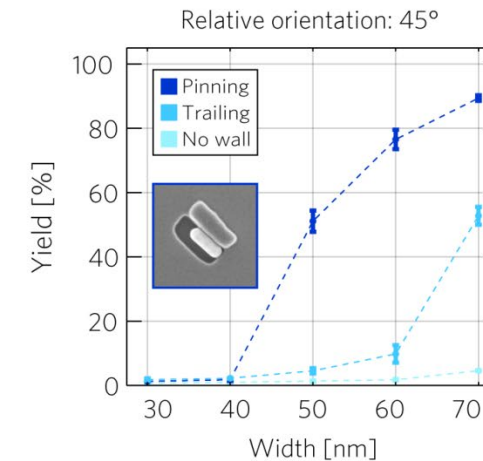
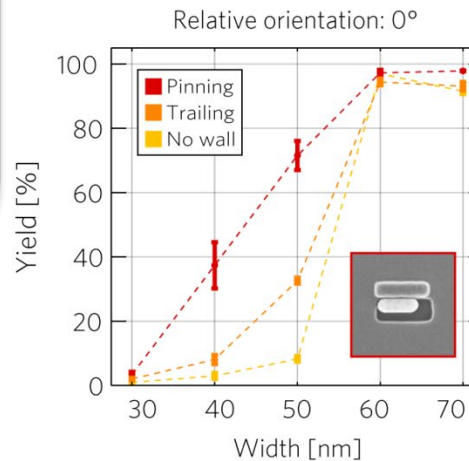
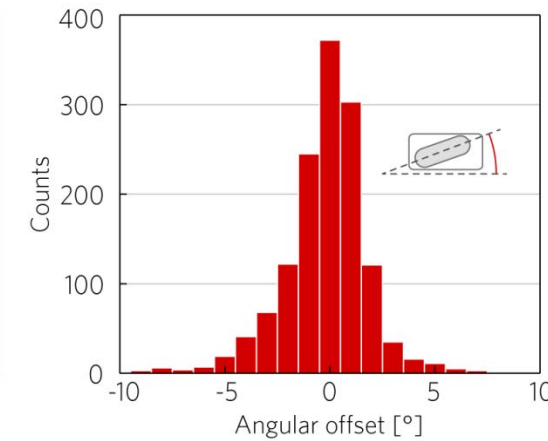
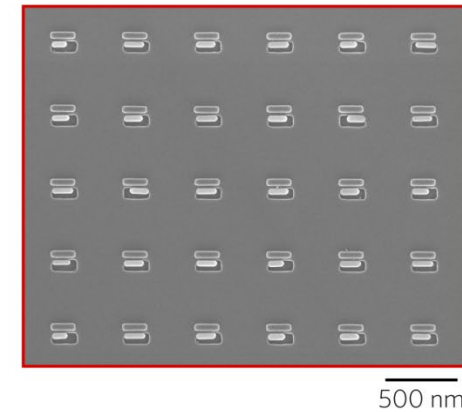
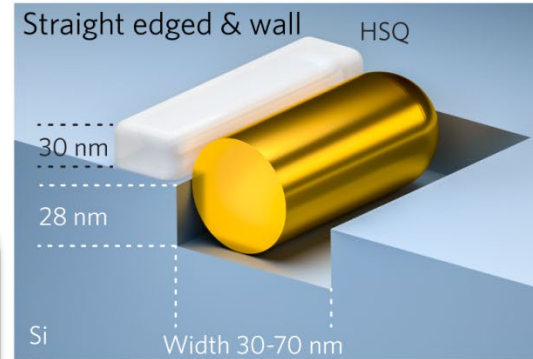


Biased evaporation

- Deterministic positioning in loose traps

Sidewall position

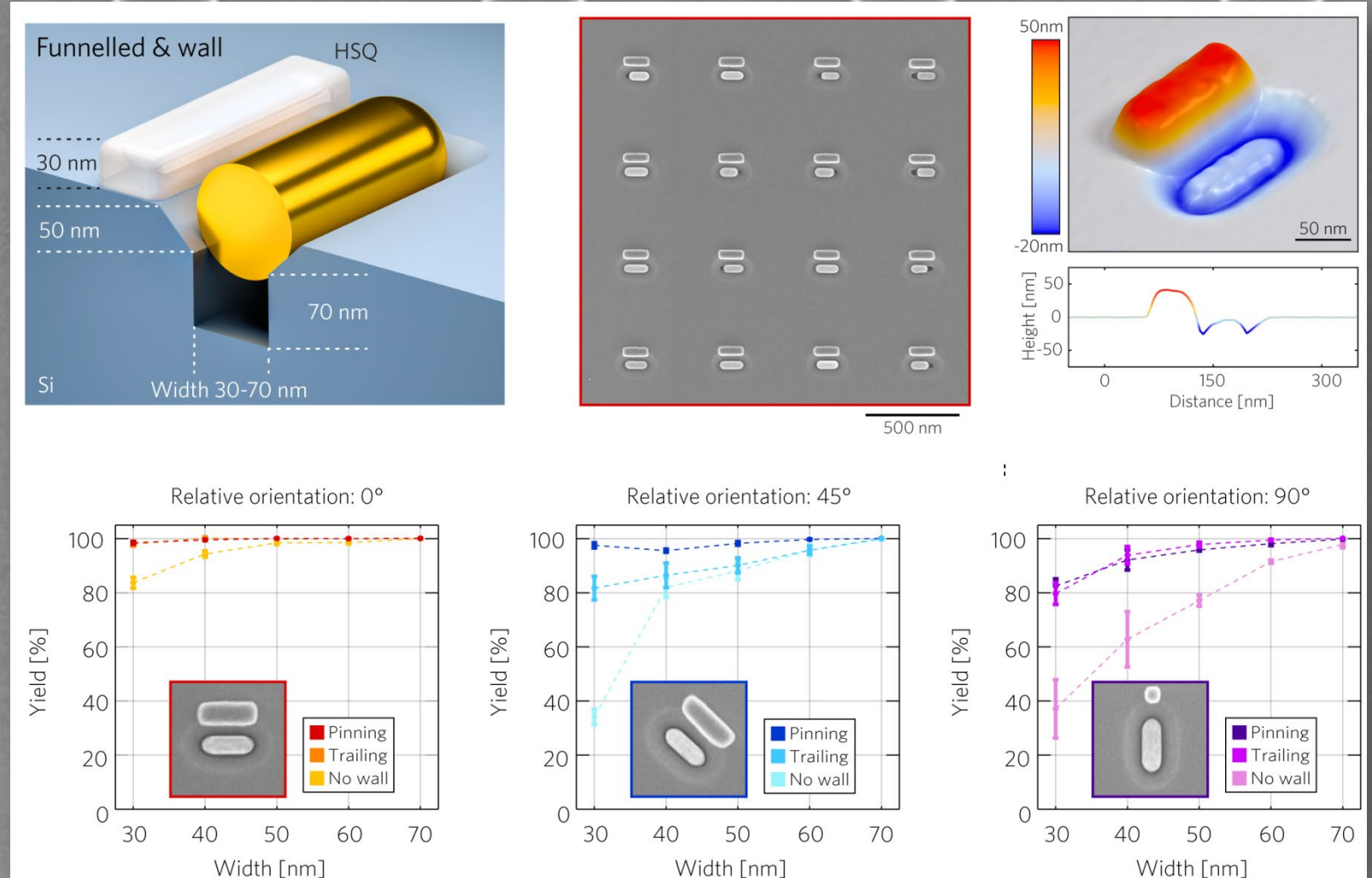
- High yield improvement



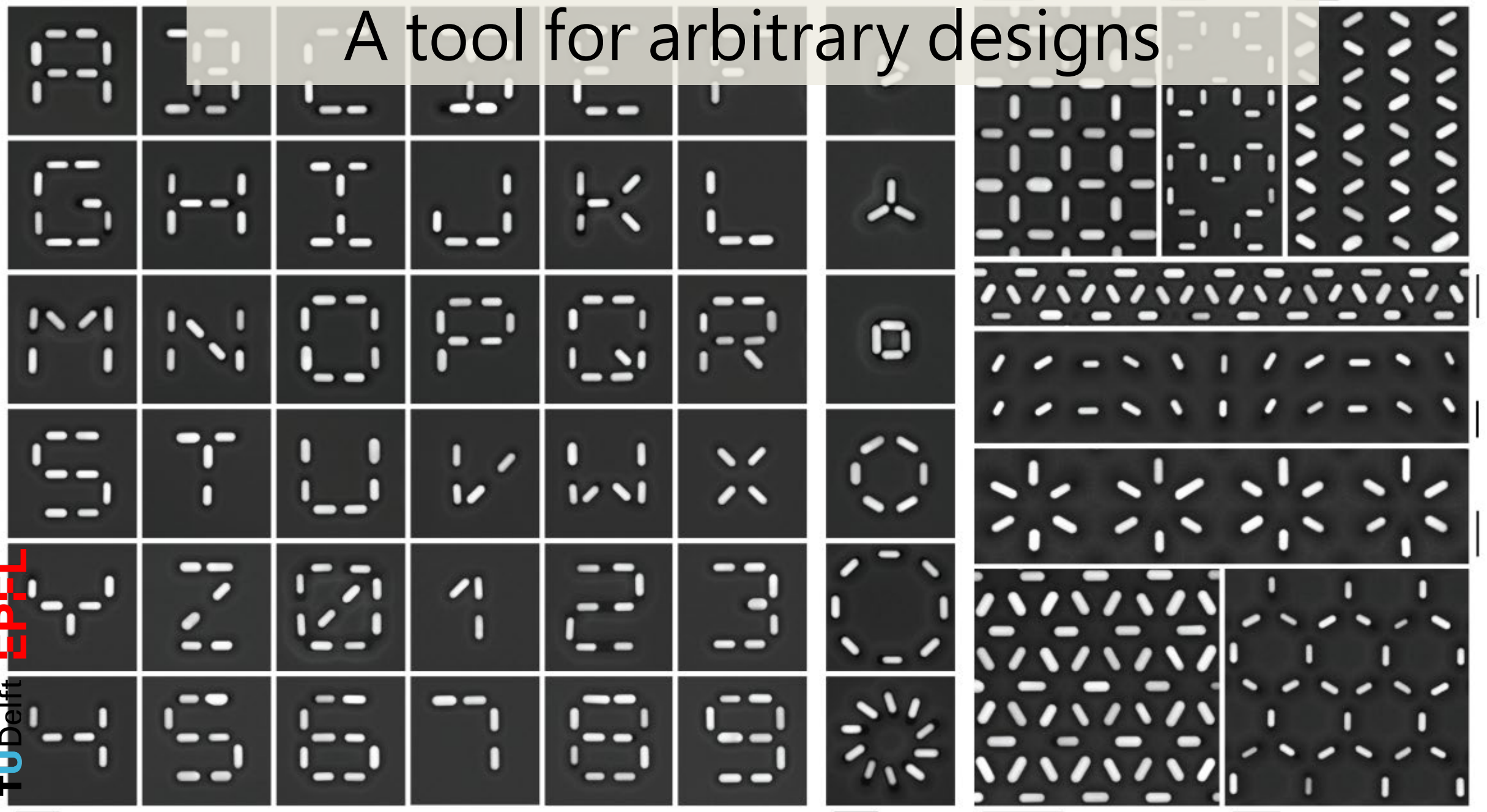
The ideal trap

Exploits all three stages of assembly

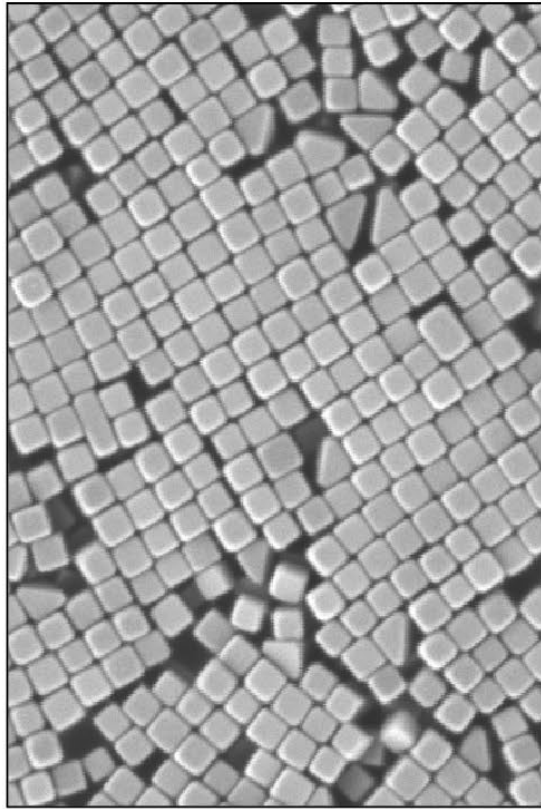
- Up to 100% yield for all orientations
- Absolute positioning control



