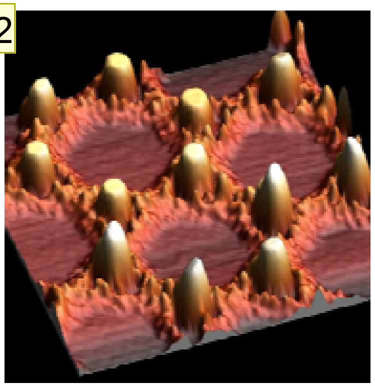
Hilfe2



EMPA

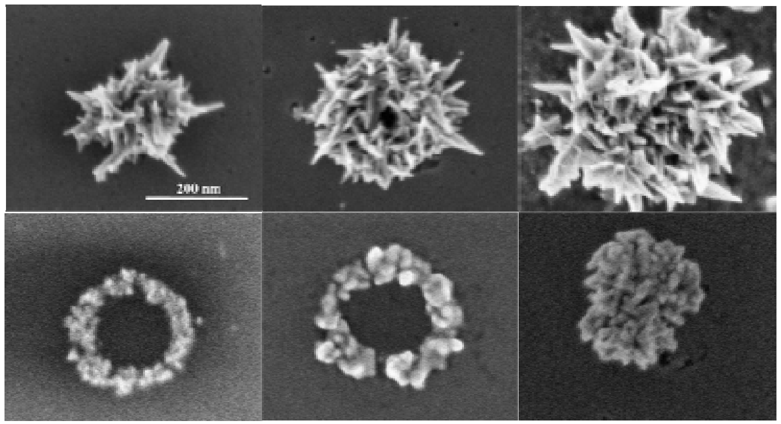
Materials Science & Technology

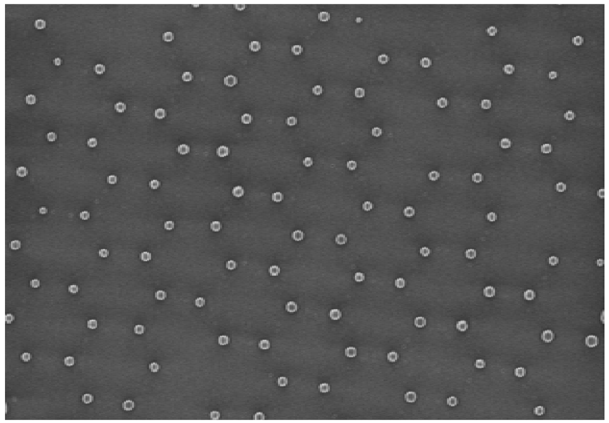
Grippers and surfaces structuring for micro and nanomanipulation

Mikhael BECHELANY

Laboratory for Mechanics of Materials and Nanostructures

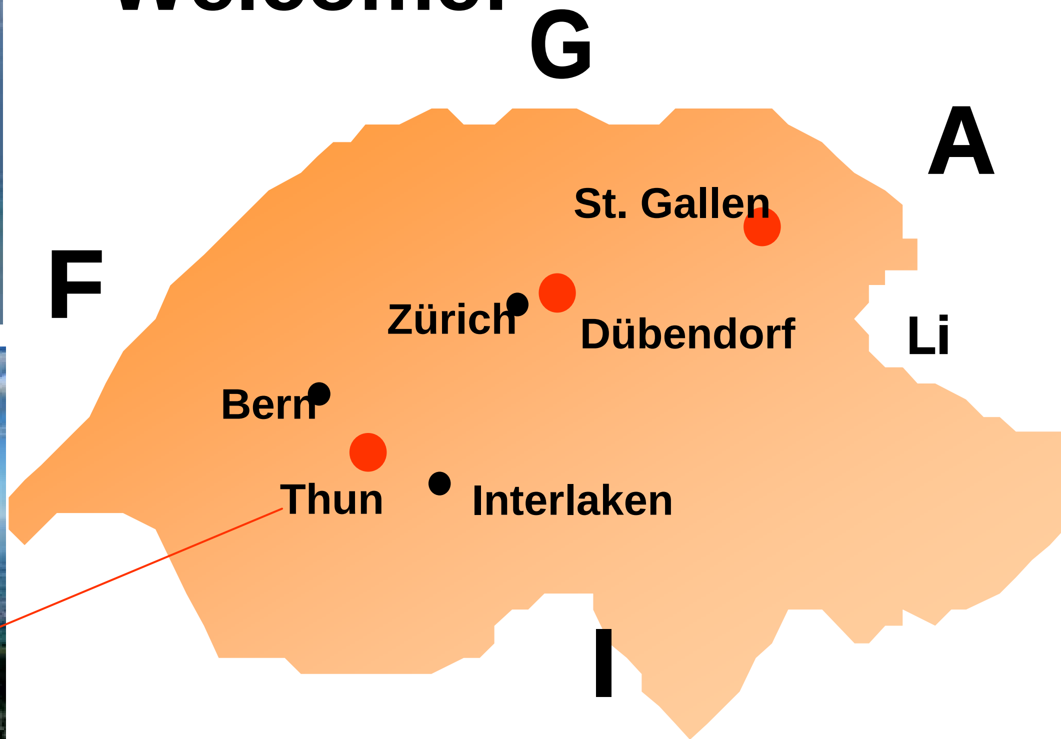
EMPA, Thun, Switzerland





EMPA in Thun Switzerland

Bienvenue!
Willkommen!
Welcome!



...at the Swiss Federal Laboratories for Materials Testing and Research in Thun

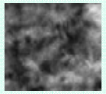
Major research areas of the lab

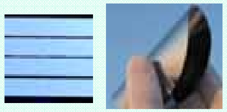
Nanomechanics

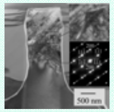

superlat.

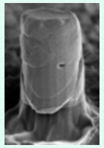

Si NW

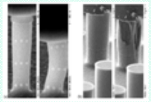

a-Me

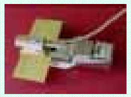

nc hard coat.


„Wafer mechanics“


Pillar GaAs

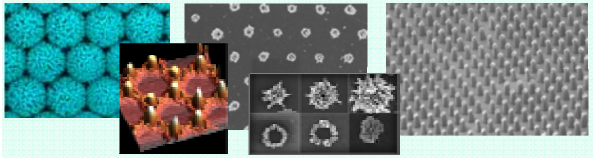

CNT in Cu

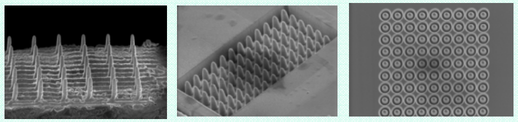

fracture of silicon

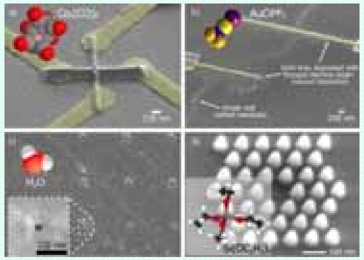

F&E Picoindenter

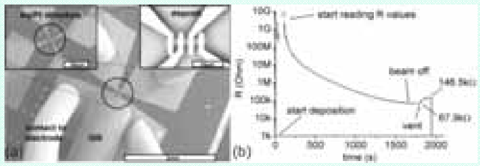

Ni/Co alloys

Nano/Micro Processing


Nanopatterning by NSL

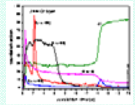

Nanopatterning by Gas assisted FIB/FEB

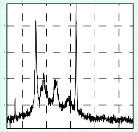

Device Prototyping by Gas assisted FIB/FEB

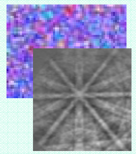

SEM integrated electronic measurements

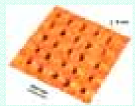
Nano/Microanalysis

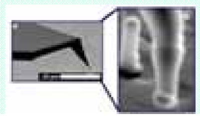

AFM in SEM


composition:
GDOES, GDTOFMS,
LAGDTOFMS,
FIBSIMS, EDX


phase & stress state:
micro Raman
spectroscopy


microstructure:
electron microscopy
with STEM, EBSD


surface metrology:
stereomicroscopy,
profilometers, SEM


residual stress
via TERS

Start-up’s of the lab:
Tofwerk AG
Alemnis Gmbh
Galvatron Gmbh

Equipment

Microanalysis
GD-OES, GD-TOF-MS, LAGDTOFMS, XRD, DTA, Micro-RAMAN

Electron Microscopy/ FIB
3 SEMs with STEM, EDX, EBSD, VP, AFM, e-beam lithography, 3D BSE, GIS
Tescan Lyra/XMU Dual beam FIB

In-situ Micro-/Nanomechanics
Micro- tensile, -bending, -push-out, -indenter
Nano-indenter, -tensile, -bending, -compression, -vibrational

Materials & Surface Mechanics
2 Nanoindenters, 2 Universal testing machines

Electrochemistry
analytical potentiostats, ac impedance module
Variety of electrochemical cells

Clean room
Maskless UV lithograpy system, Hotplates for photoresist curing, Spin coaters, Oven

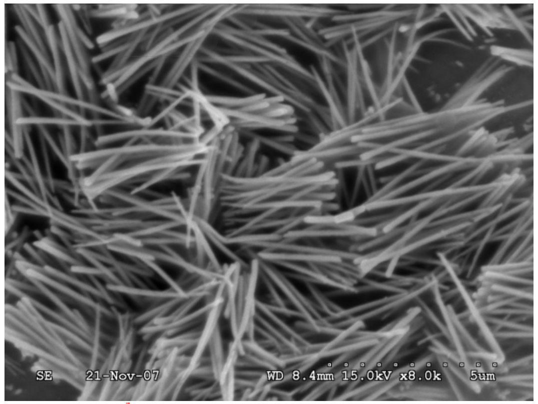
Nanorobotics

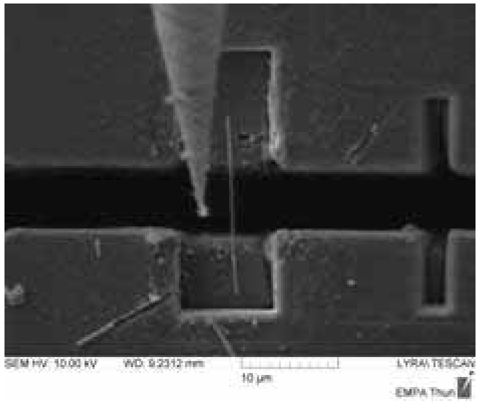
- **Nanorobotics divided into two main focus areas:**

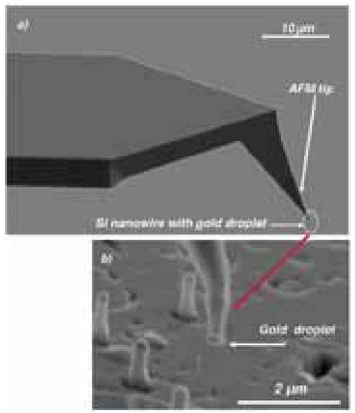
  - **Design, simulation, control and coordination of nanorobots**
    - ➲ **Highly theoretical because difficulties in fabricating such devices**

  - **Manipulation and/or assembly of nanoscale components**
    - ➲ **Macroscale instruments or robots (i.e., nanomanipulators)**

- **Measurement/manipulation at the nm scale crucial to the progress of nanoscale science and technology**







Why grippers?

- Scanning probe microscopes (SPMs): STM/AFM
  - Working at length scales as small as a single atom
  - Single probe tips limit these tools' ability
    - 1 tip cannot grab an object
    - Electrical measurements cannot be made without a second contact to structures

- Strategies for three-dimensional integration
  - Direct growth of nanostructures on microelectrodes
  - Assembly in liquid solution using flow alignment
  - Direct mechanical assembly using sharp tips
  - Tweezers and grippers
    - Overcome limitations of STMs and AFMs

Structuring of Grippers/Surface

- Gripping at the nanoscale is a challenge
  - Difficult to control balance of forces:
    - Object
    - Surface
    - Tool (gripper)

- Capillary, Van der Waals and electrostatic forces may dominate

- Micro- or nano-objects can stick to a gripper and jump uncontrolled onto it
  - Disturb an automatic assembly

Structuring of Grippers/Surface

- **Several methods have been proposed to control surface forces**
  - **Environmental conditions (pH, T)**
  - **Chemical functionalization**

    } ➲ **Very difficult inside the SEM**
  - **By applying vibration**
    - ➲ **Vibration less efficient and less controlled with decreasing size**
  - **Design of the gripper itself**
  - **Nanostructuring of the grippers/surfaces**
  - **Material composition of grippers/surfaces**

- **Nanostructuring on the gripper arms/surfaces**
  - **Reducing the contact area will lower contact forces and VdW force**
- **Material composition of grippers/surfaces**
  - **Electrically conductive materials can minimize electrostatic forces**

Structuring of Grippers/Surface

Objectives

- Natural lithography
  - Structuring of large substrates
    - Different structures
    - Different materials

- Natural lithography
  - Structuring of grippers
    - Some limitations

- FIB (Focused ion beam) and FEB (Focused electron beam)
  - Structuring of grippers
    - Local and flexible gripper structuring
    - Limited surface area

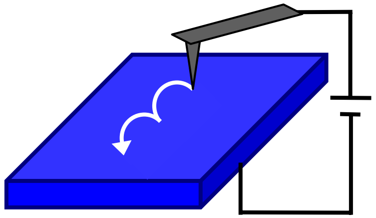
Why Natural lithography?

- **Different lithographic methods for synthesizing NP arrays:**

  Beam lithography (electron, X-ray, ion …etc.)

  SPM lithography (localized electrodeposition)

  ⇨ **Low throughput & High cost equipment**



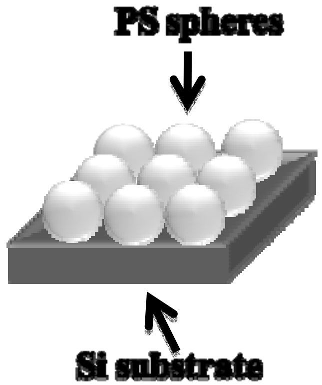
- **Natural lithography using PS spheres:**

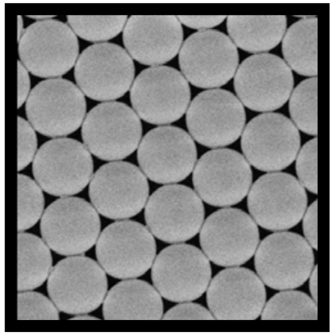
  Electroless deposition (Galvanic displacement)

  Physical vapor deposition (PVD)

  Electroledeposition

  Metal assisted chemical etching

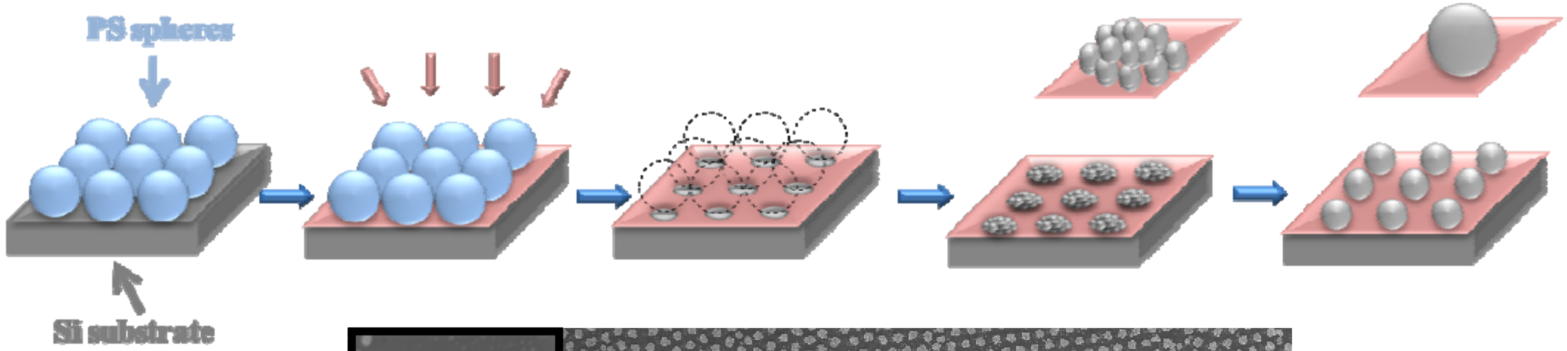


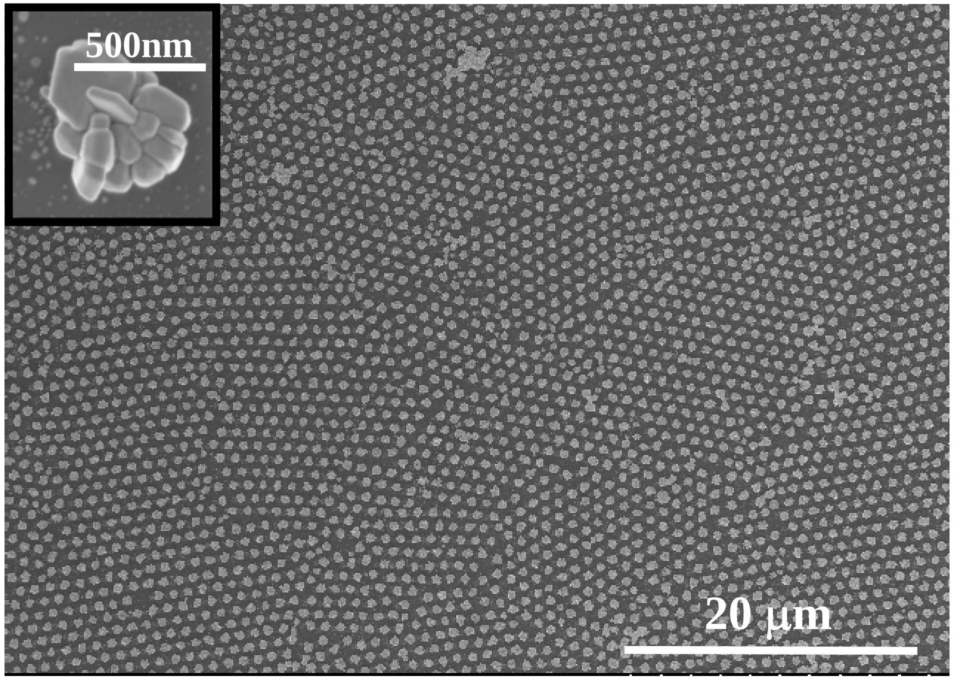


  ⇨ **Easy process, Low-cost & High-throughput**

Patterning of Ag NPs arrays

➢ **Natural Lithography + Electroless deposition**





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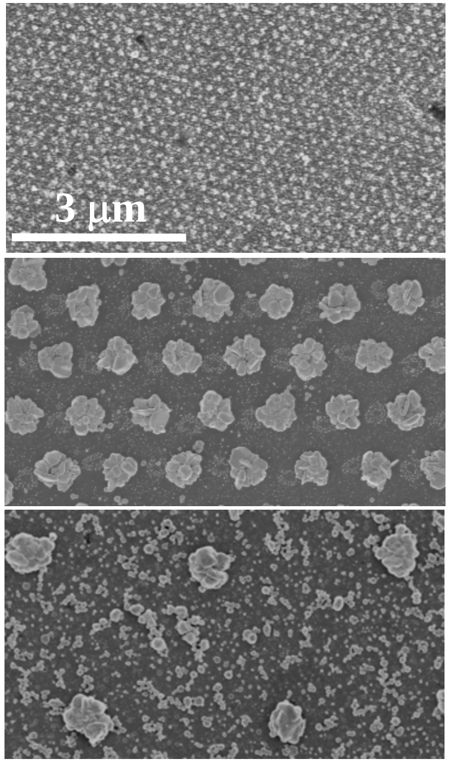
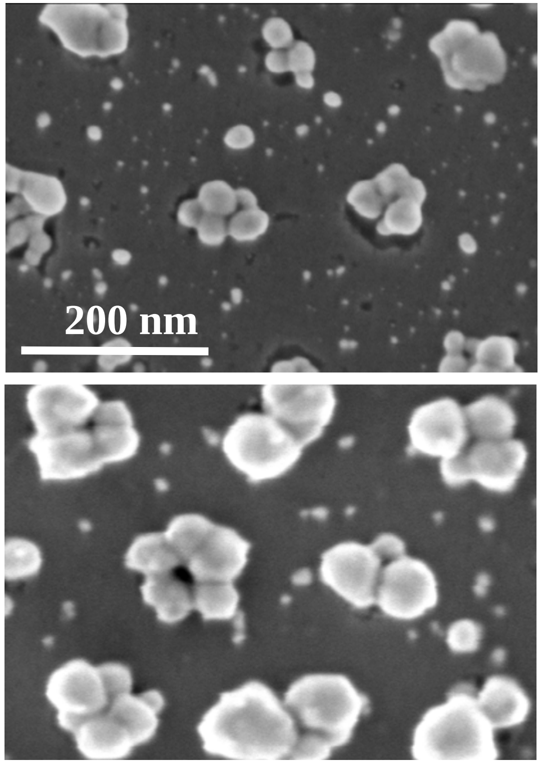
Patterning of Ag NP arrays