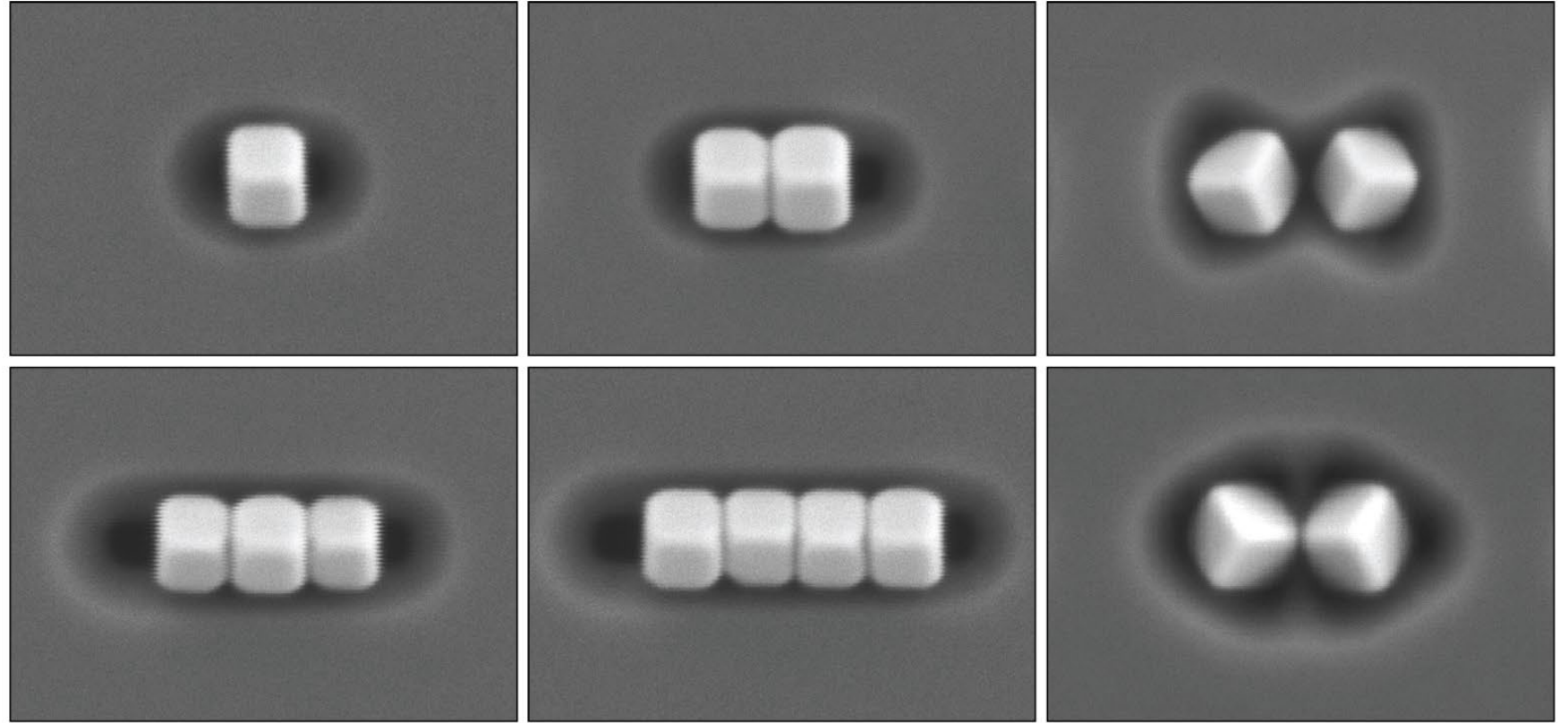
A tool for arbitrary designs



3D orientation of Ag nanocubes



400 nm



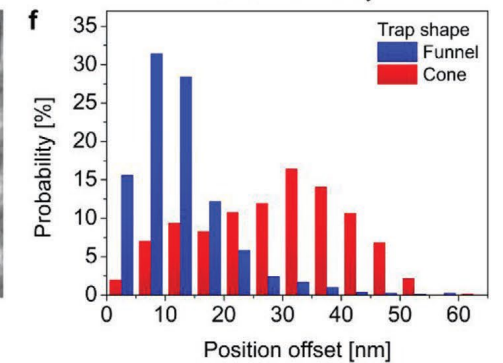
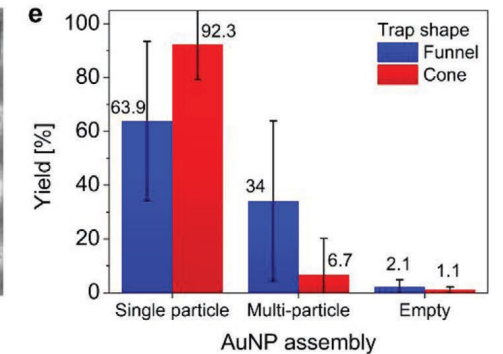
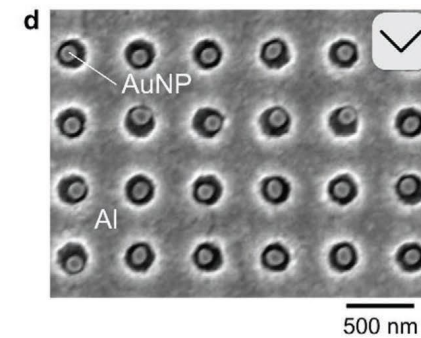
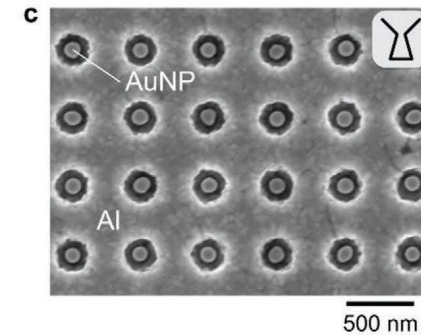
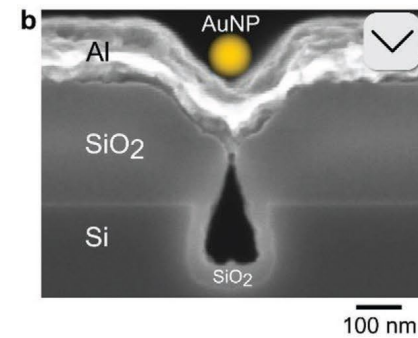
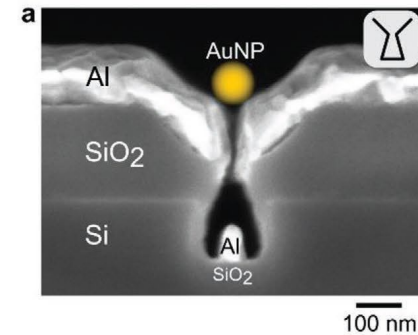
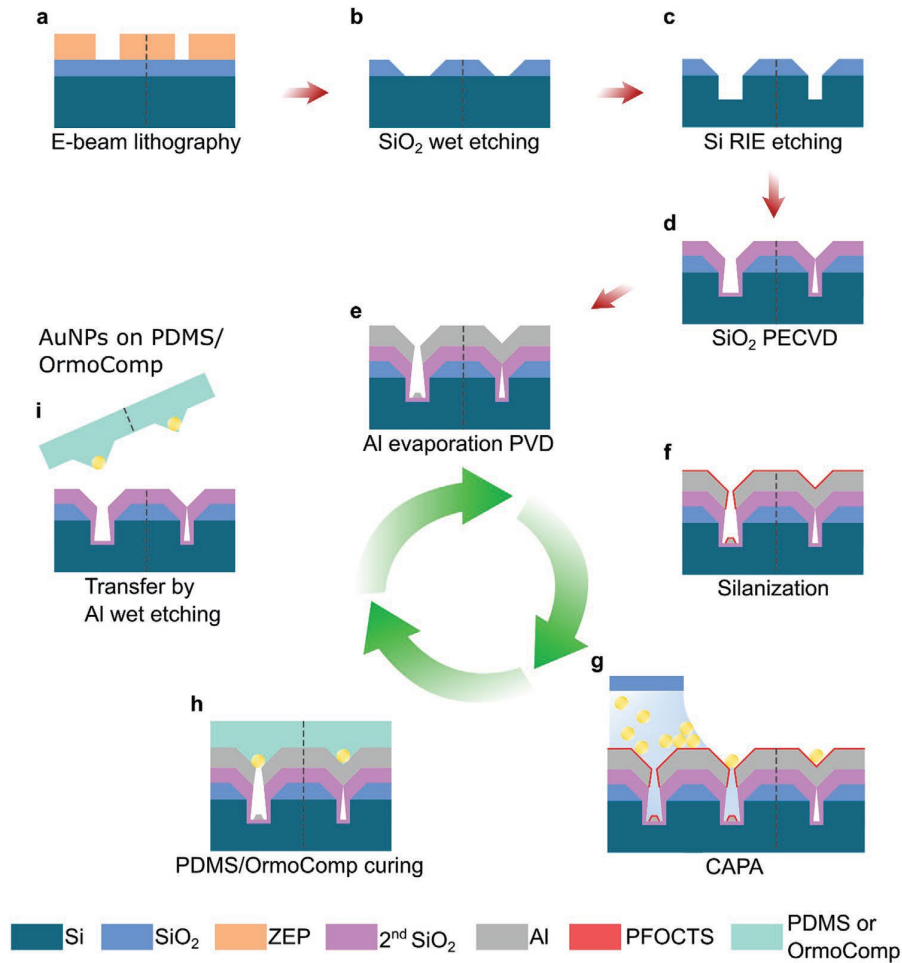
200 nm

Flauraud *et al.*, *Nat. Nanotechnol.* 12, 73 (2017)

3D orientation of anisotropic nanoparticles

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Funneled traps in reusable template



Yu et al., Part. Part. Syst. Charact. 39, 2100288 (2022)

3D orientation of anisotropic nanoparticles

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Updated wisdom

1. Contact line

- Boundary
- Drags particles across substrate
- Last push

2. Accumulation

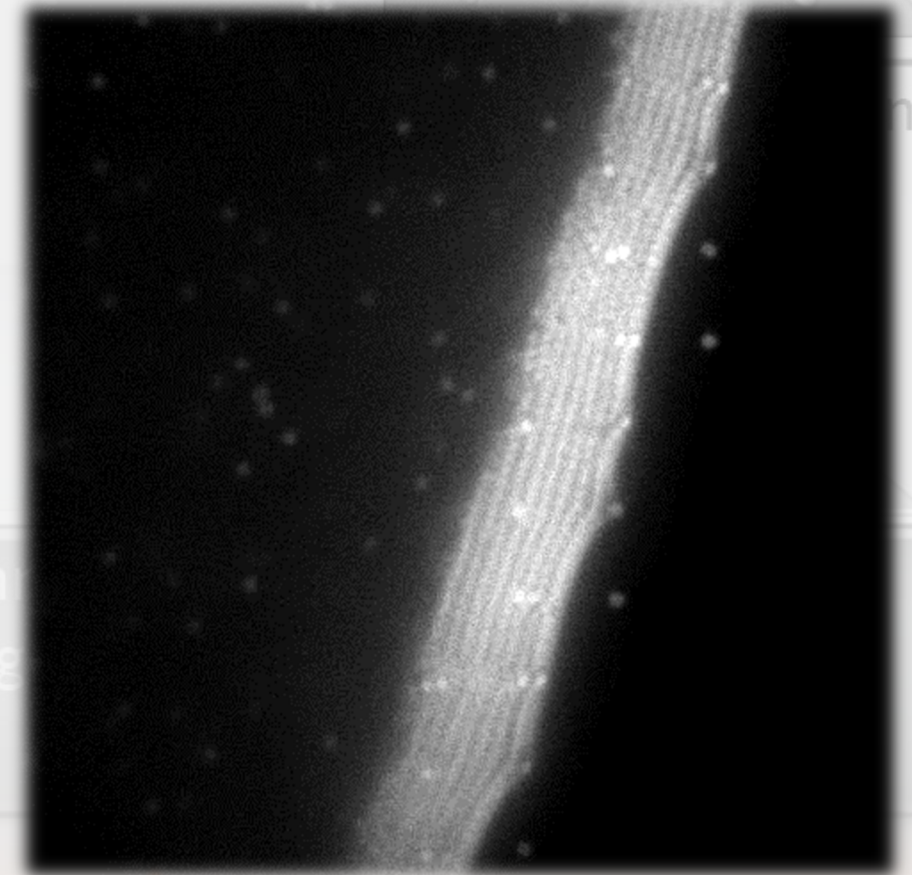
- Width facilitates injection
- Confinement dependent

3. Particle trajectory

- Advection, immobilization, drag
- Injection: pressure, entropy

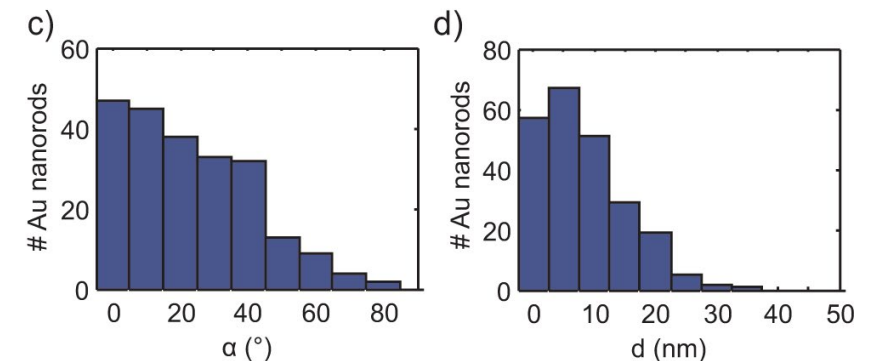
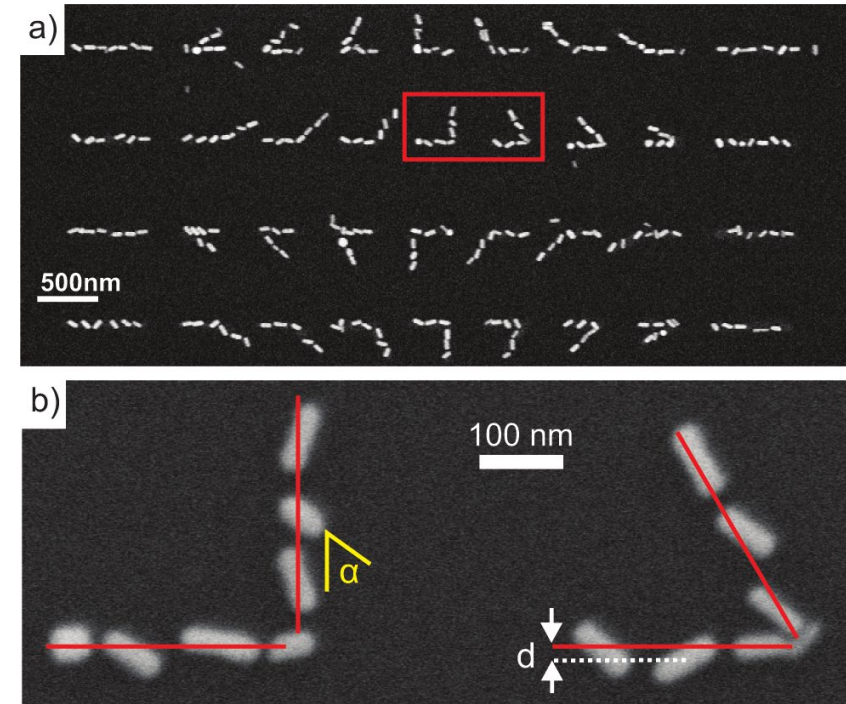
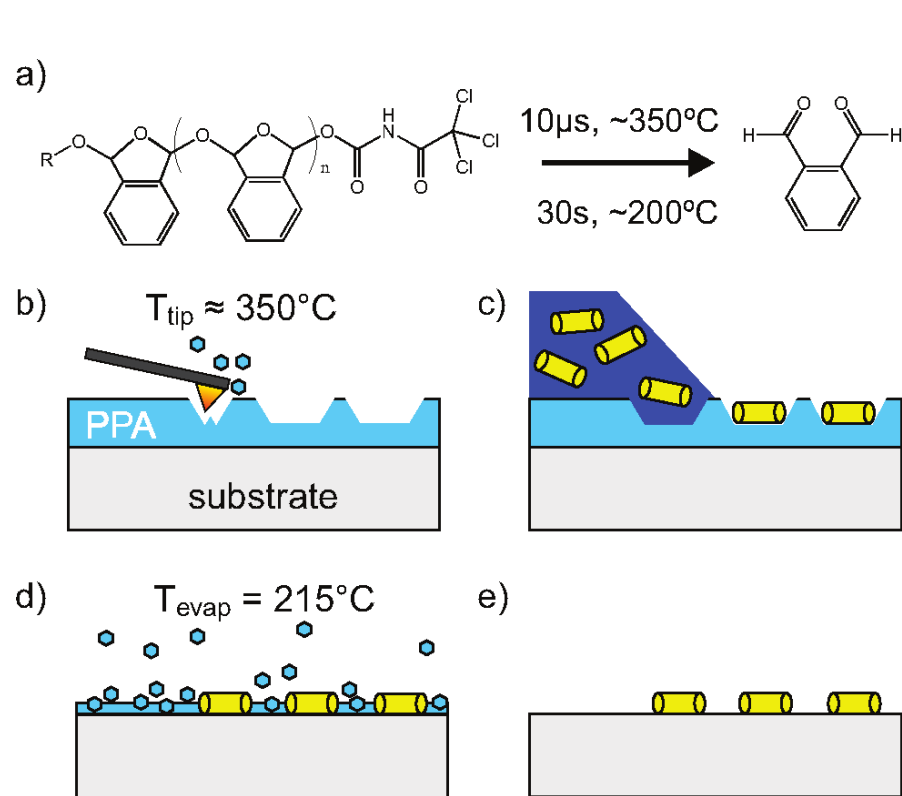
4. Assembly stages

- All impact assembly yield
- Tailored trap geometry



Ni *et al.*, *Farad. Discuss.* 181, 225 (2015)

Trap design: t-SPL-based sculpting

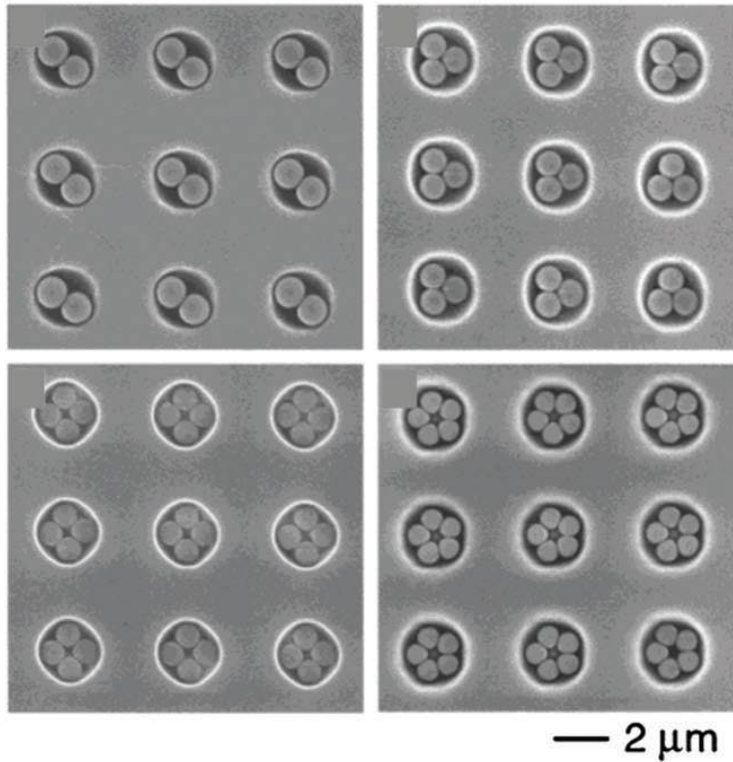


Holzner *et al.*, *Nano Letters* 11, 3957 (2011)

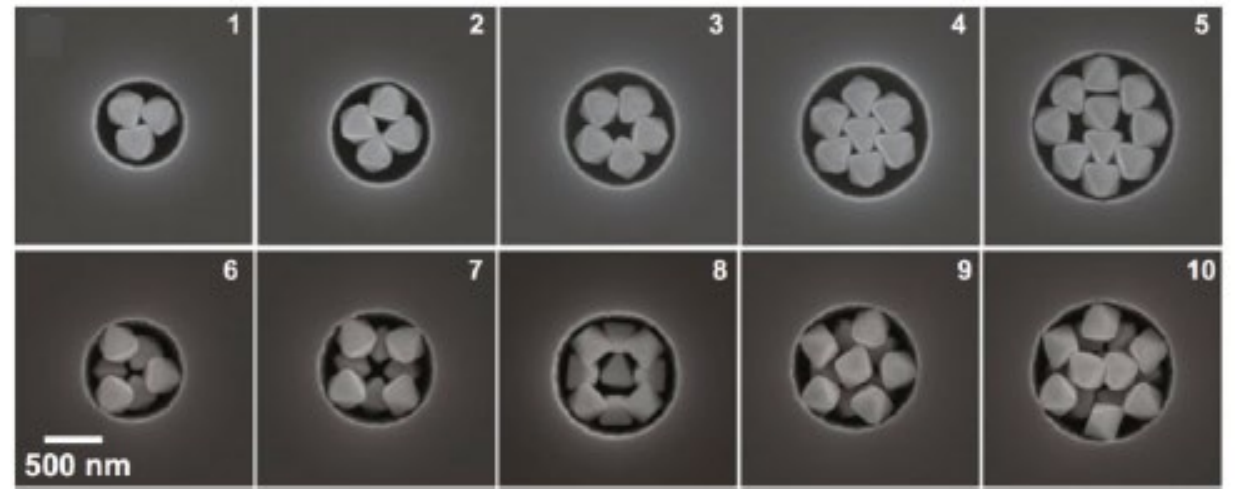
Further trap designs

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Trap design: Trap-to-particle ratio



Yin *et al.*, *J. Am. Chem. Soc.* 123, 8718 (2001)

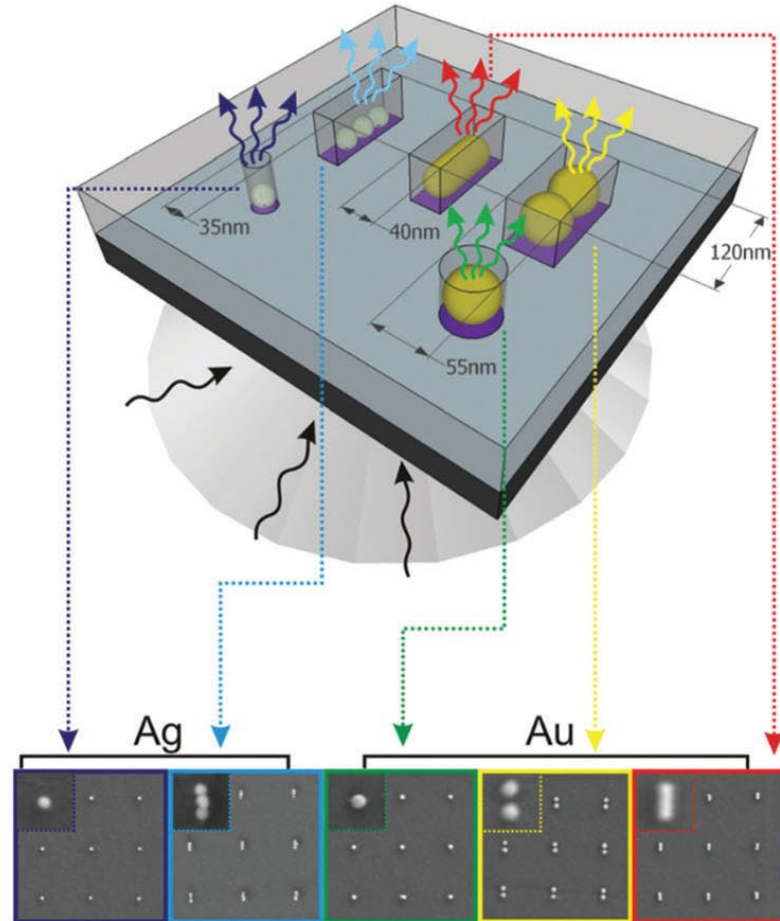
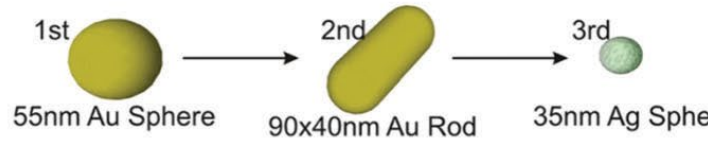


Henzie *et al.*, *Proc. Natl. Acad. Sci* 110, 6640 (2013)

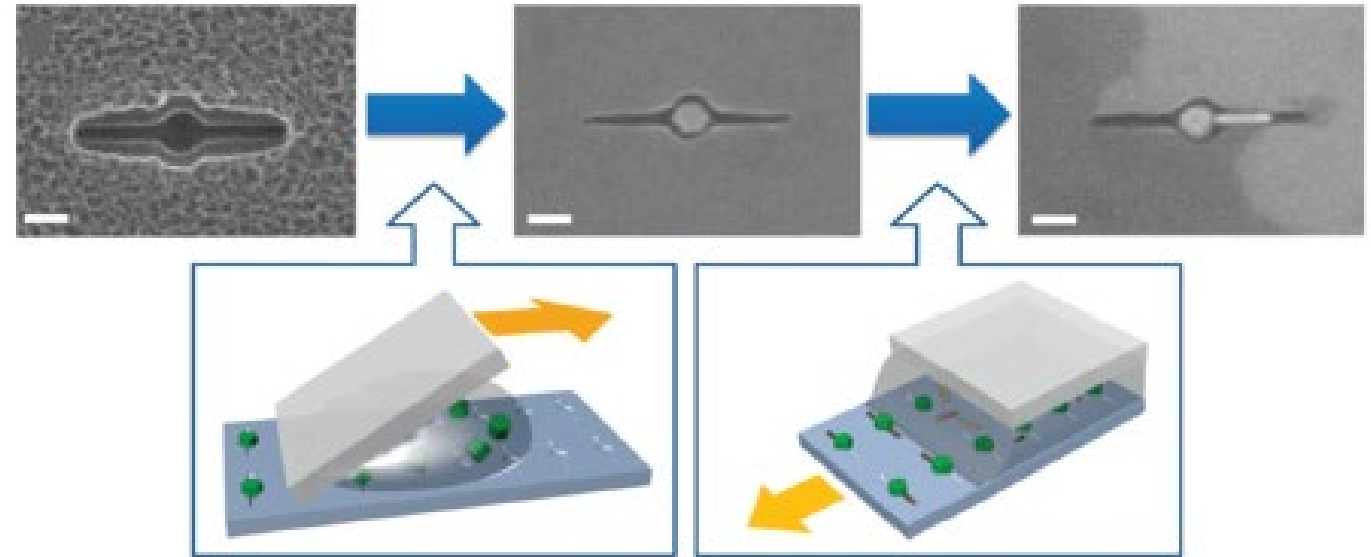
Further trap designs

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Trap design: Shape matching



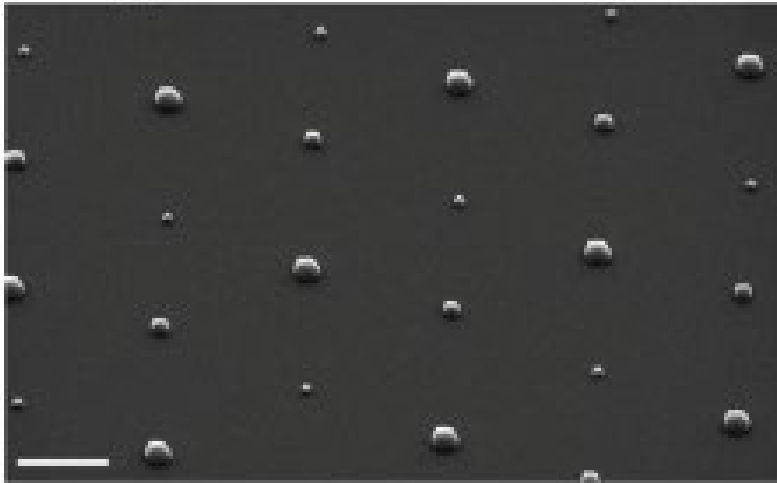
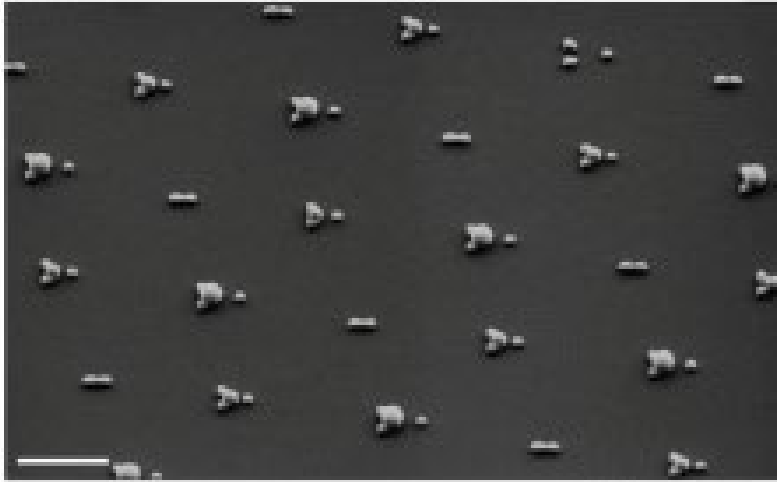
Chen & Reinhard, *Adv. Mater.* 28, 3522 (2016)



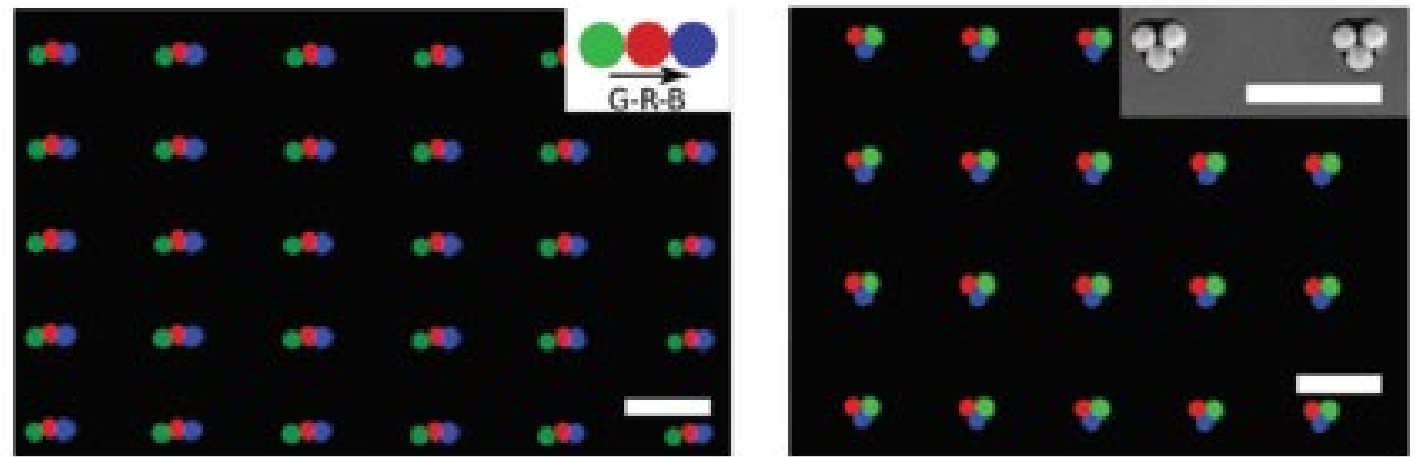
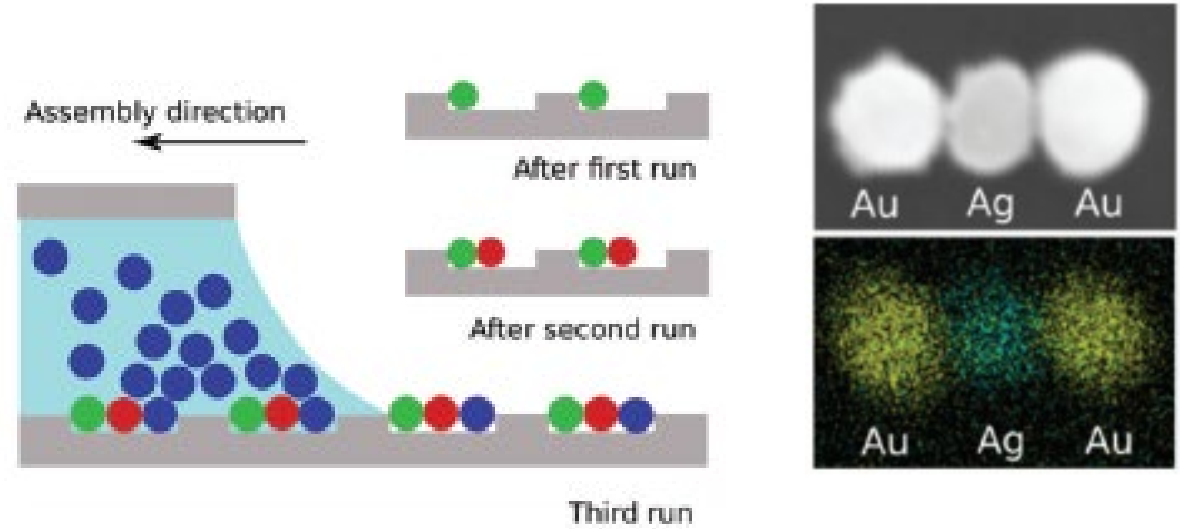
Greybush *et al.*, *ACS Nano* 9, 9482 (2014)

Further trap designs

Trap design: Shape matching



Kuemin *et al.*, *Adv. Mater.* 22, 2804 (2010)