➢ **Natural Lithography + Electroless deposition**

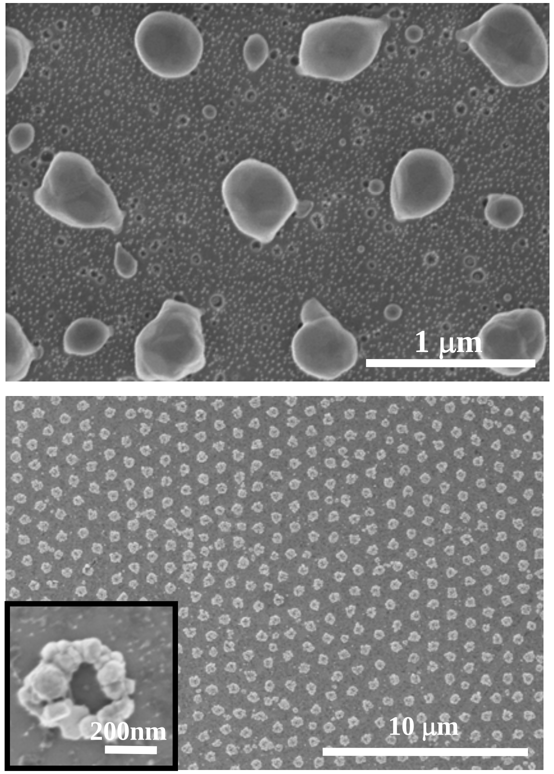
➡ **Density**



➡ **Size**



➡ **Shape**



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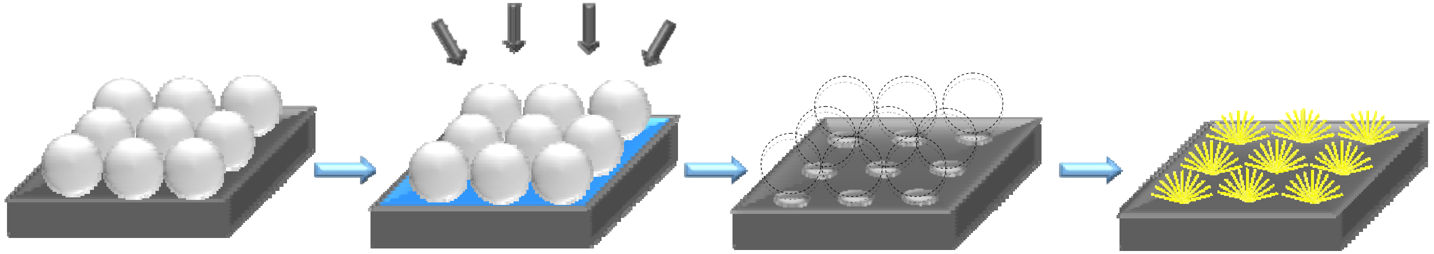
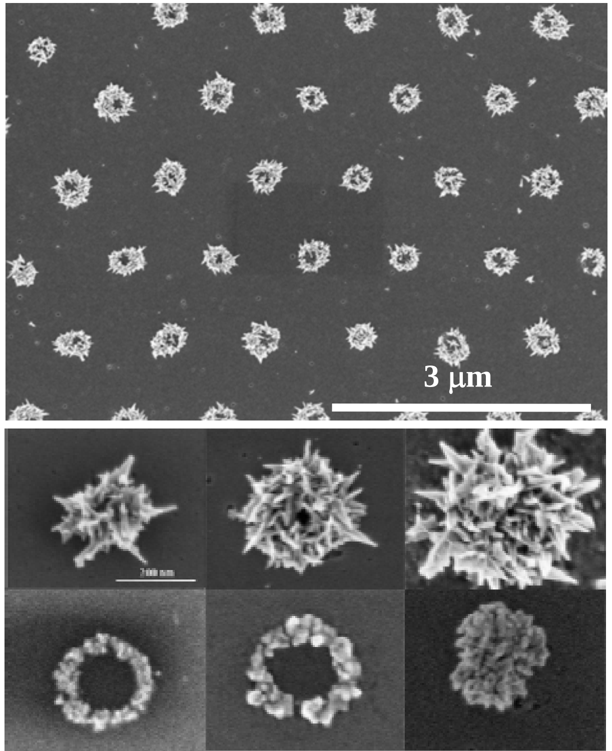
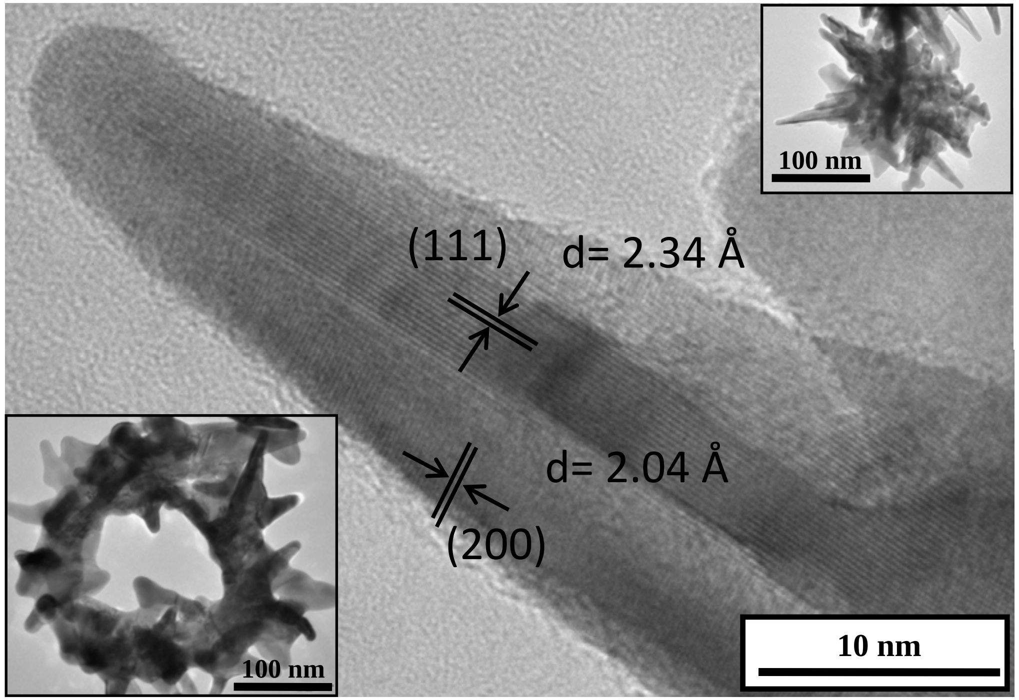
Patterning of Au NP arrays

➢ **Natural Lithography + Electroless deposition**







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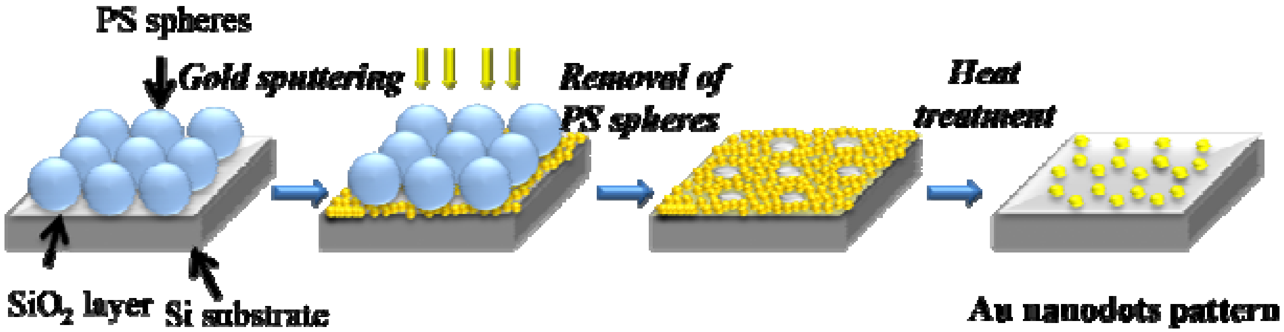
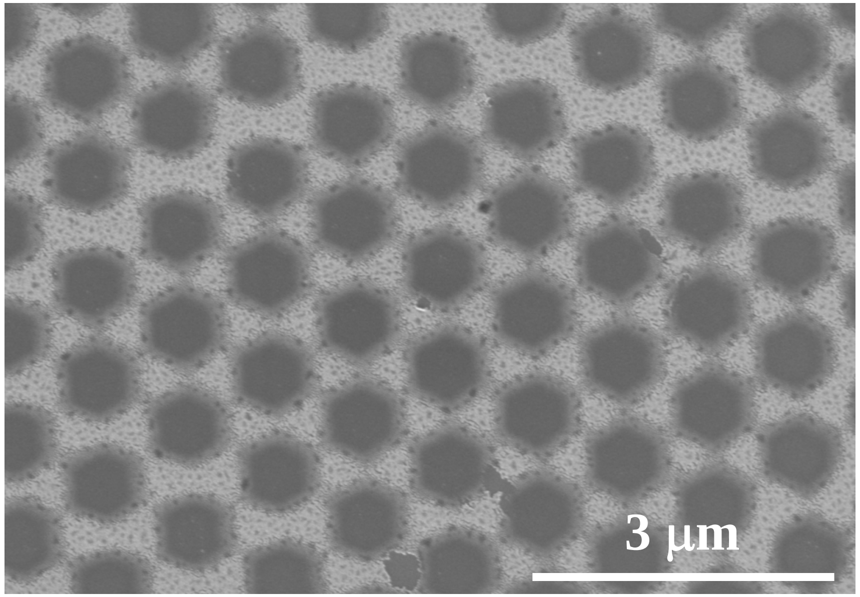
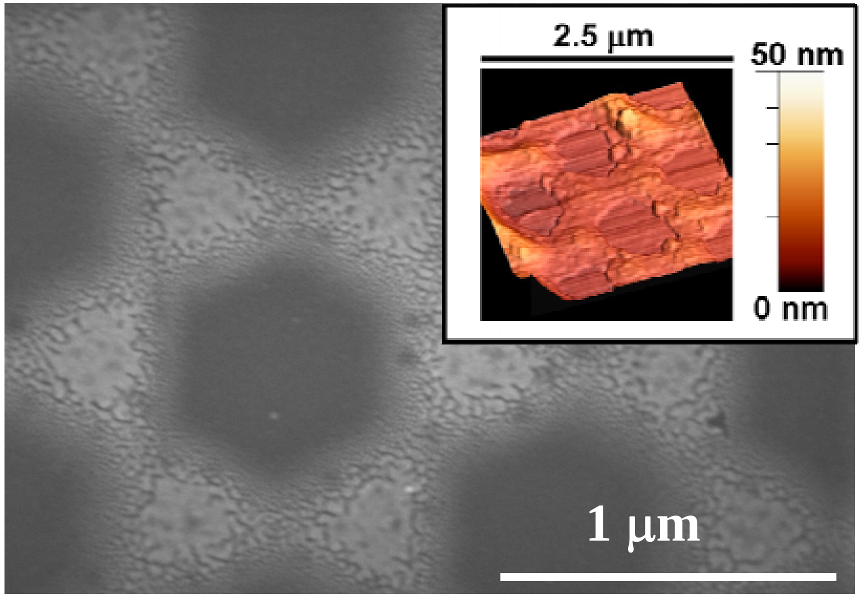
M. Bechelany et al. Accepted in **Langmuir, 2010**

Patterning of Au NP arrays

➢ **Natural Lithography + Physical Vapor Deposition**







M. Bechelany et al. **Crys. Grow. & Desi.** 2010

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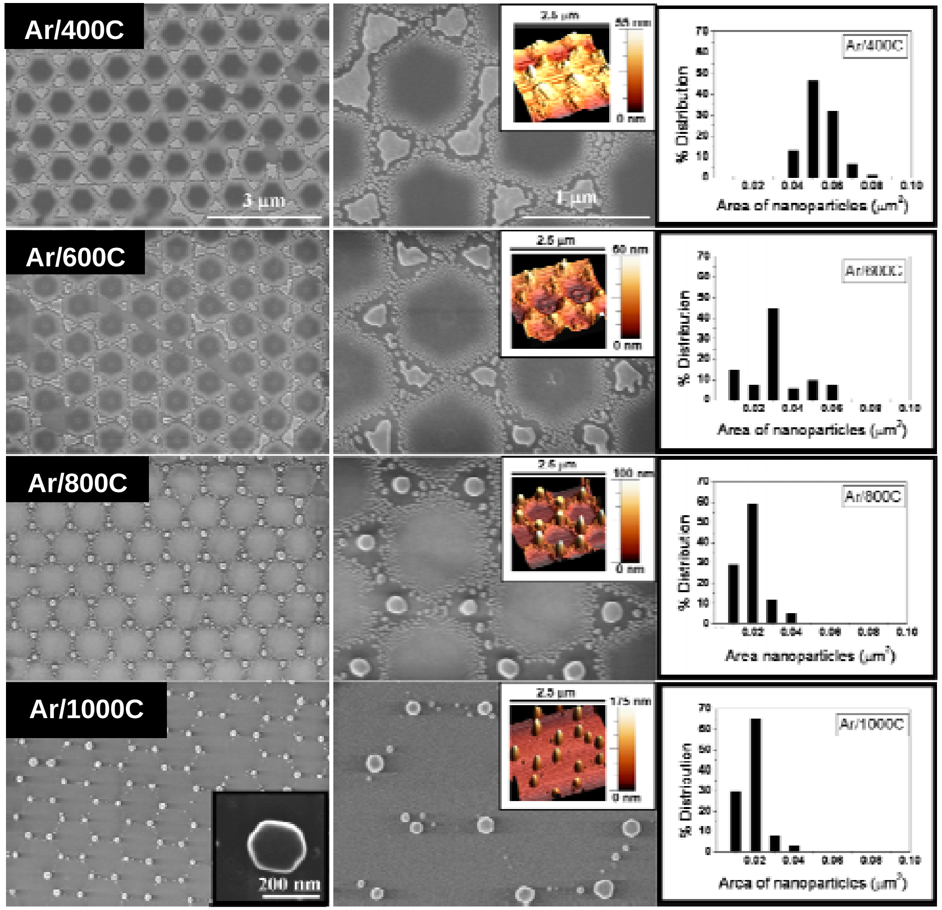
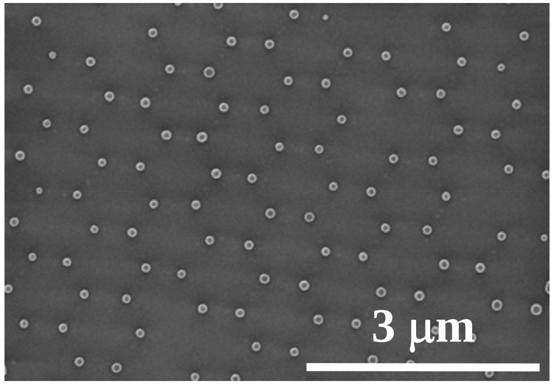
Patterning of Au NP arrays