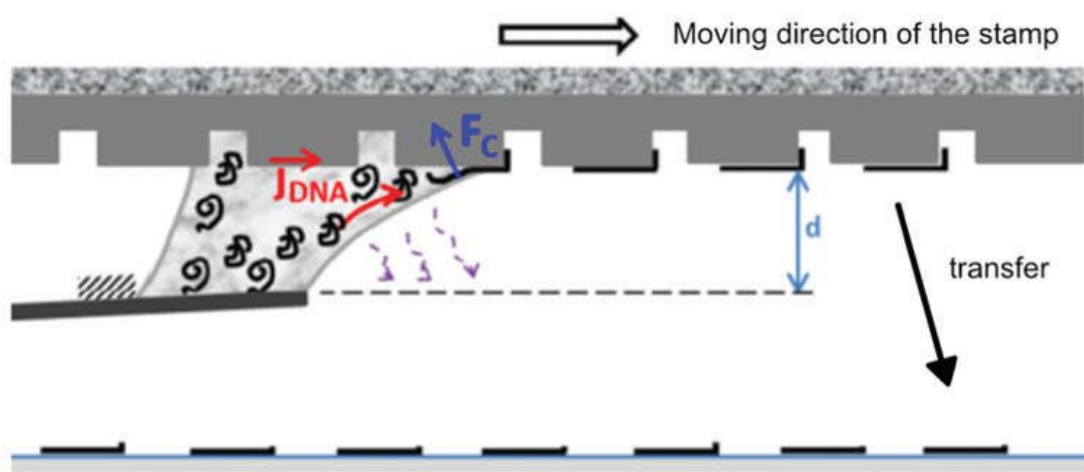


Ni *et al.*, *Soft Matter* 2018

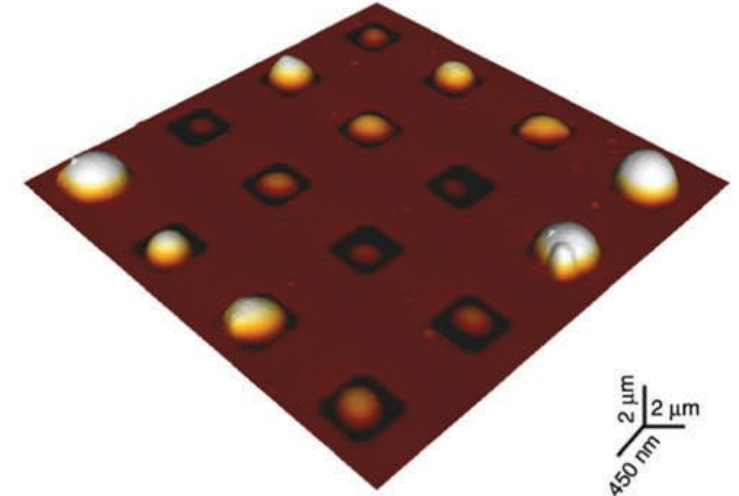
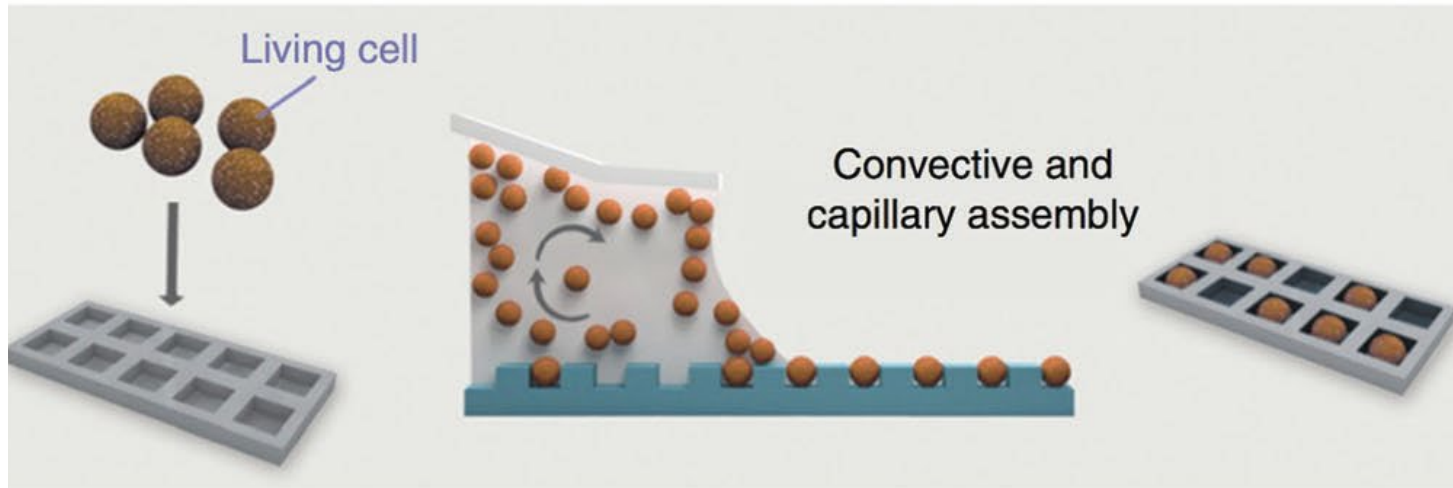
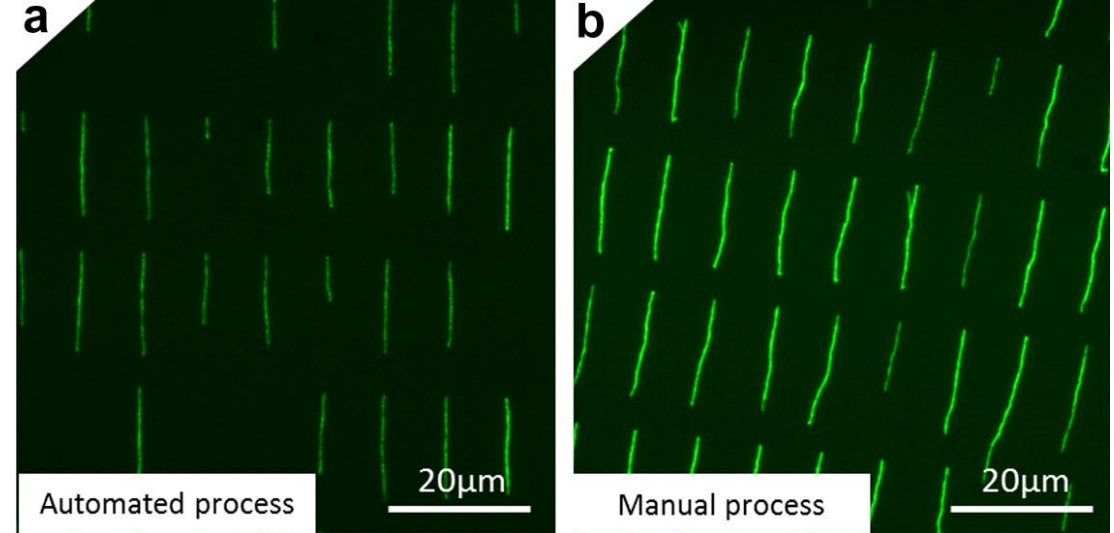
Further trap designs

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Biomolecules & cells



Cayron *et al.*, *Microelectron. Eng.* 135, 1 (2015)

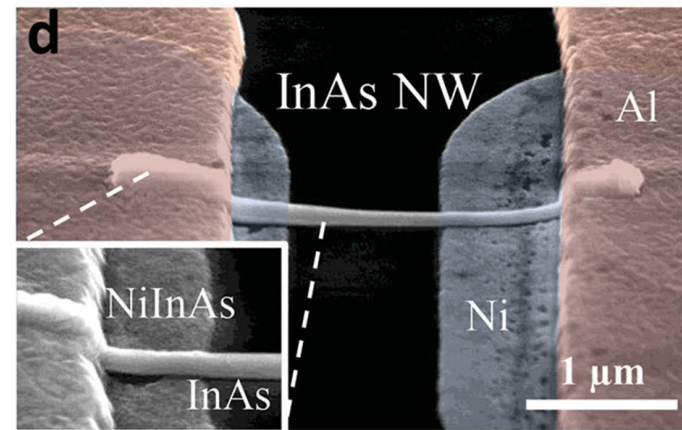
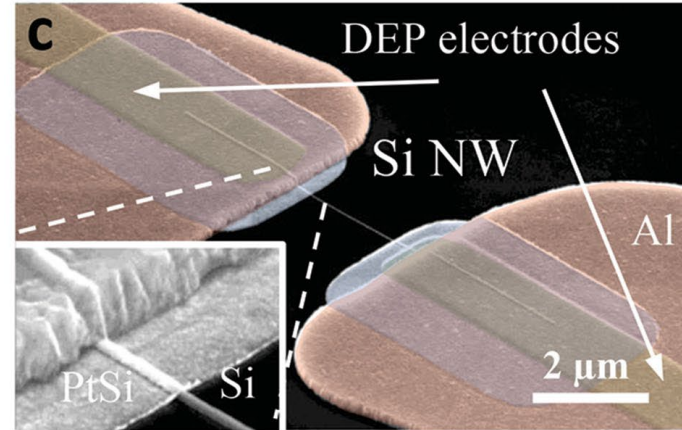
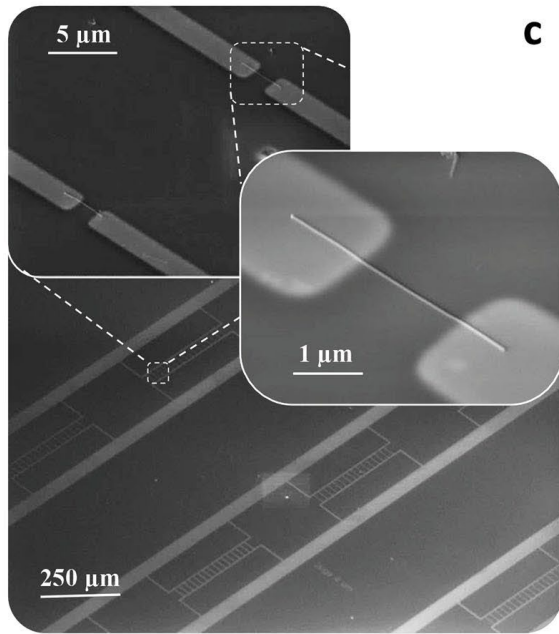
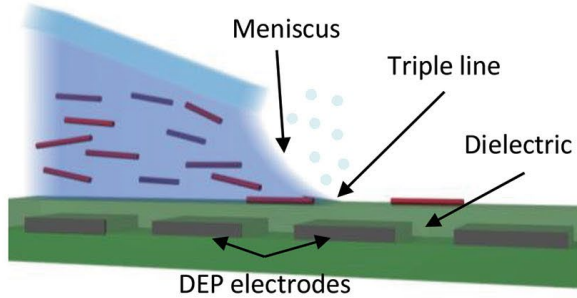


Formosa *et al.*, *Nat. Protoc.* 10, 199 (2015)

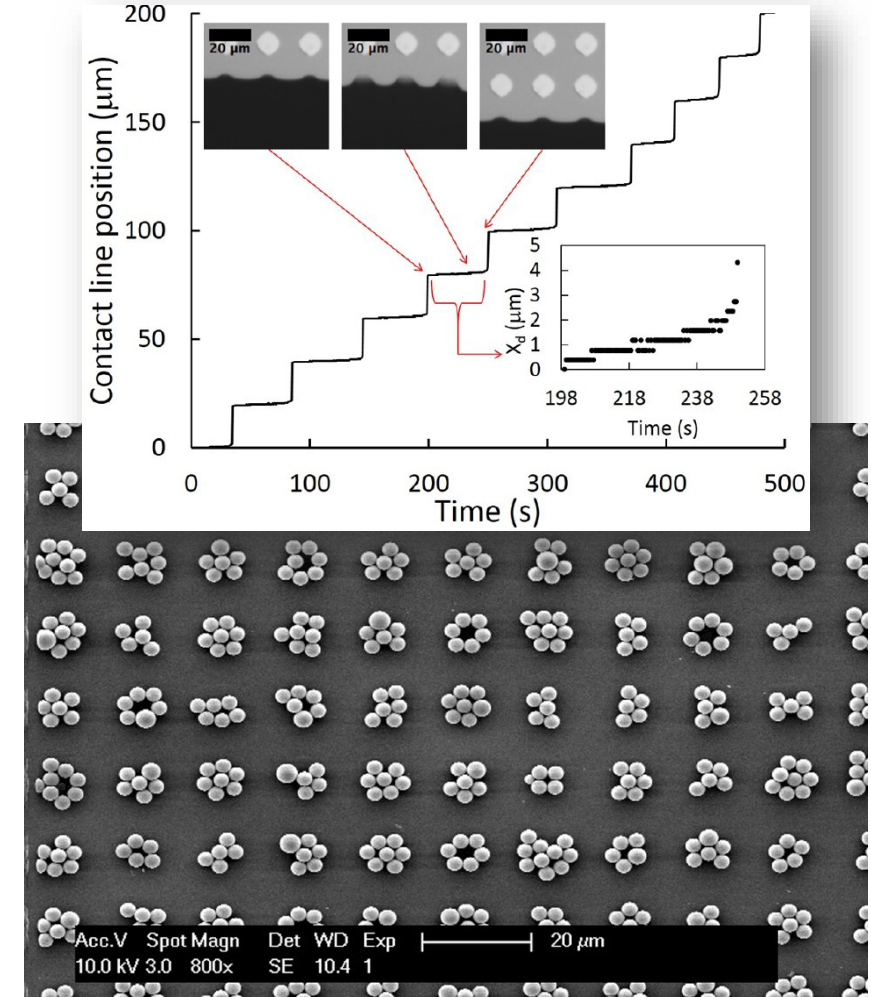
Further trap designs

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Non-topographical traps



Collet *et al.*, *Adv. Mater.* 27, 1268 (2015)

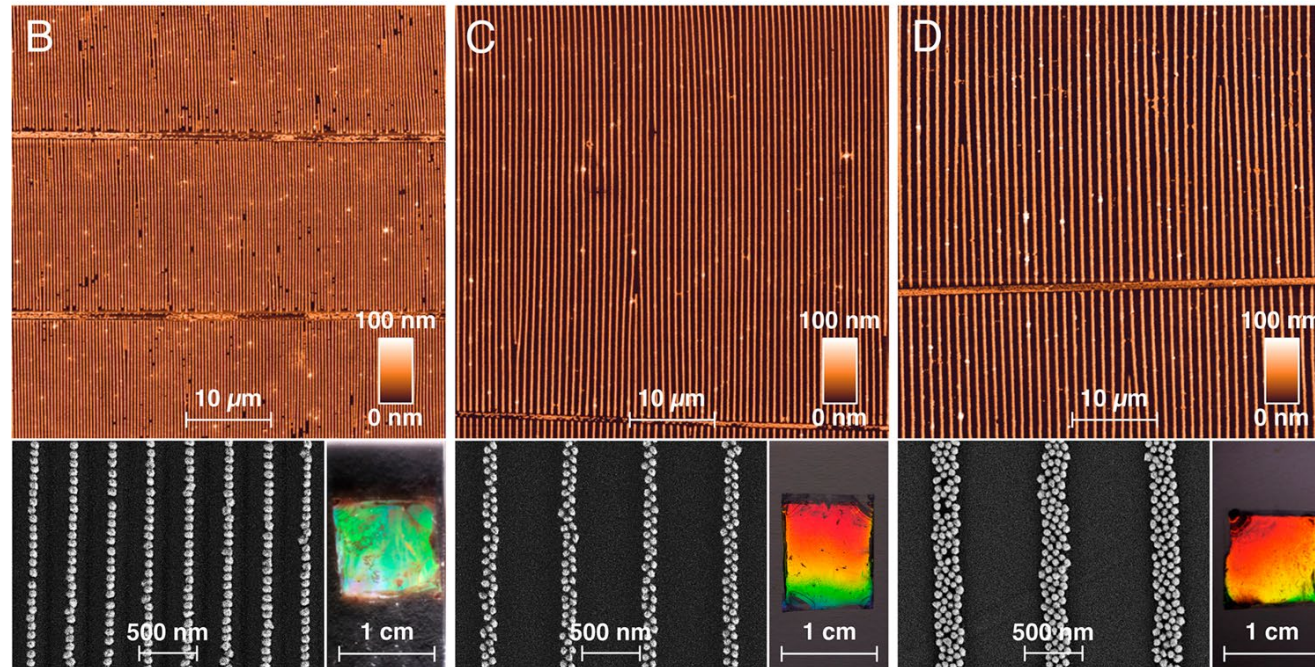
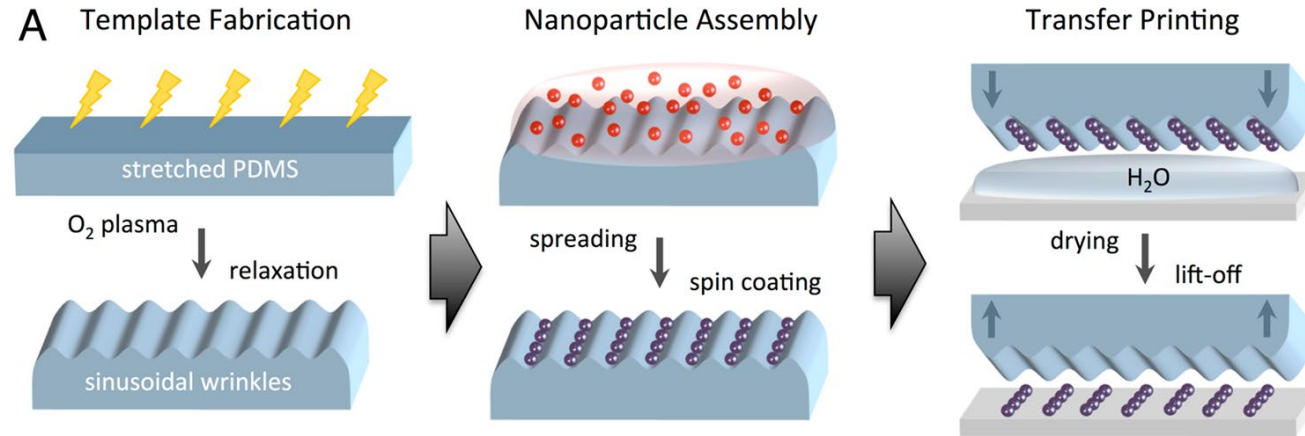