➤ **2 Synthesis mechanisms: Coalescence &/or Ostwald ripening**

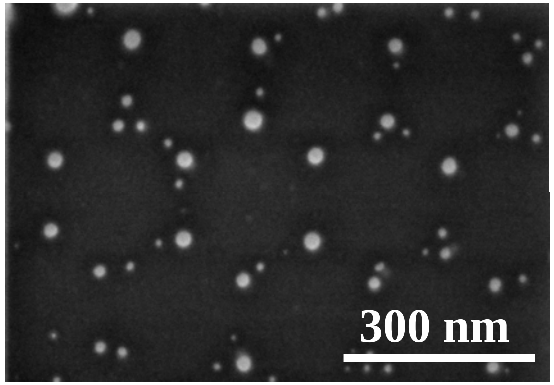
Temperature



Time



PS size



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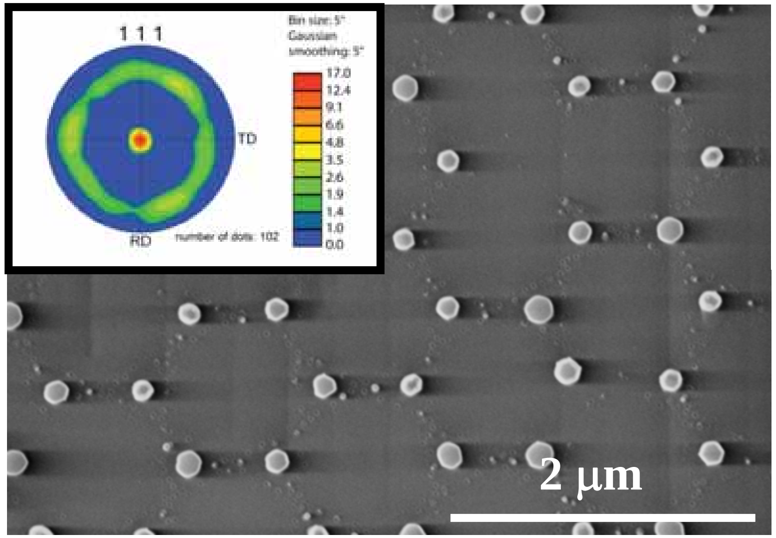
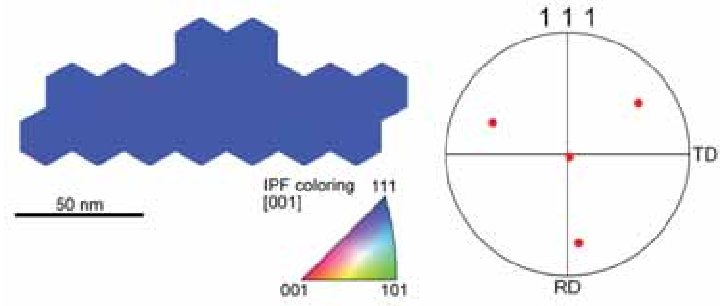
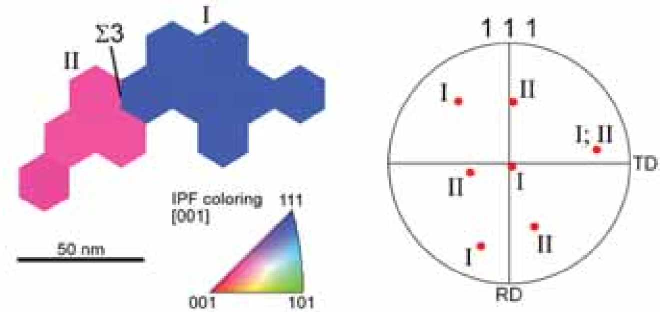
Patterning of Au NP arrays

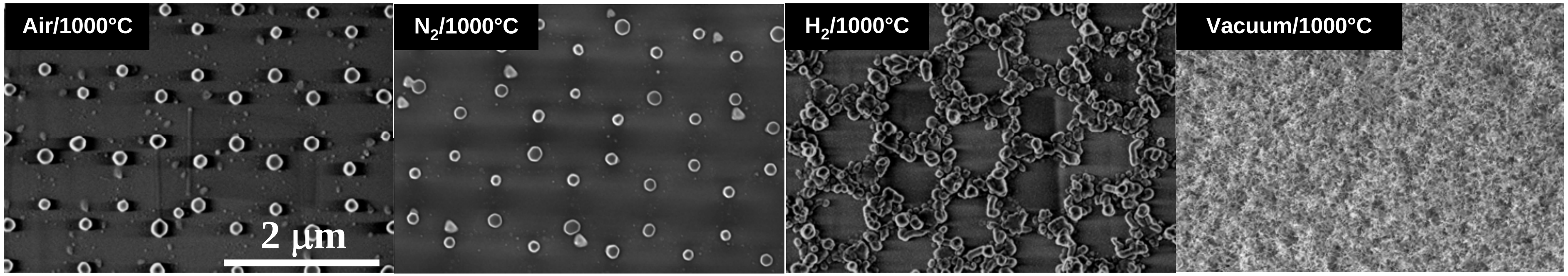
Crystallinity and orientation: Ar/1000C







Atmospheres: Mechanisms, Morphology, & Crystallinity



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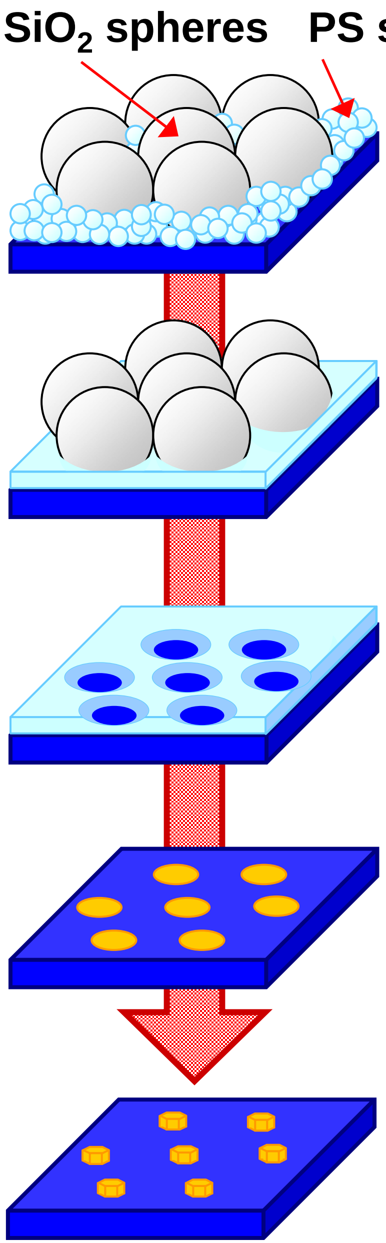
Patterning of Au NP arrays



◆ Self-assembled SiO_2 spheres (ϕ3μm) formed close-packed structure with filled smaller PS spheres (ϕ200nm) on Si substrate

◆ Heating (100°C, 1h) to fix the honeycomb structure was composed with smaller PS spheres on Si substrate

◆ PS honeycomb mask was appeared on Si substrate after removal of SiO_2 spheres by immersing in HF

◆ Au dots pattern produced by PVD (190V, 25mA) through PS honeycomb mask, and then PS honeycomb mask was removed by ultrasonication in toluene

◆ Heating (600 °C, 1h in Ar) to decrease the diameter of

Au dots sizes

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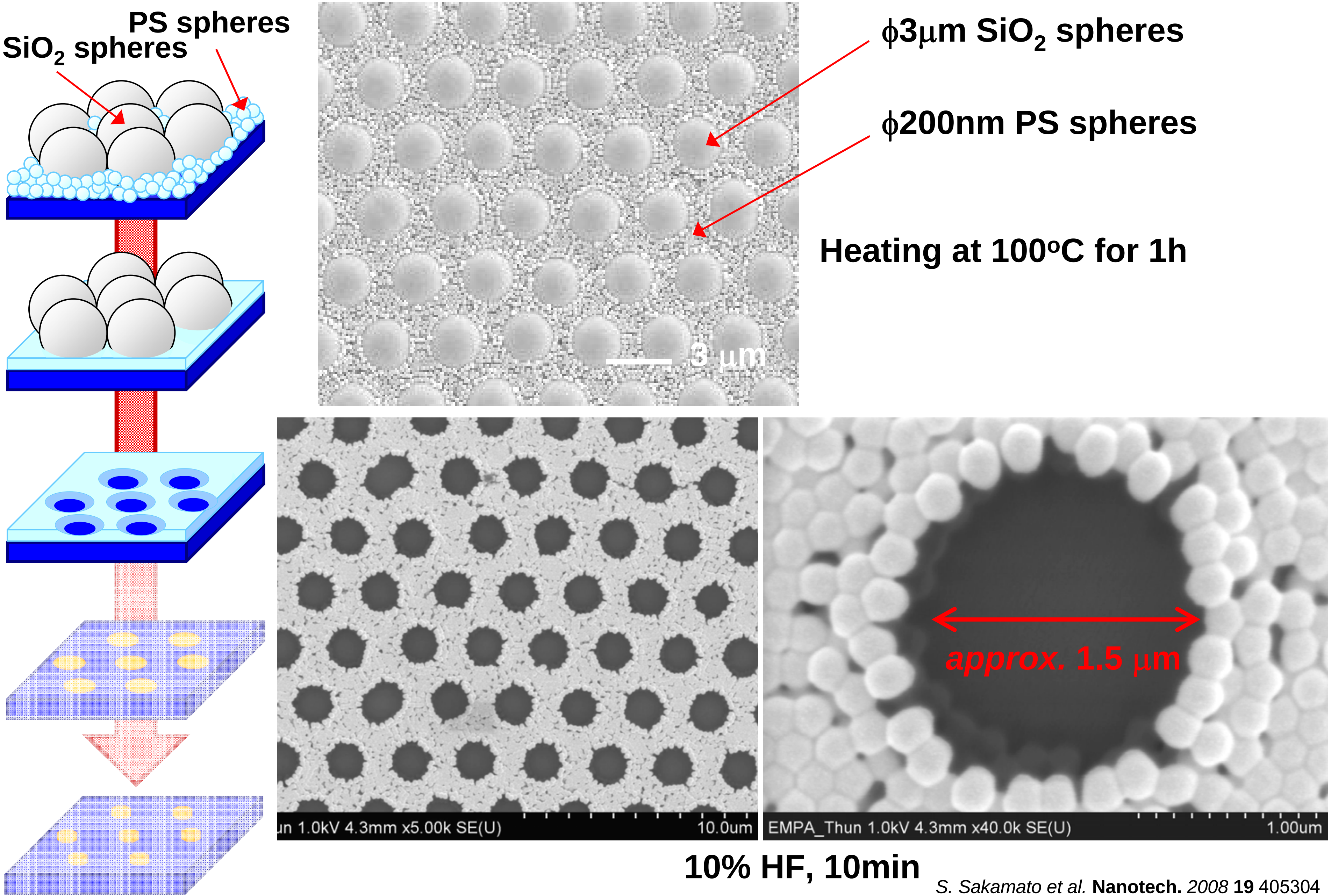
Patterning of Au NP arrays