Wirth *et al.*, *Langmuir* 31, 1632 (2015)

Further trap designs

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Non-lithographical traps

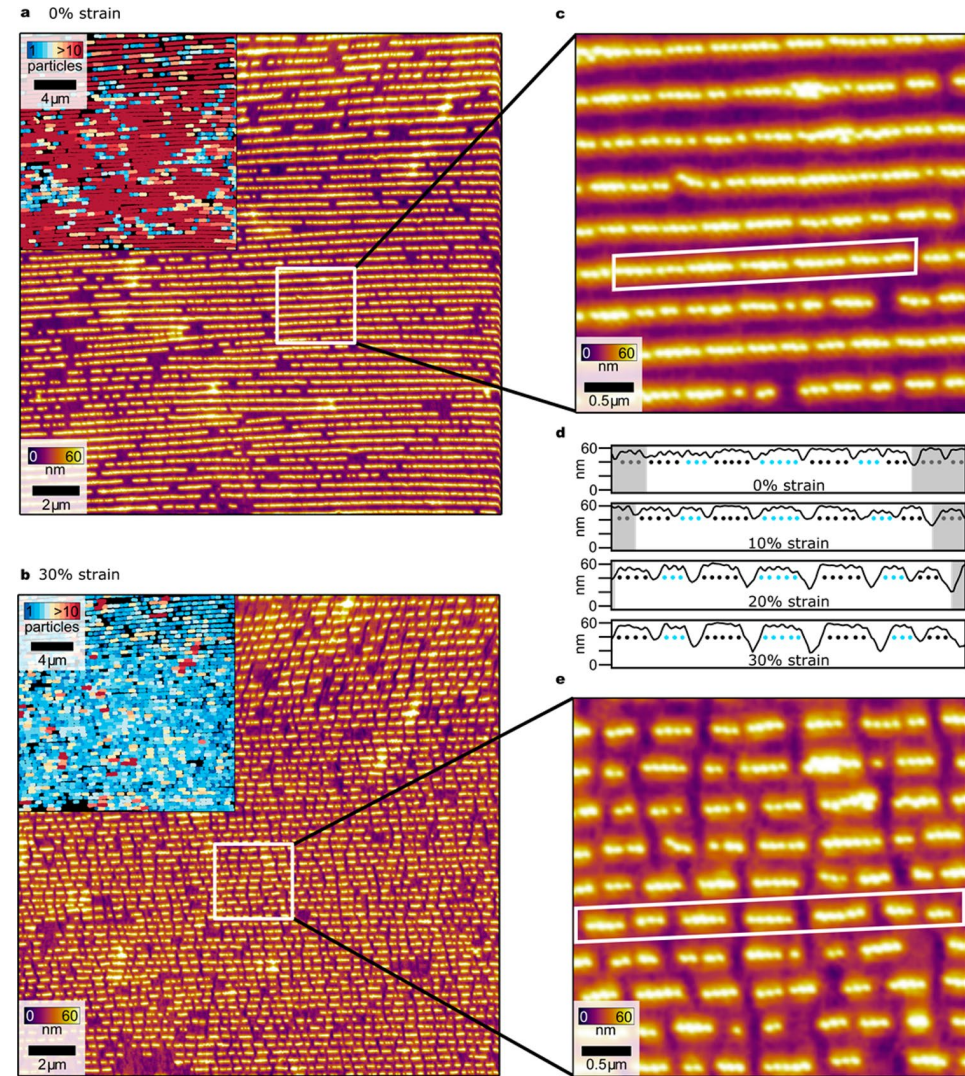
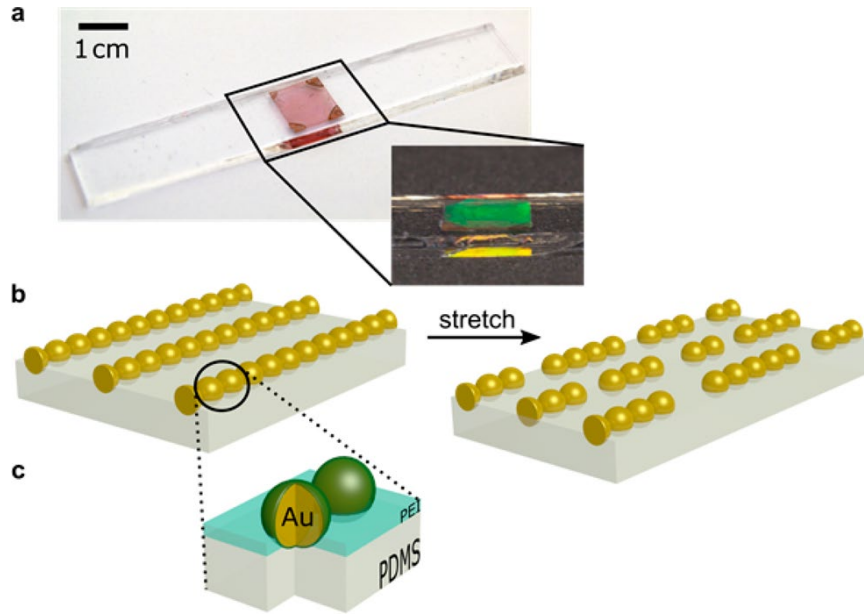


Hanske *et al.*, *Nano Letters* 14, 6863 (2014)

Further trap designs

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Non-lithographical traps



Steiner *et al.*, *ACS Nano* 11, 8871 (2017)

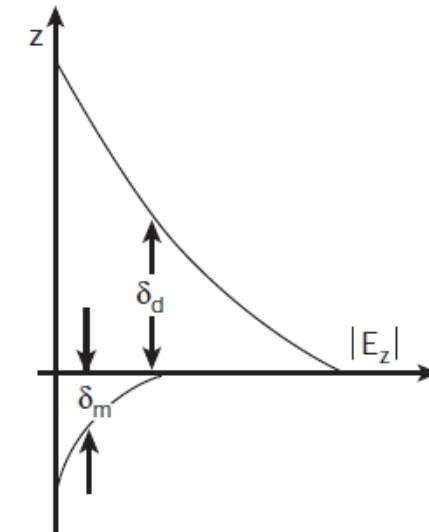
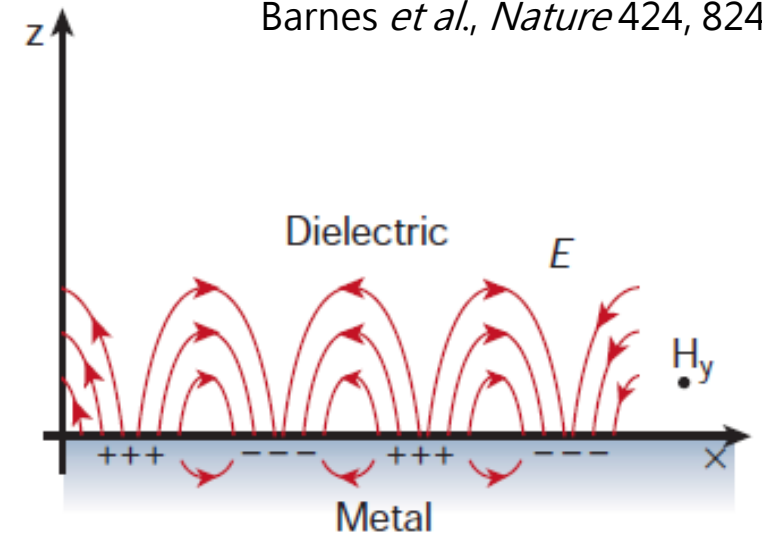
Further trap designs

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Surface plasmon polaritons

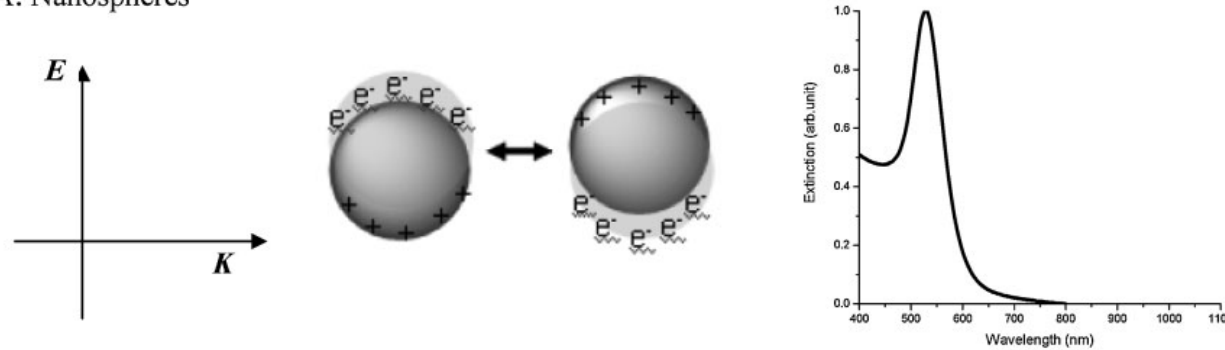
- Combined EM wave & surface charge
- Bound at metal/dielectric interface
- Perpendicular field component
 - enhanced at the surface
 - exponentially decaying
- Confinement of EM energy in sub-wavelength volumes

Barnes *et al.*, *Nature* 424, 824 (2003)