10% HF, 10min

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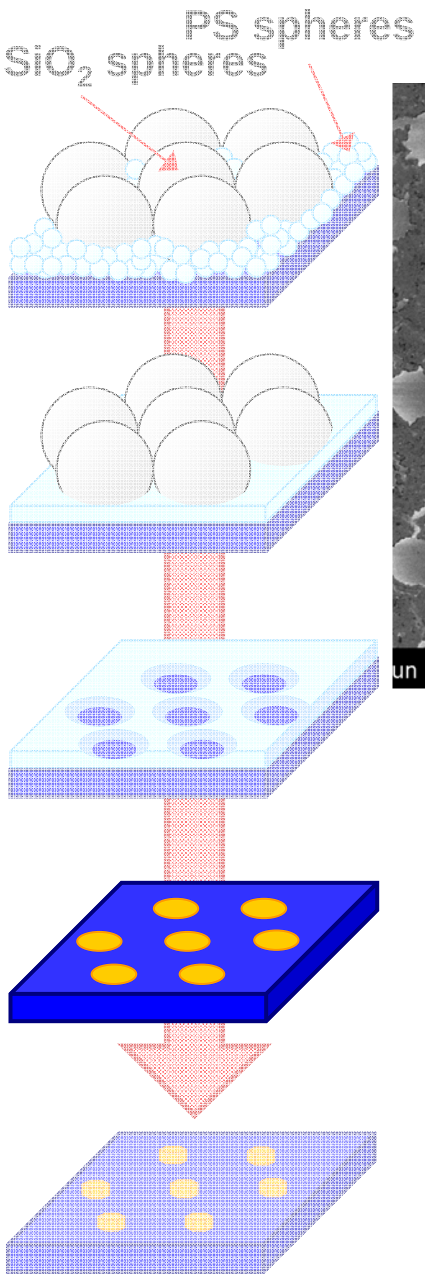
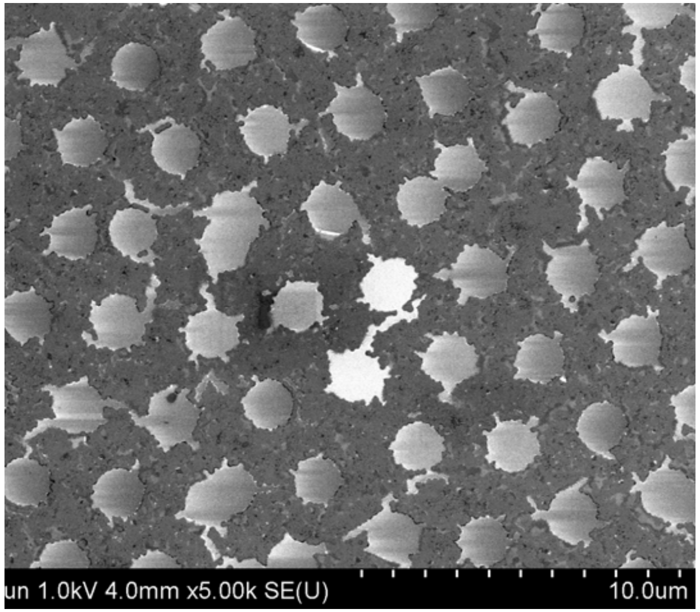
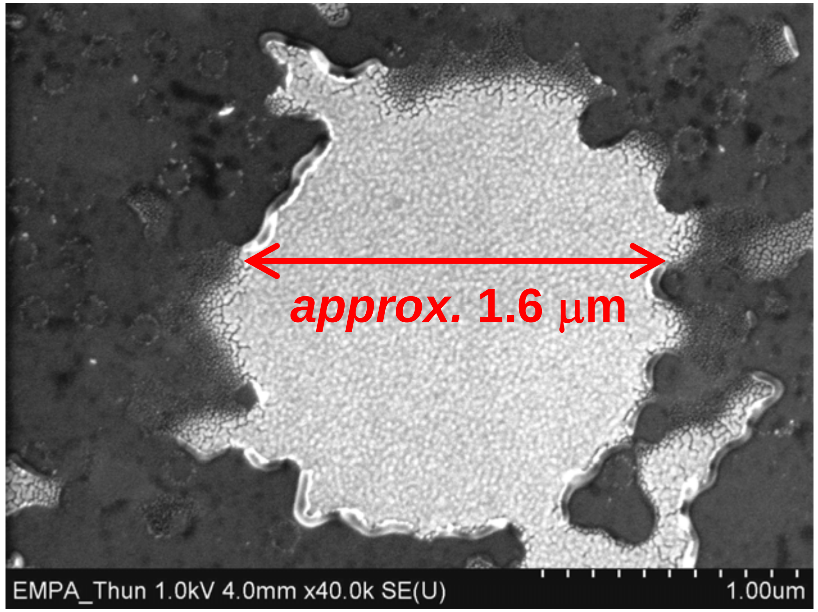
Patterning of Au NP arrays







PVD(190V, 25 mA), 10min

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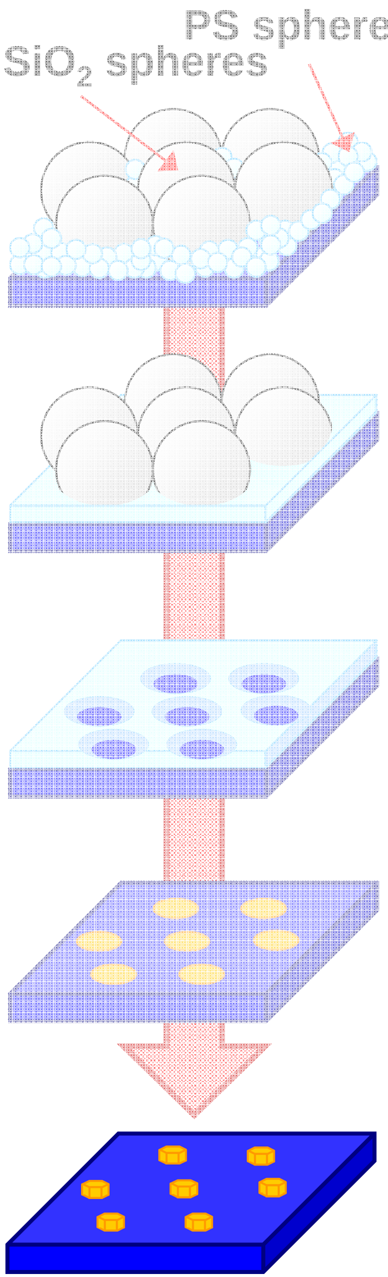
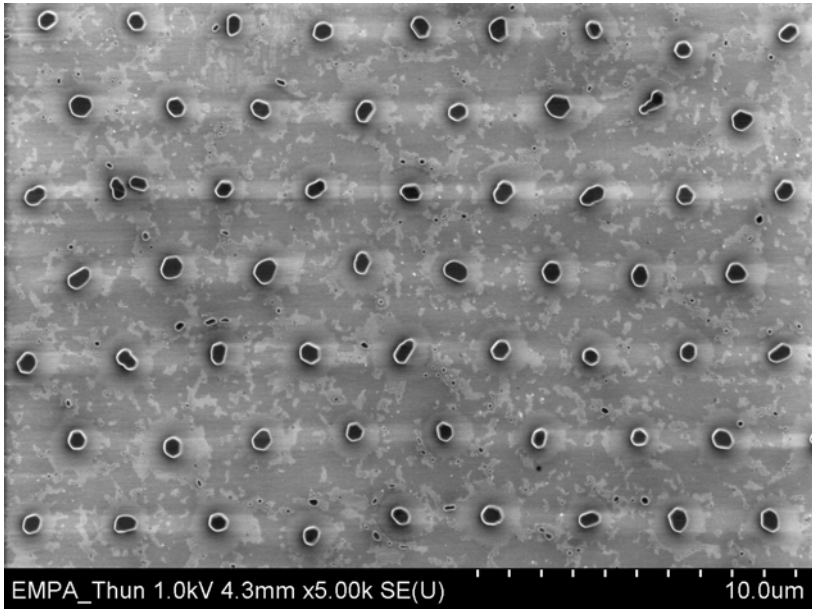
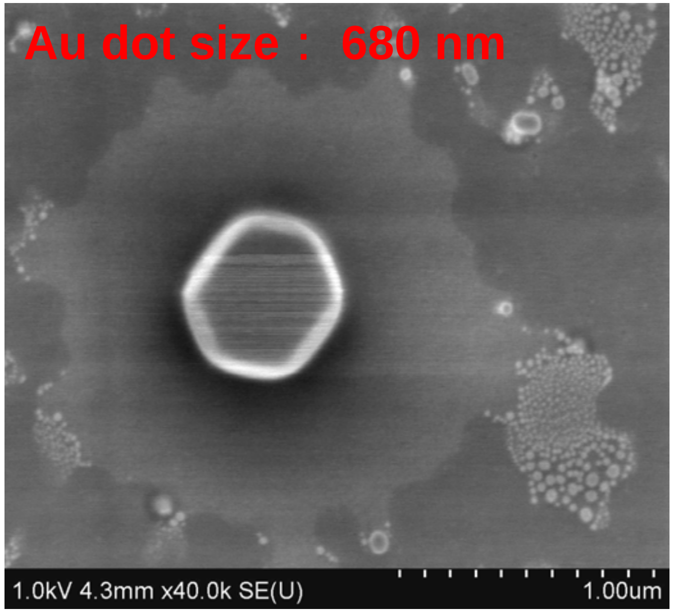
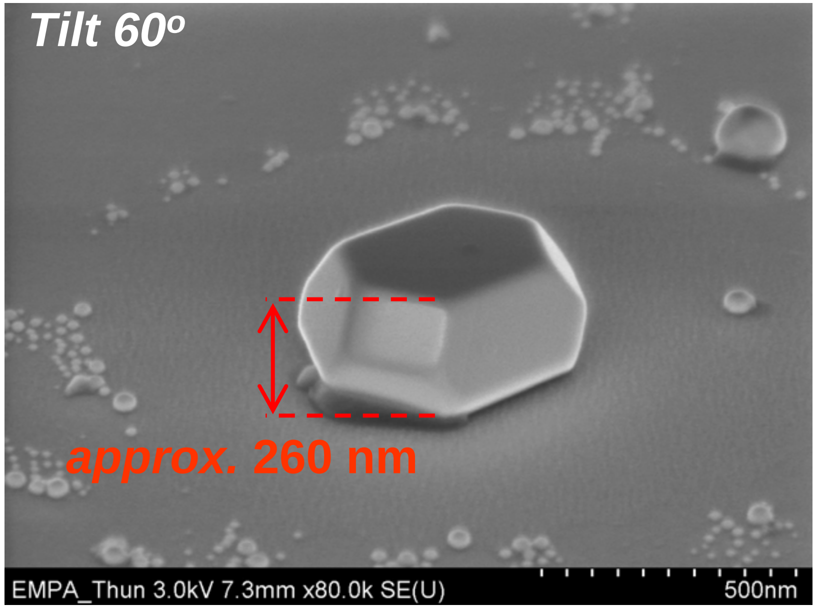
Patterning of Au NP arrays







Heating at 600^{o}C, 1h in Ar



Au dots have approximately 260 nm in height, hexagonal shape.