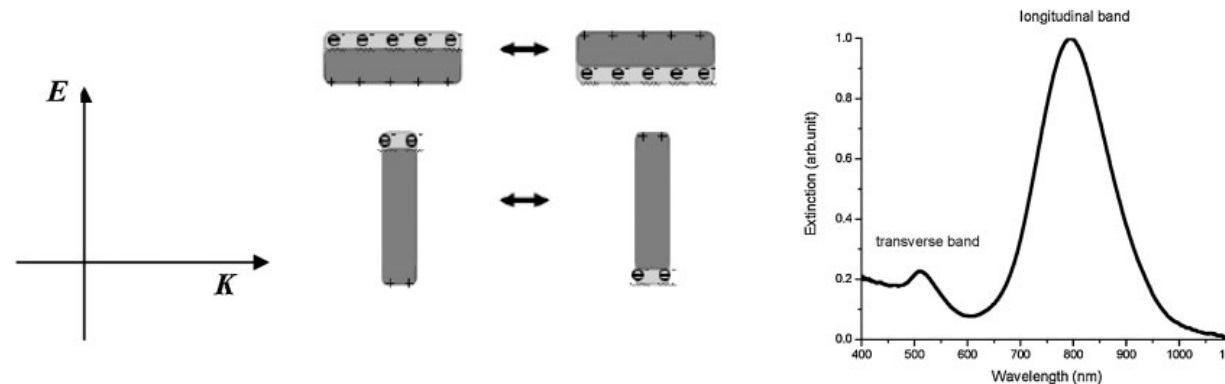


Localized surface plasmon resonances in NPs

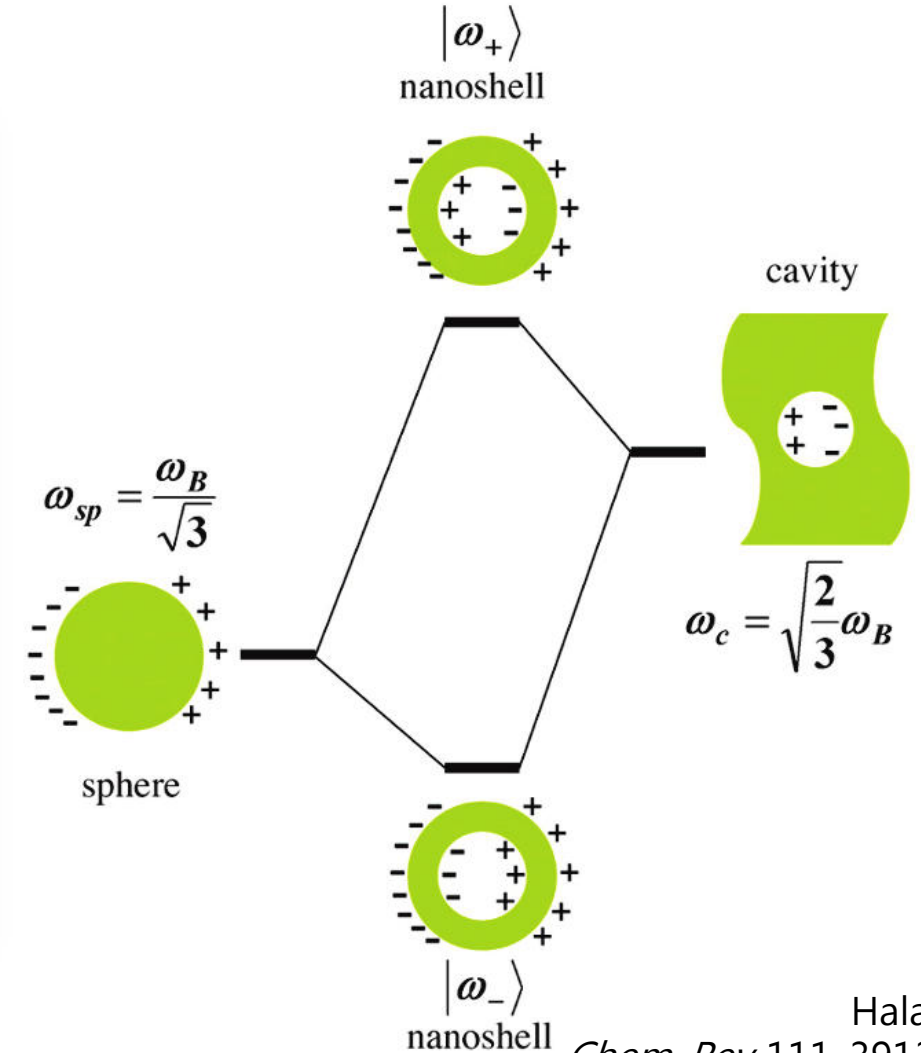
A: Nanospheres



B: Nanorods

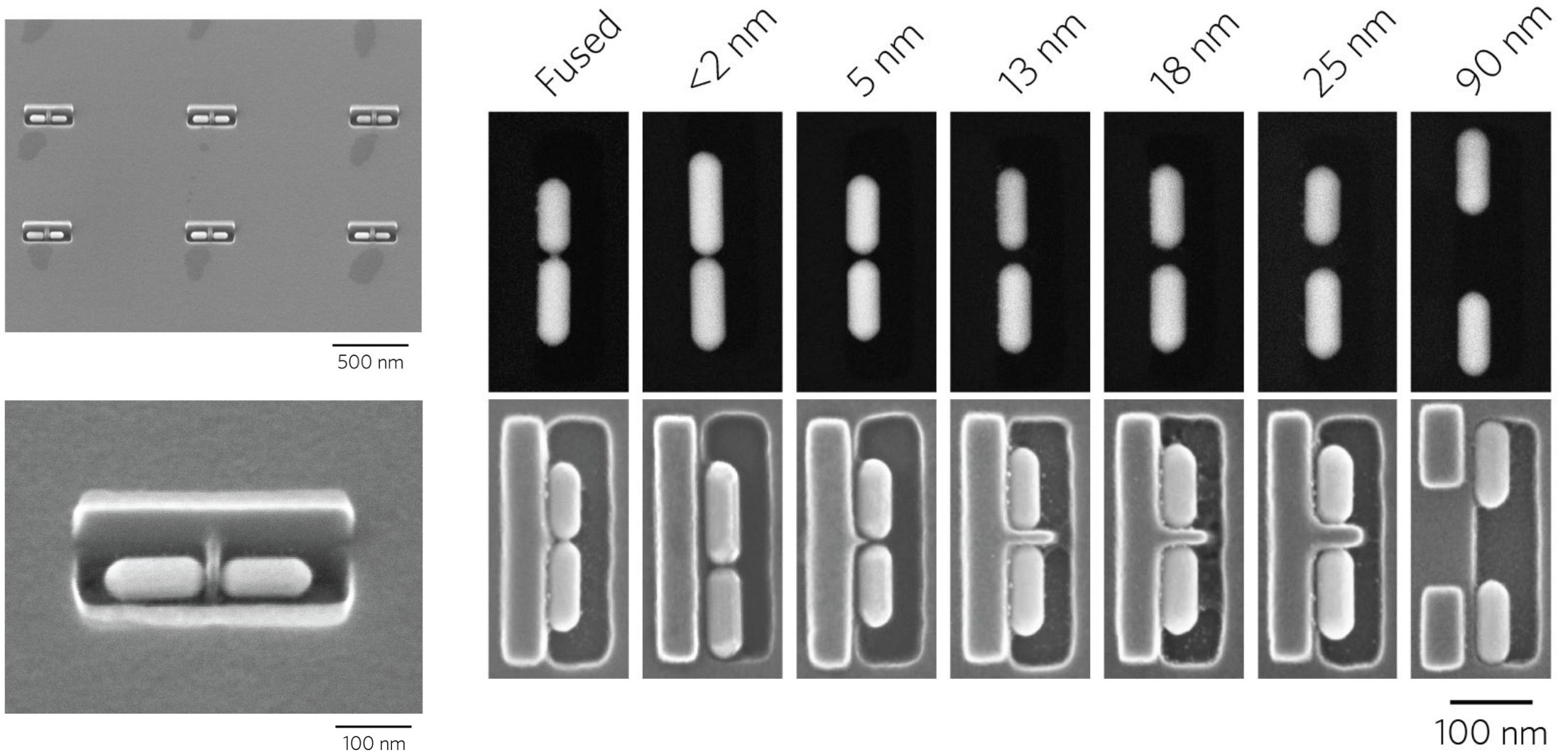


Huang *et al.*, *Adv. Mater.* 21, 4880 (2009)



Halas *et al.*,
Chem. Rev. 111, 3913 (2011)

Nanogap tuning in nanorod dimers

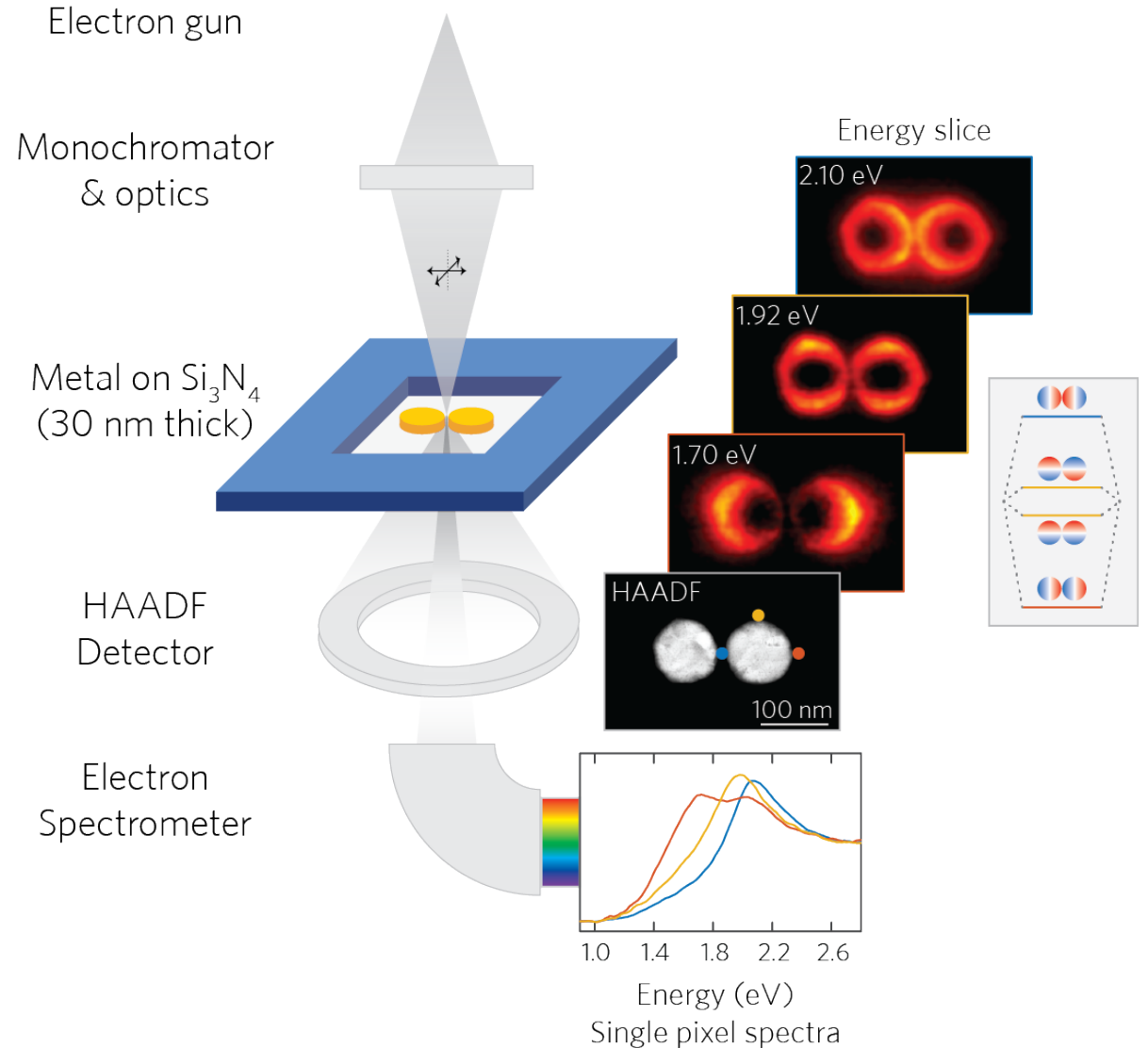
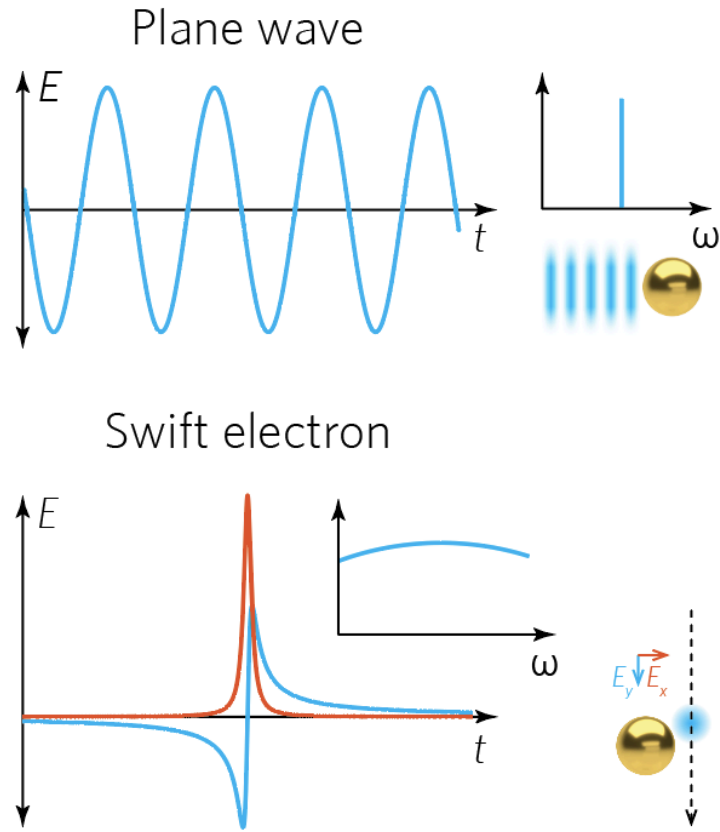


Flauraud *et al.*, *Nat. Nanotechnol.* 12, 73 (2017)

Plasmonic antennas

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Electron energy-loss spectroscopy: plasmon maps



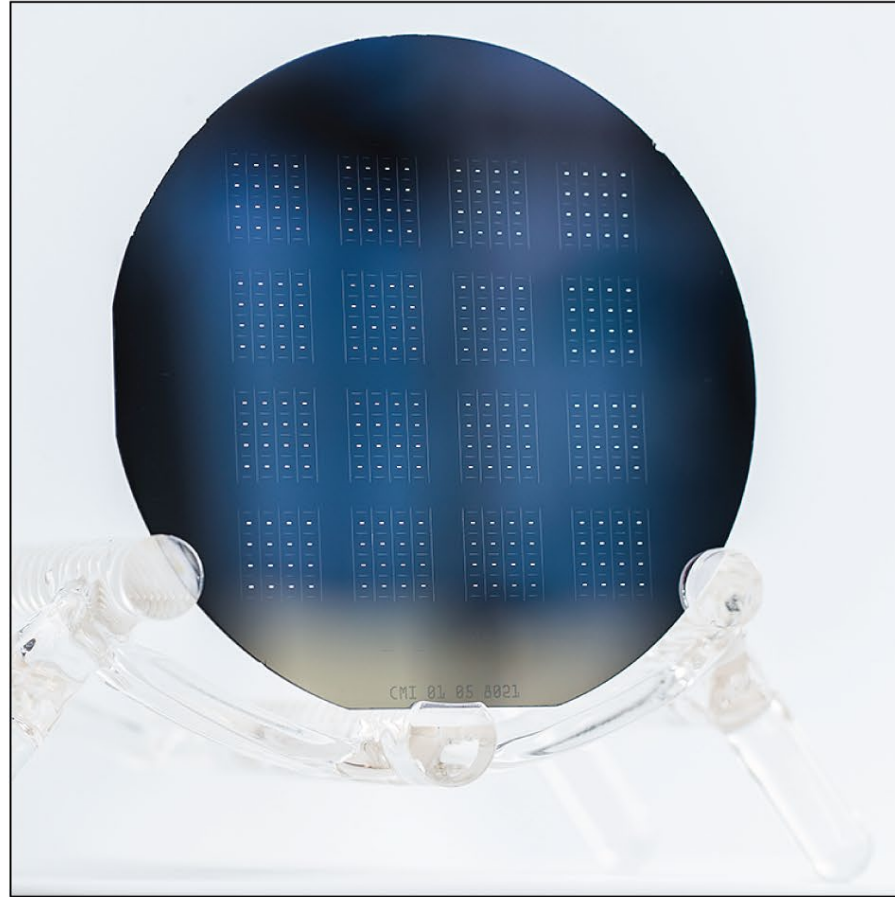
Courtesy of V. Flauraud

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Fabrication of TEM compatible substrates

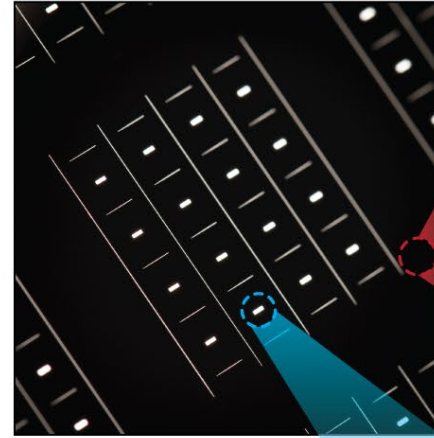
4" wafer: 256 Si_3N_4 TEM samples, 30 nm-thick



2 cm

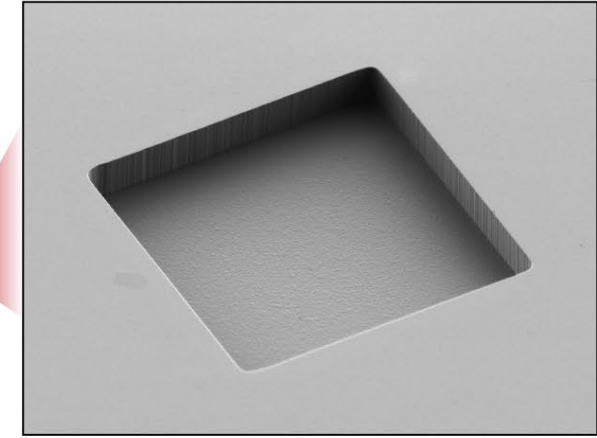
Courtesy of V. Flauraud

Windows: 500x100 μm

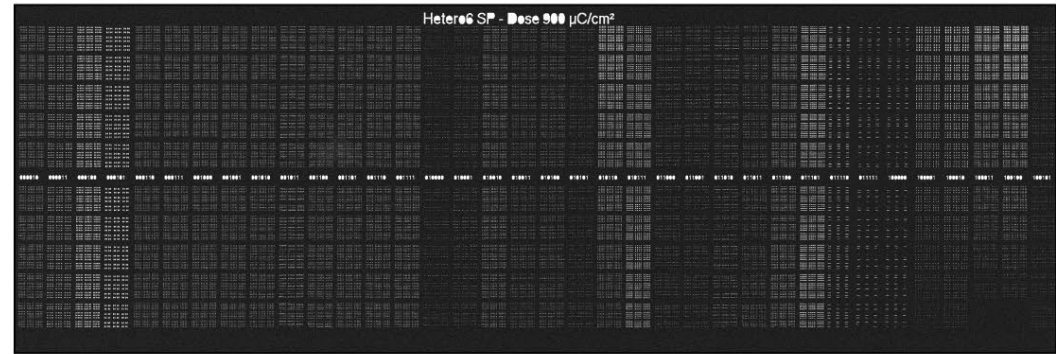


5 mm

Ebeam alignment markers



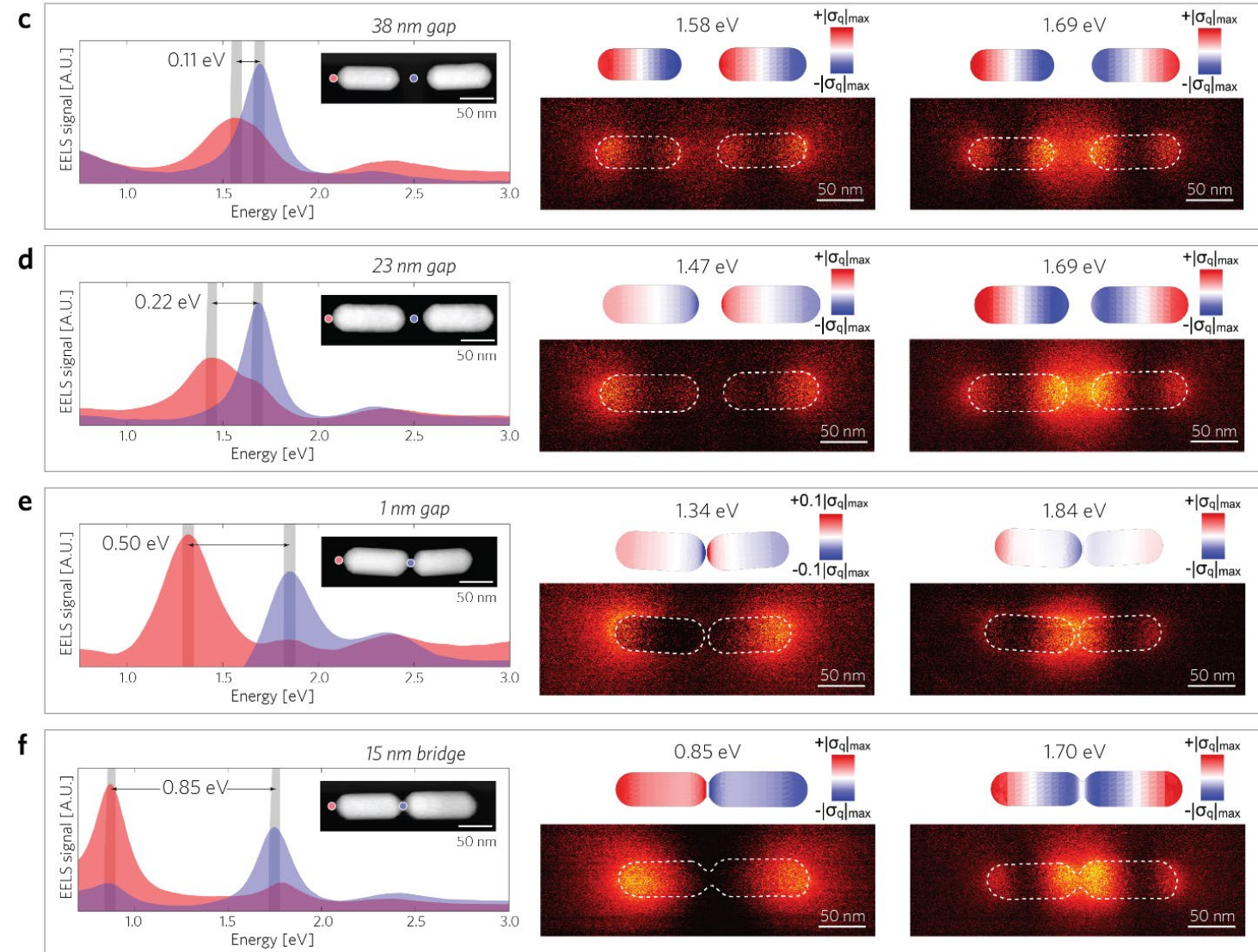
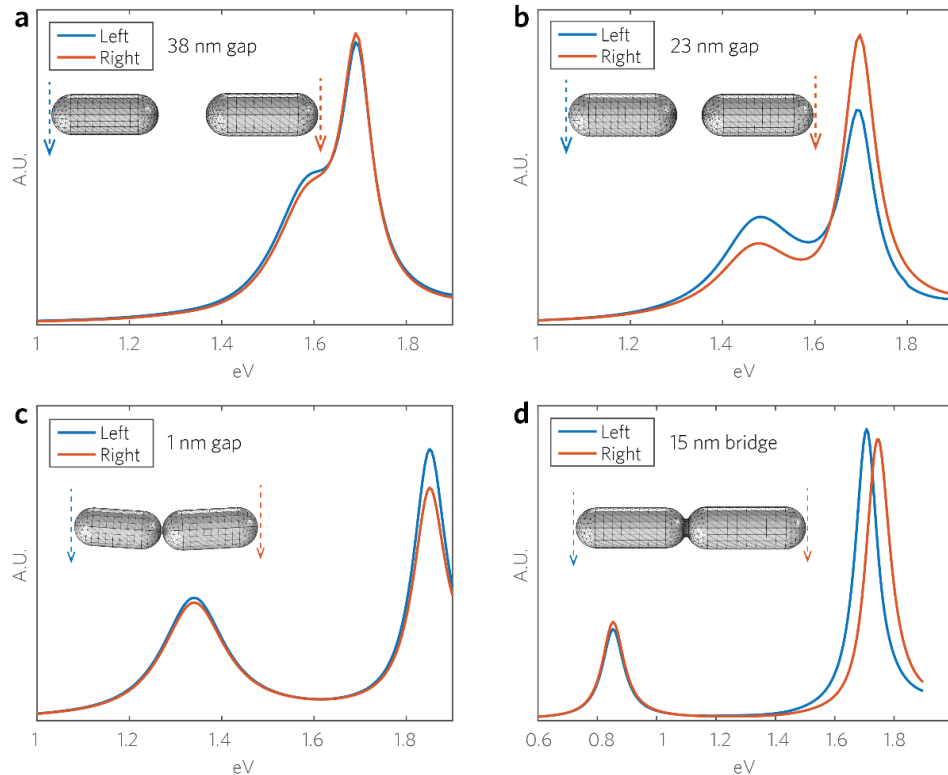
10 μm



50 μm

From weak to strong plasmonic coupling

FEM simulations

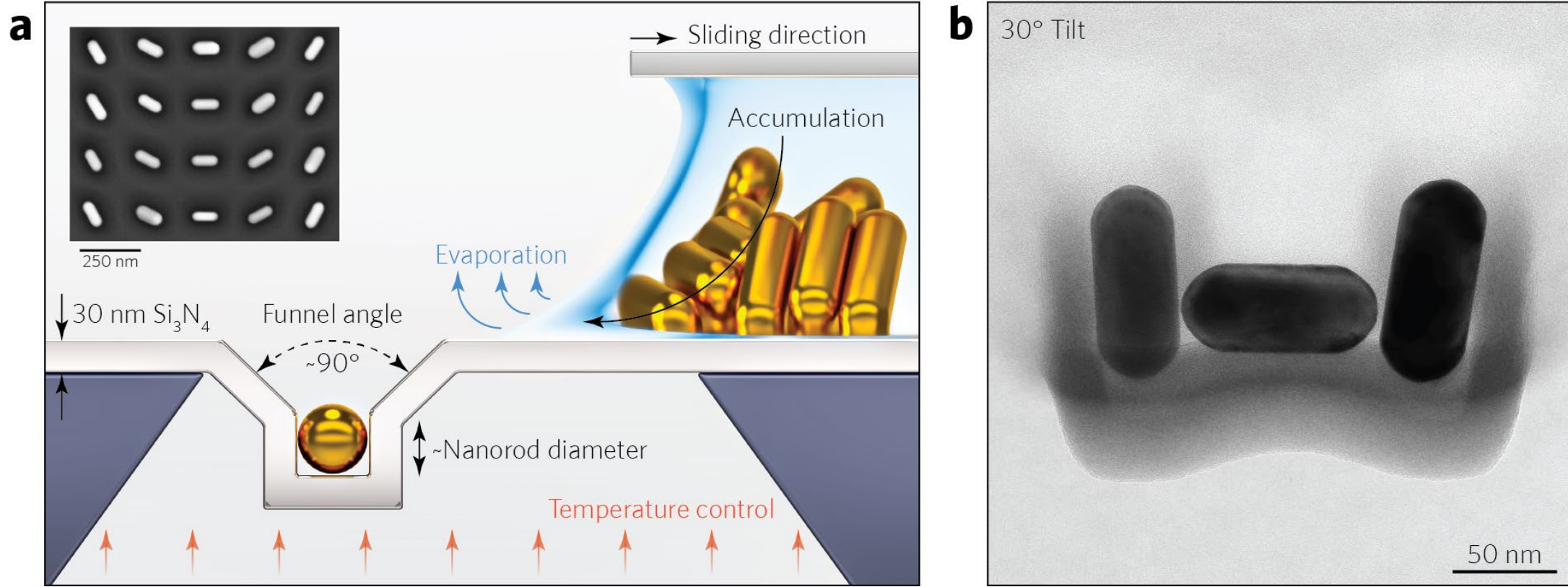


Flauraud *et al.*, *Nat. Nanotechnol.* 12, 73 (2017)

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Plasmonic dolmens

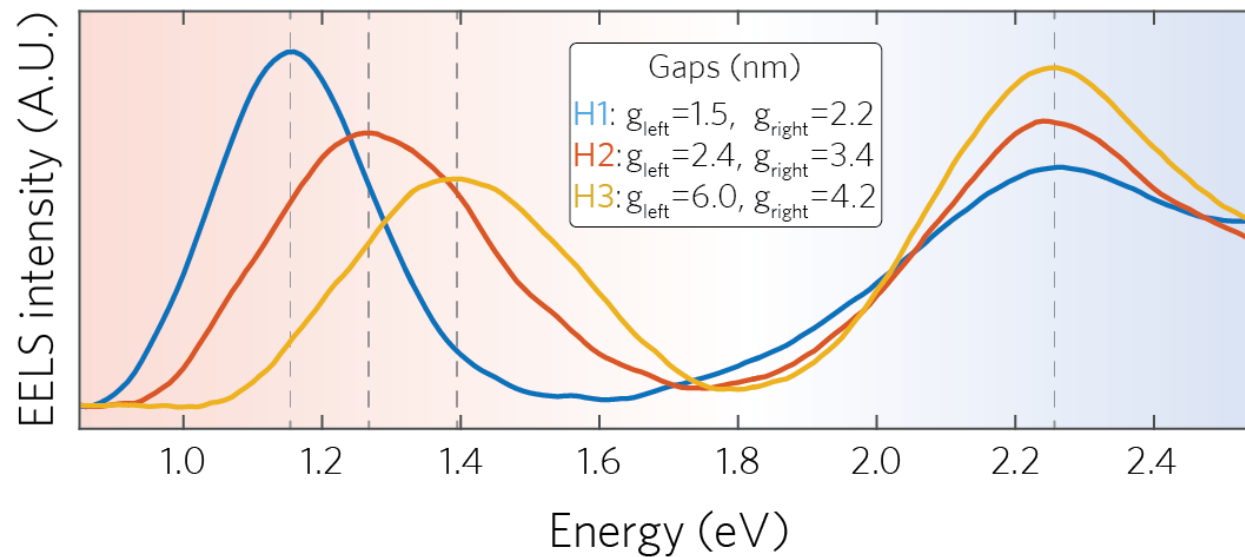
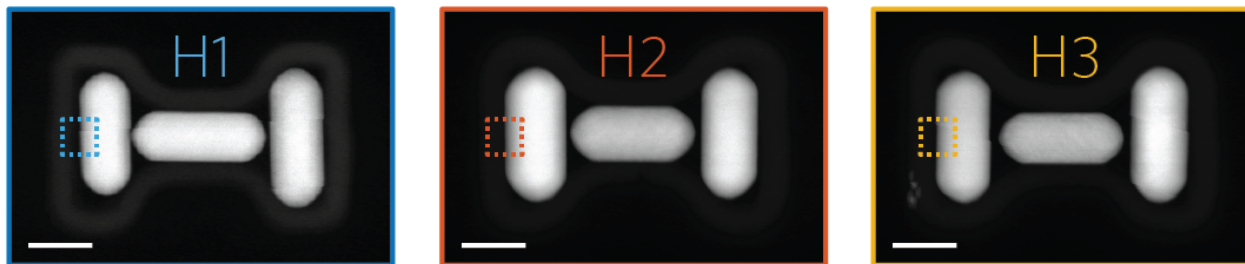


Flauraud *et al.*, *ACS Photon.* 4, 1661 (2017)

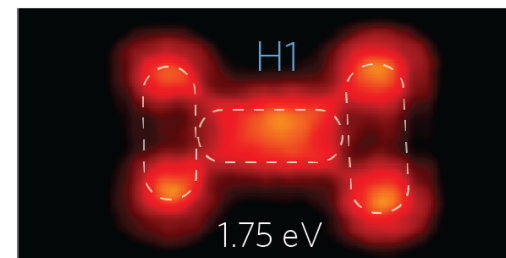
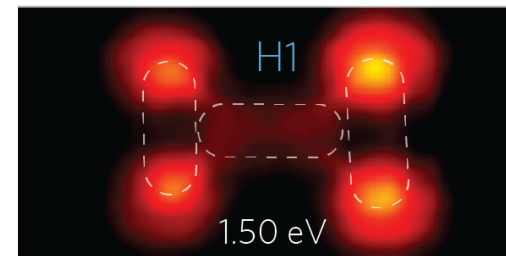
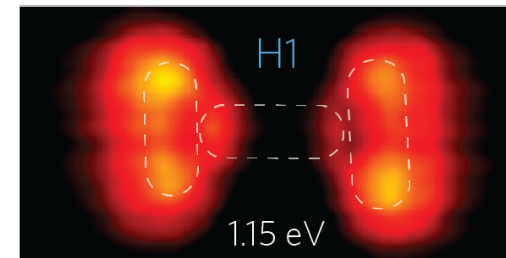
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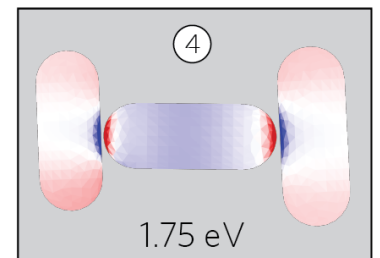
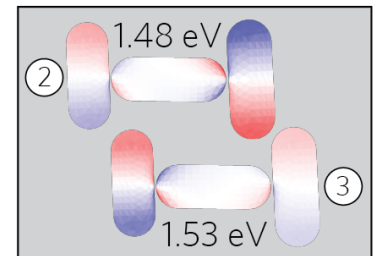
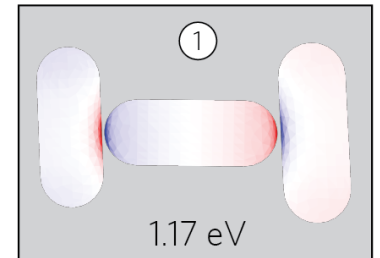
Plasmonic dolmens