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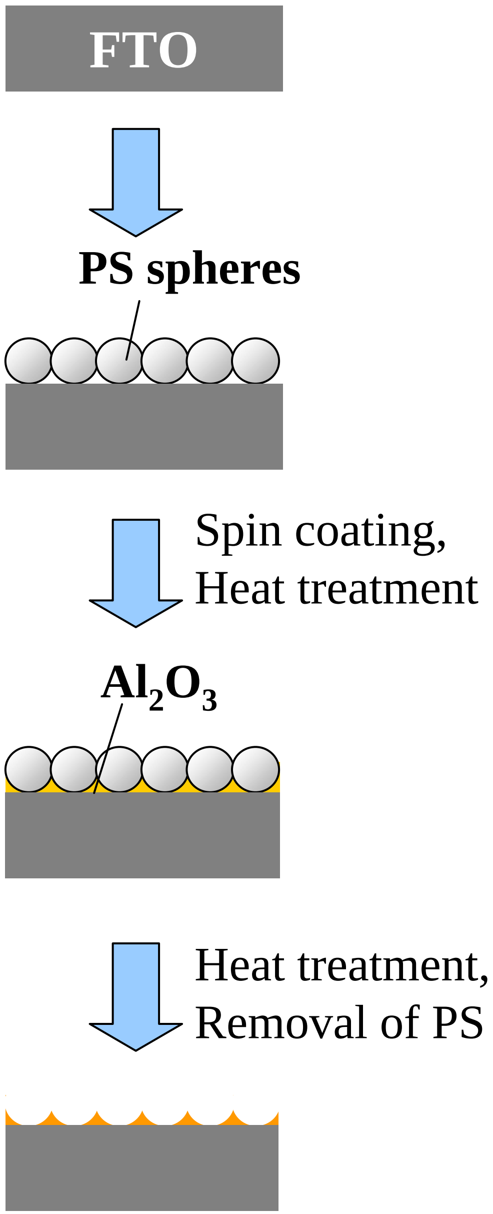
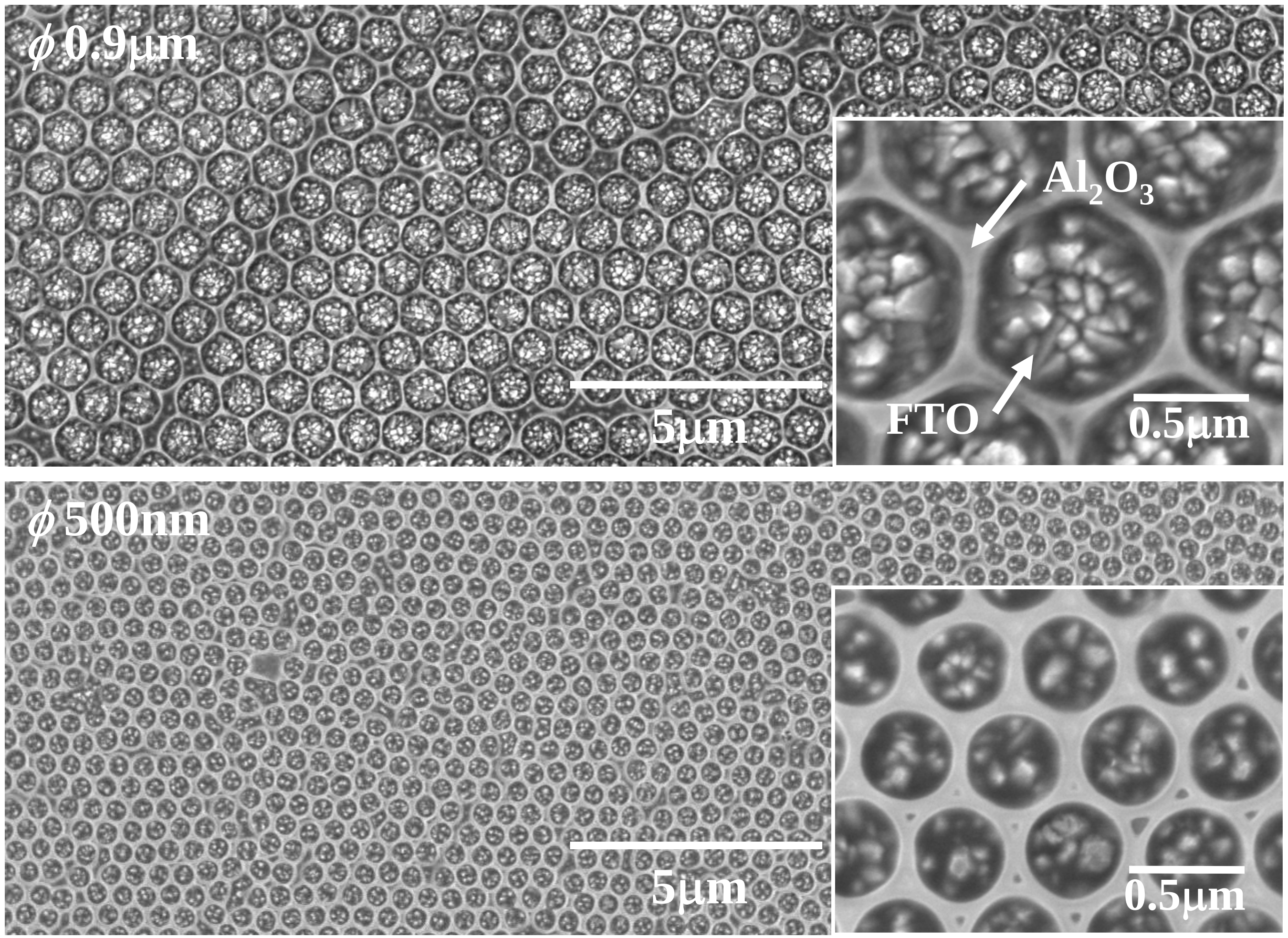
Fabrication of Al2O3 mask on FTO substrate

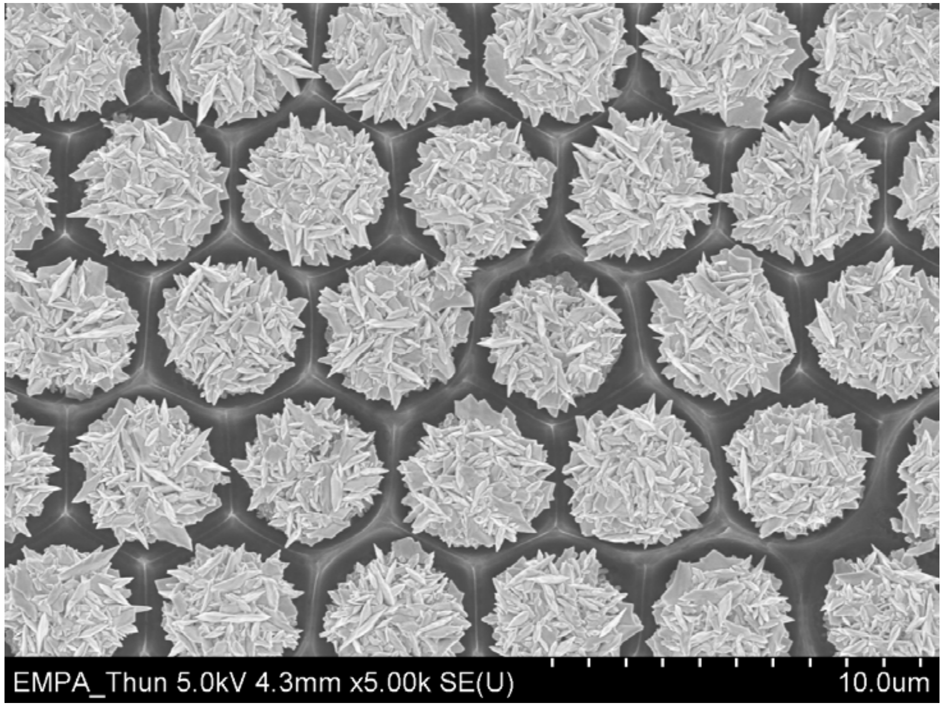


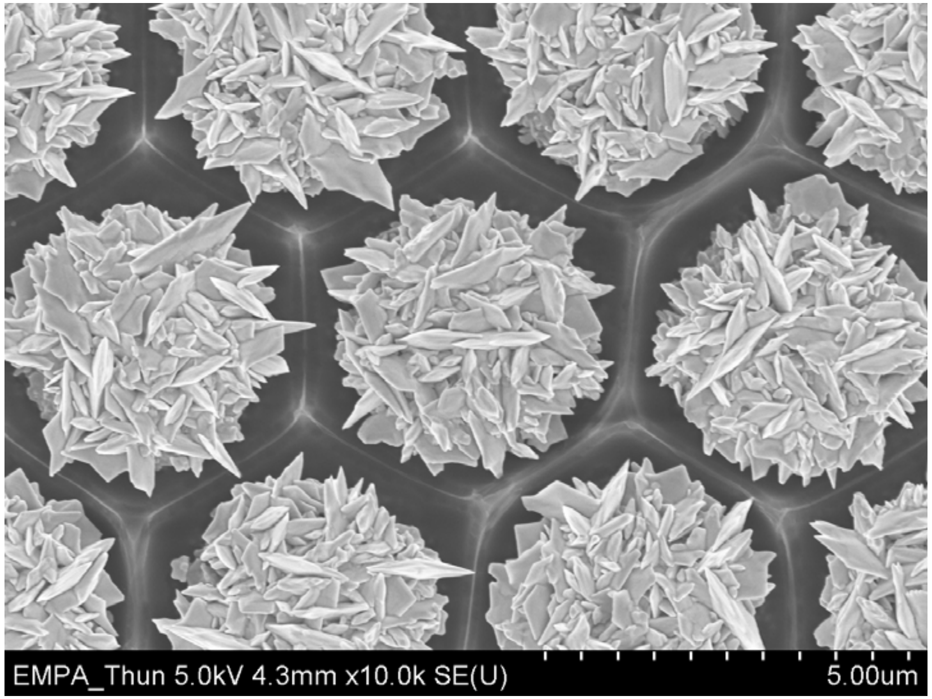


By downsizing the PS spheres used as a template, Al_2O_3 mask with different periodicity can be formed.

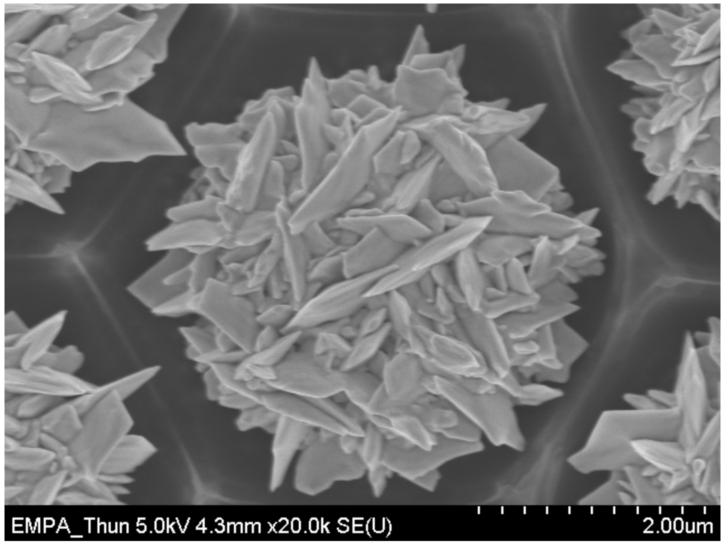
This structure have potential use application as a mask for deposition of metal or metal oxide.