Experimental EELS maps



Simulated eigenmodes

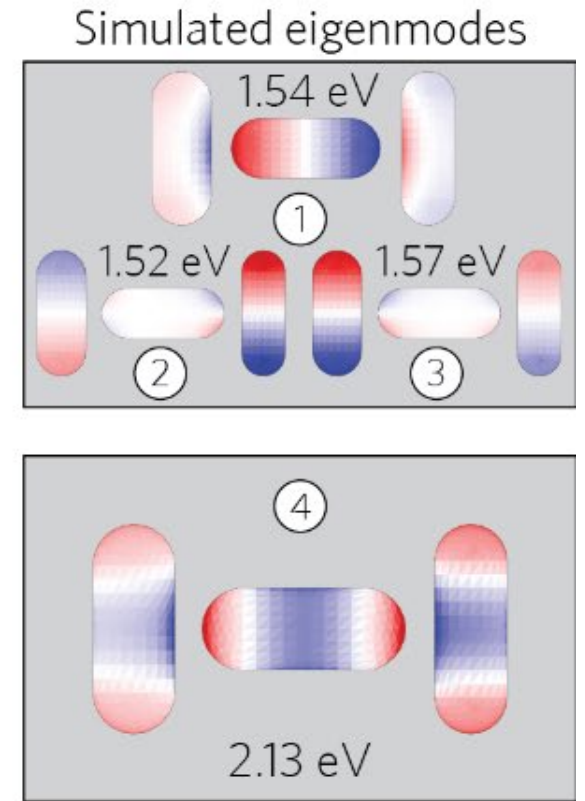
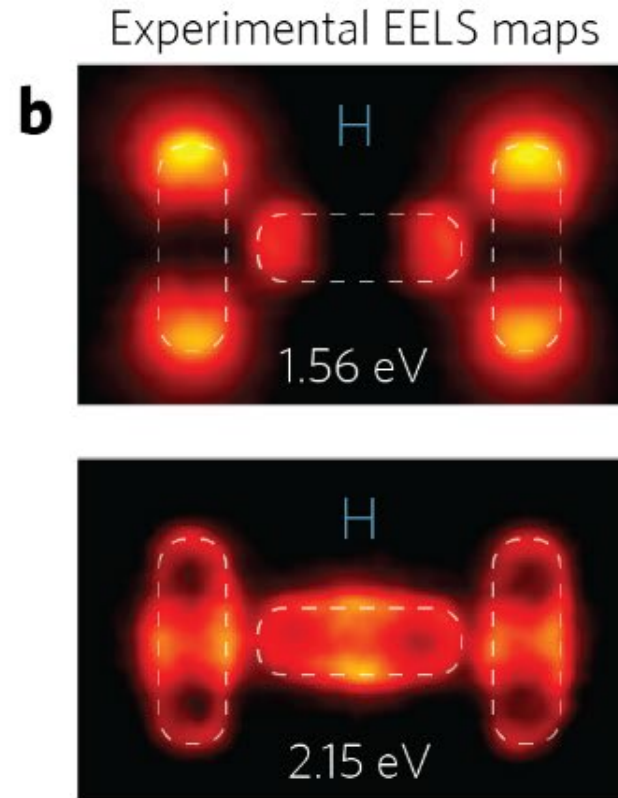
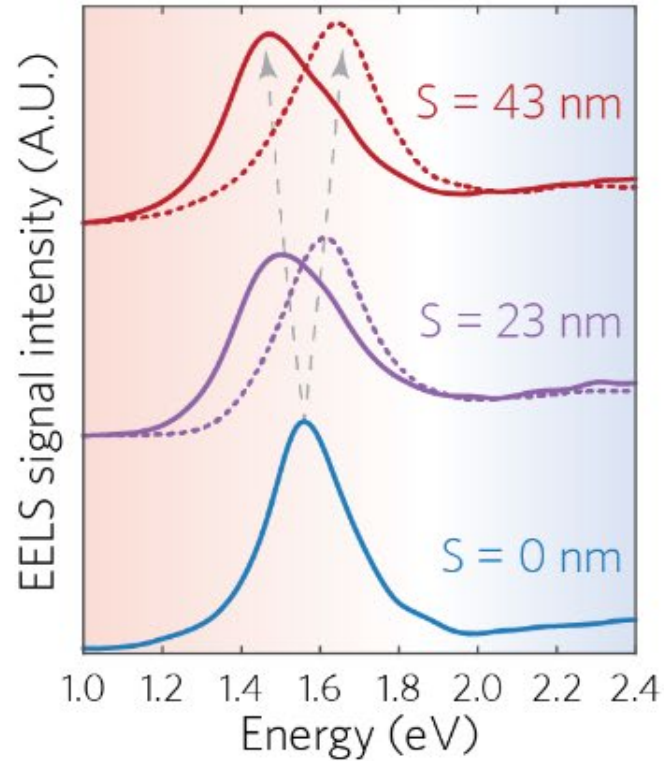
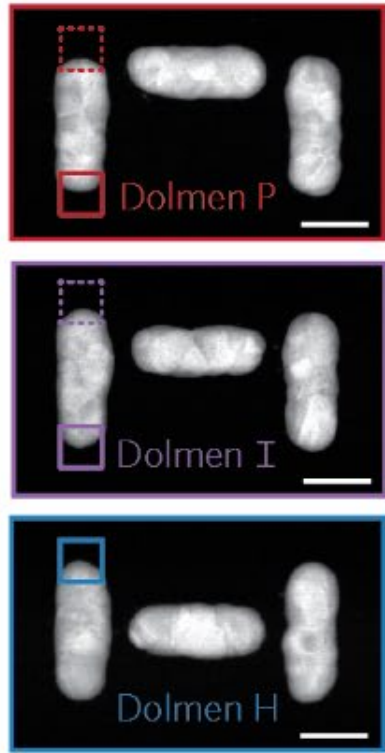


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Plasmonic dolmens

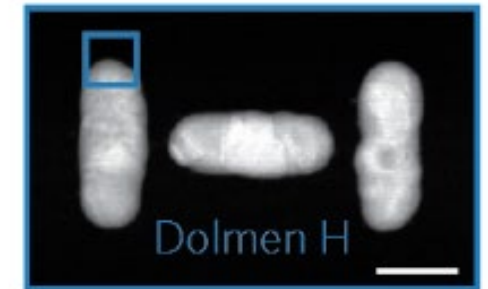
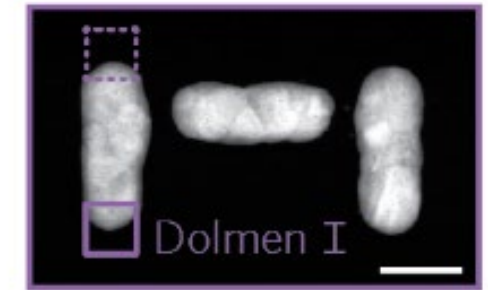
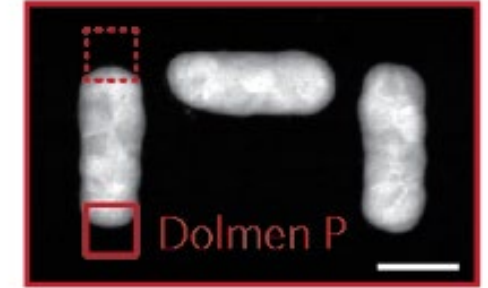
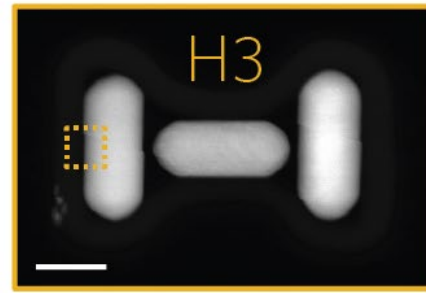
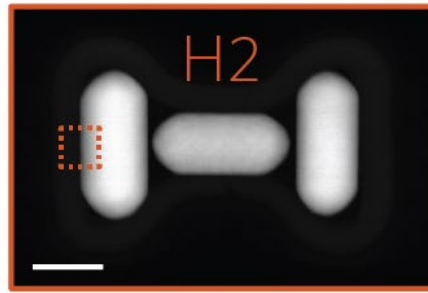
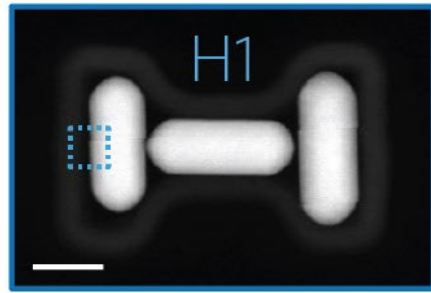


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Crystallinity & edge roughness

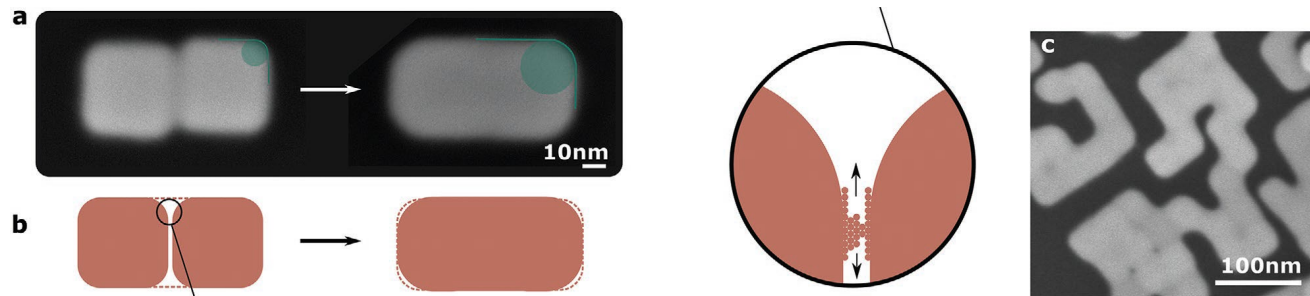
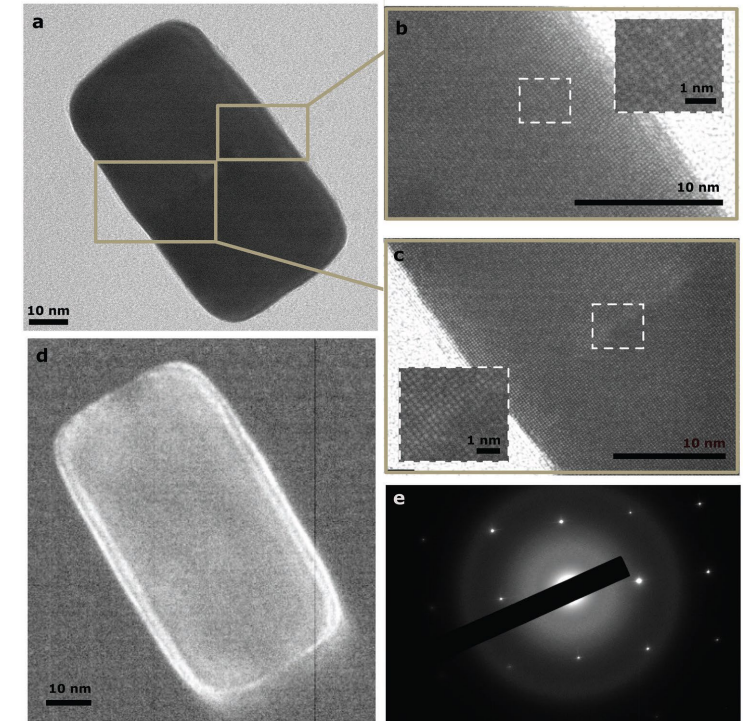
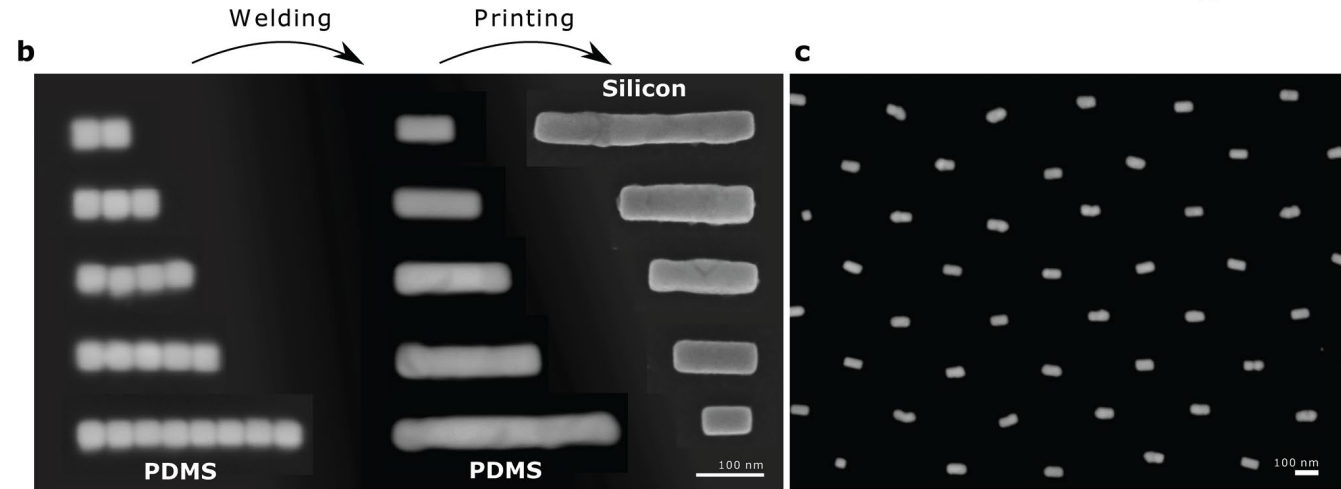
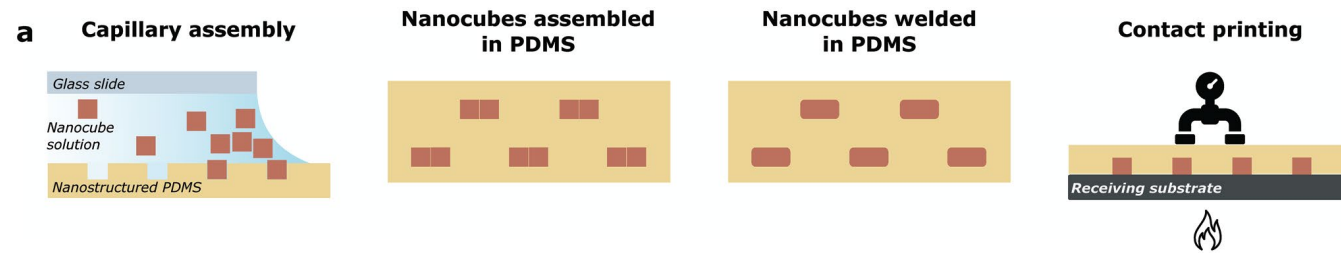


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Monocrystals from nanocube epitaxy

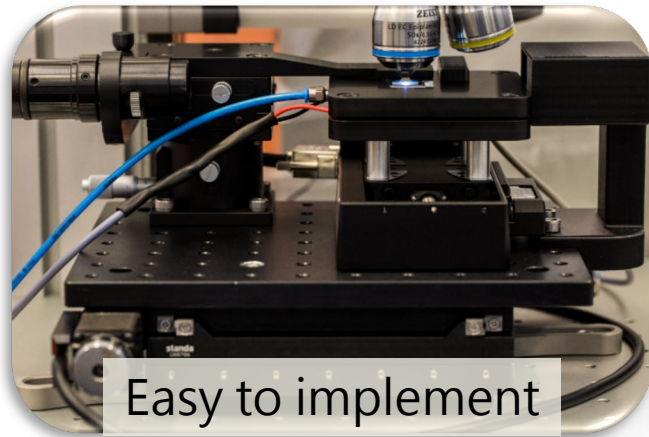


Capitaine & Sciacca, *Adv. Mater.* 34, 2200364 (2022)

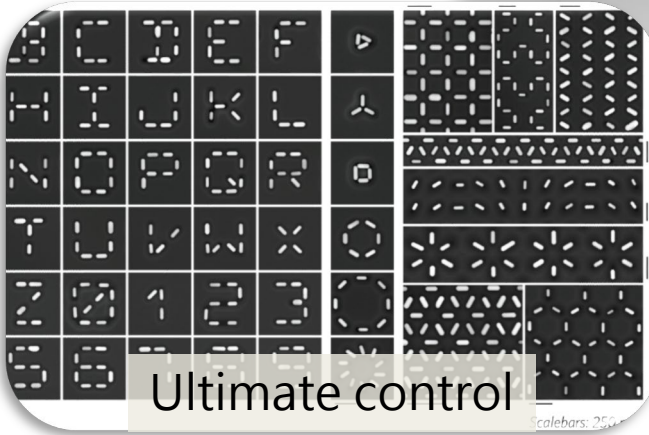
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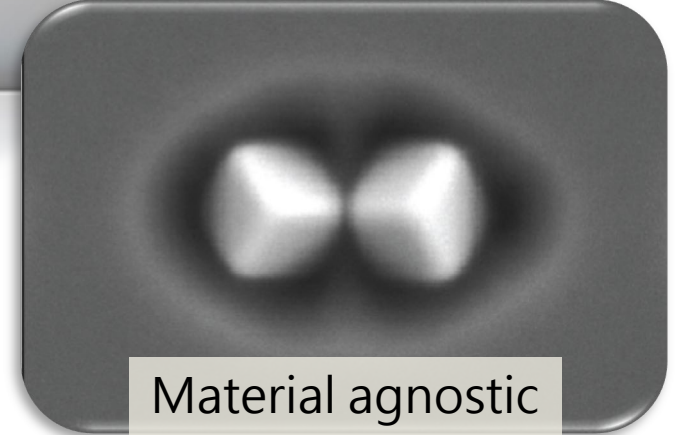
Take home messages



Top-down patterning



Bottom-up assembly



Conclusions

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