Fabrication of gold flowers by electrodeposition





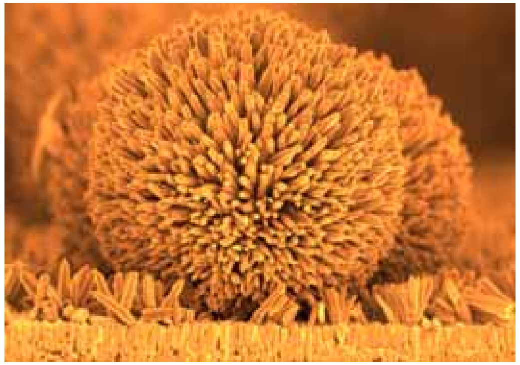
KH2PO4 100 g/l
KAu(CN)2 10 g/l
-1.2 V Ag/AgCl
55 °C

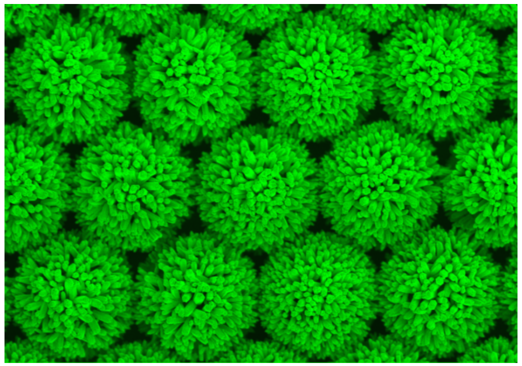


Electrodeposition of hollow ordered urchin-like ZnO NWs

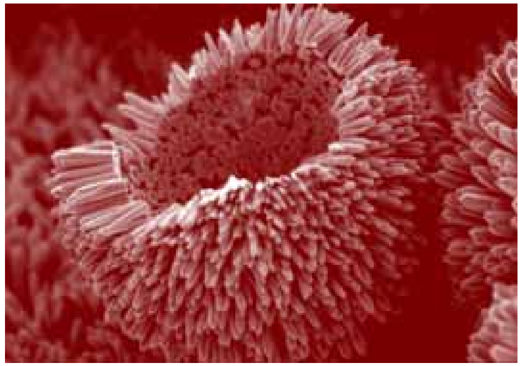
Sea urchin (Eng)
See Igel (De)
Oursin de mer (Fr)

Hollow
ZnO urchin-like





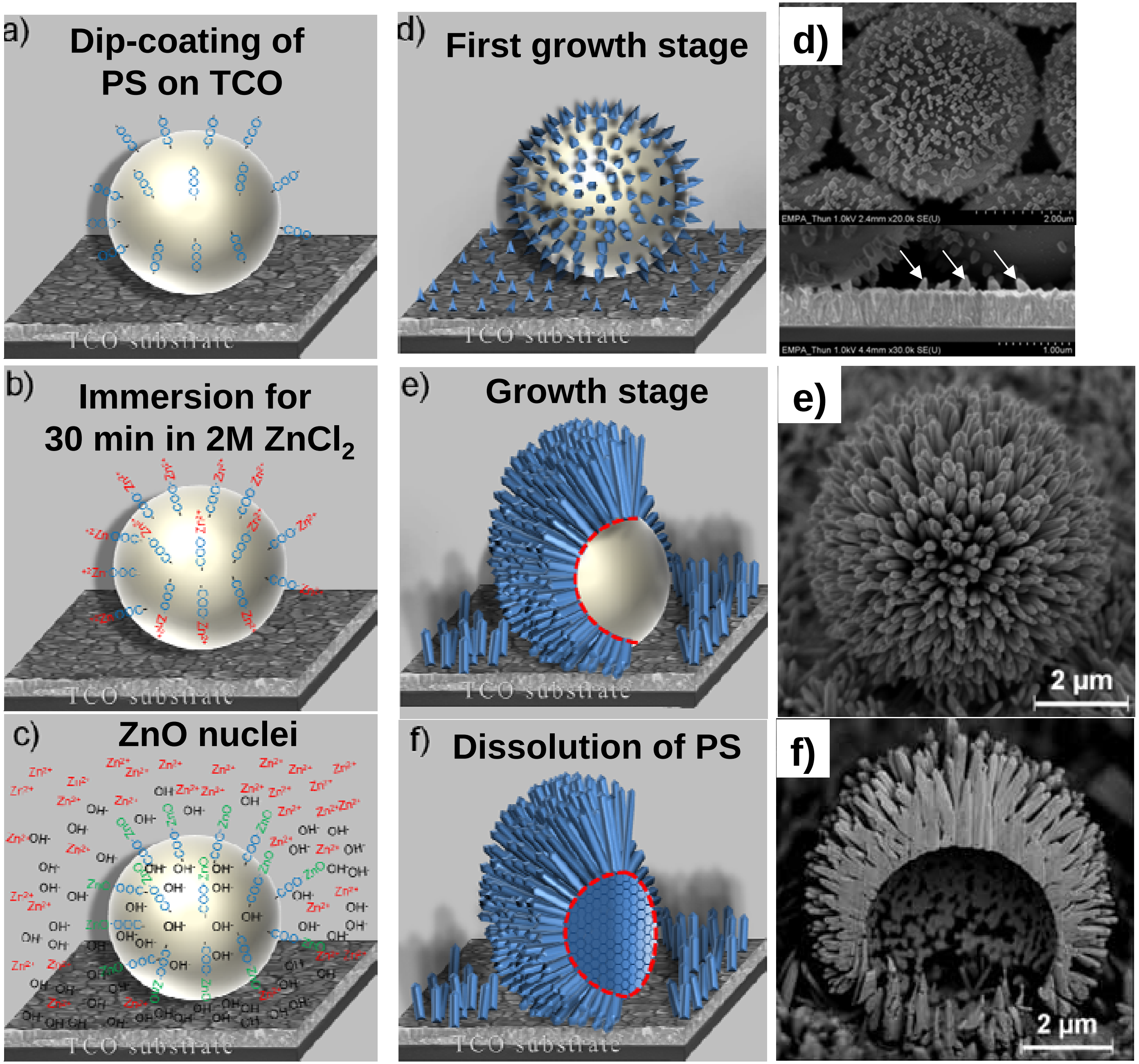
Hollow ordered
ZnO urchin-like



From inside hollow
ZnO urchin-like

Electrodeposition of hollow ordered urchin-like ZnO NWs

Process steps and growth mechanism



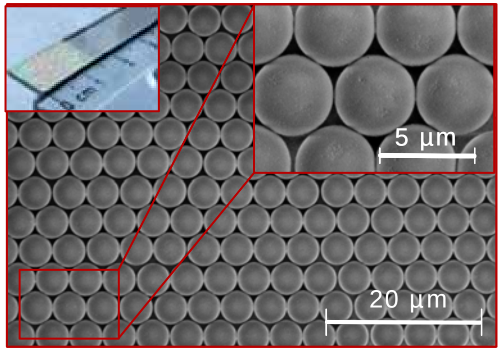
Electrodeposition of hollow ordered urchin-like ZnO NWs

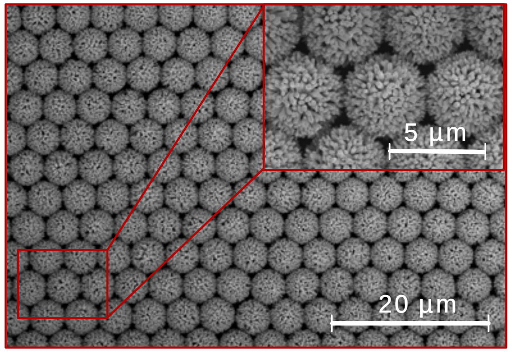
Arrays of mono/multilayers

Before ECD: Ordered Polystyrene spheres

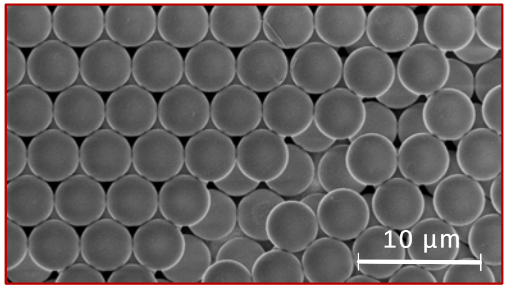
After ECD: Ordered hollow urchin-like ZnO NWs

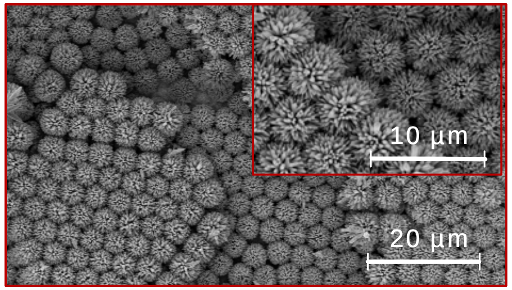
Monolayer

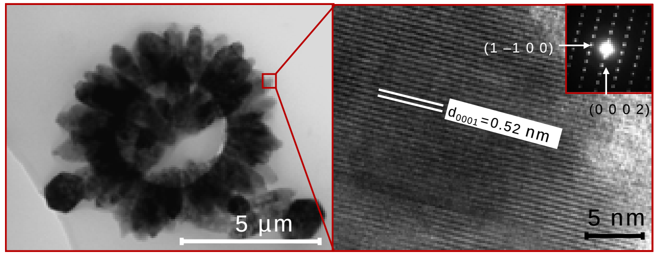




Multilayer

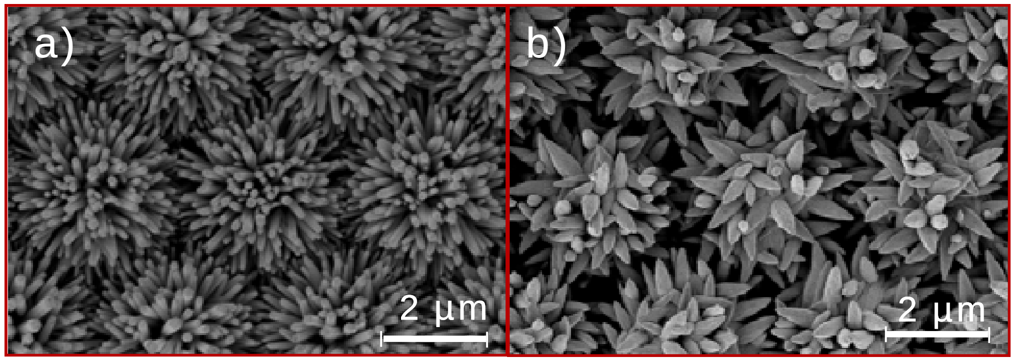






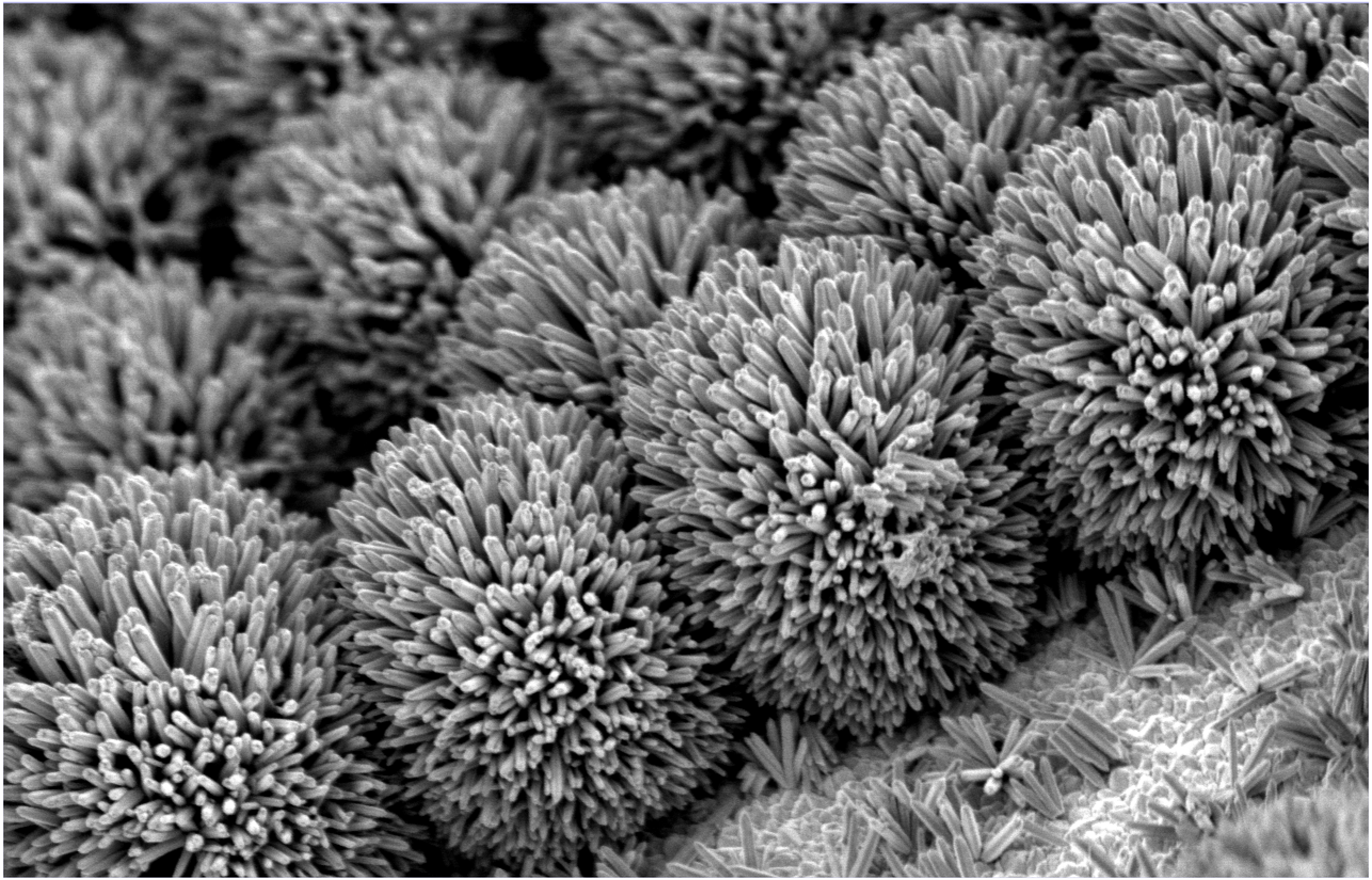
Hexagonal wurtzite structure (c axis along longitudinal NW axis) Single crystal the NWs grow along the [0001] direction.

Ability to tailor the dimensions of NWs

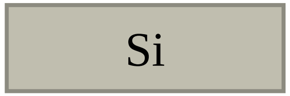


J. Elias *et al.* ***Adv. Mater.*** 2010 DOI: 10.1002/adma.200903098

Electrodeposition of hollow ordered urchin-like ZnO NWs

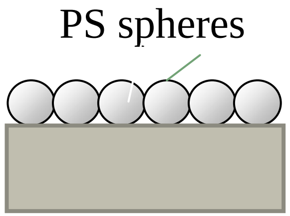


Synthesis of Si NWs by metal assisted chemical etching

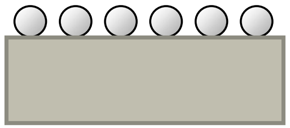


Substrate : n or p-type Si (100) wafers

Pre-treatment : Cleaning in acetone, ethanol, isopropanol (5min)
Rinsed with distilled water →1M HF (5min)
Hydrophilic treatment (H_2O_2/H_2SO_4 and O_2 plasma)

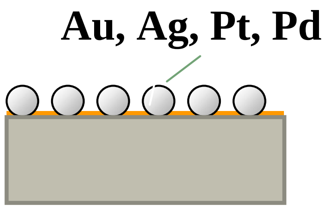


Formation of hexagonal array of PS spheres: Ø 0.2 to 4 μm

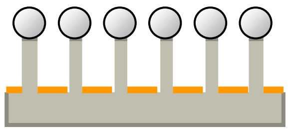


Reduction of the size of PS spheres:

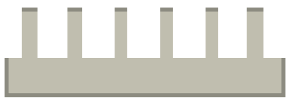
RIE (O_2 plasma) + heat treatment (to fix the spehers)



Deposition of novel metal :
Sputtering or Electroless plating



Fabrication of Si nanowire arrays
Metal-assisted chemical etching in the mixture of HF and H_2O_2
Removal of colloidal crystal and novel metal

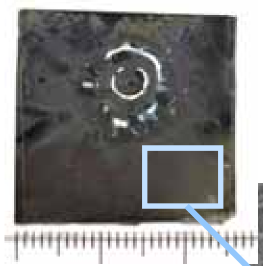


Evaluation of obtained surface structures of Si
Scanning Electron Microscope (SEM)
UV-VIS spectrometer

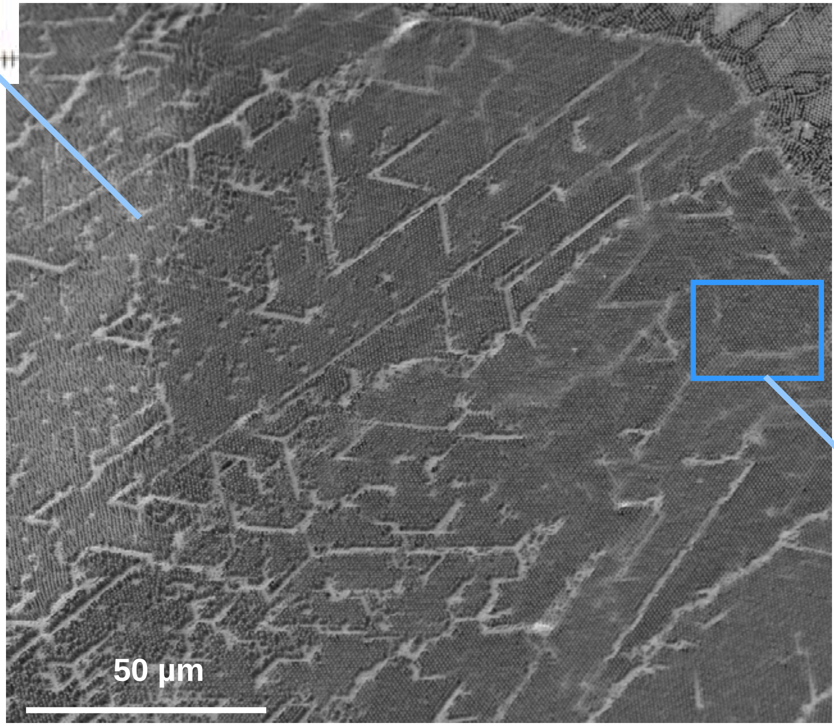
Synthesis of Si NWs by metal assisted chemical etching

Large area of ordered SiNWs

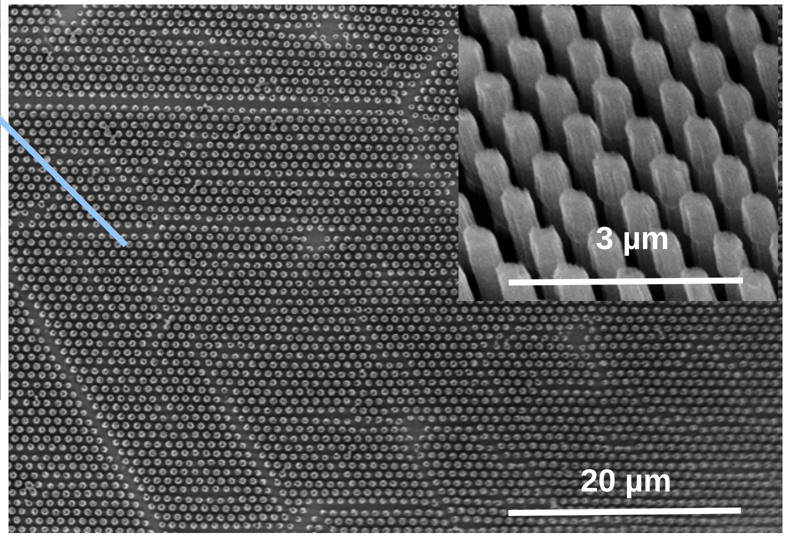
P-Si(100)
Resistivity > 10 Ω.cm
Diameter 400 nm
Length 1.5 µm
Interdistance (center to center) = 900 nm



2.5 x 2.5 cm^2

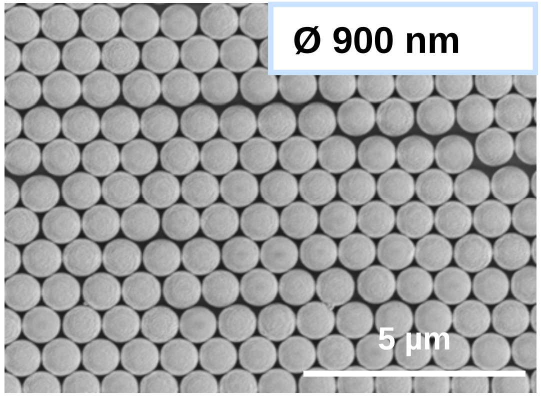


Top view *Tilted view*



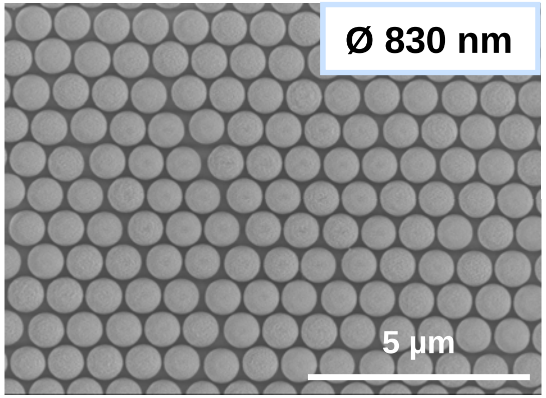
Synthesis of Si NWs by metal assisted chemical etching

Reduction of the sphere diameter

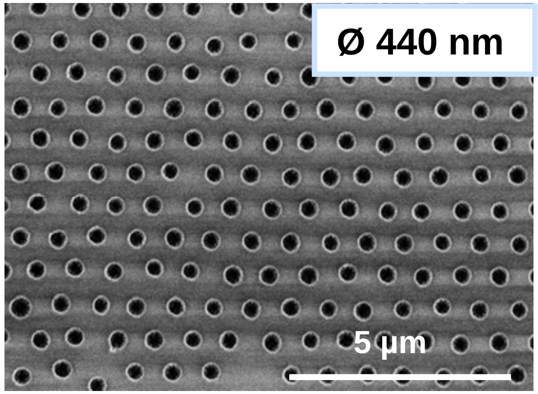


Without RIE

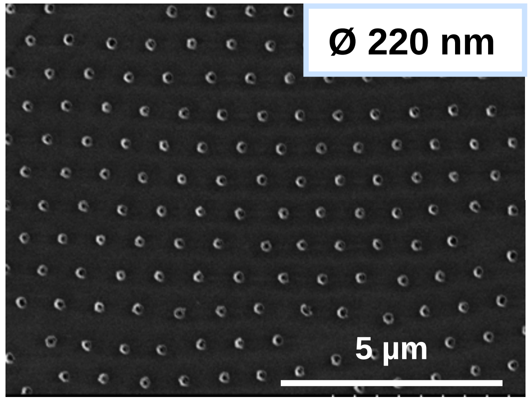
Uniform arrangement
Close-packed structure
on large areas



1 min



5 min

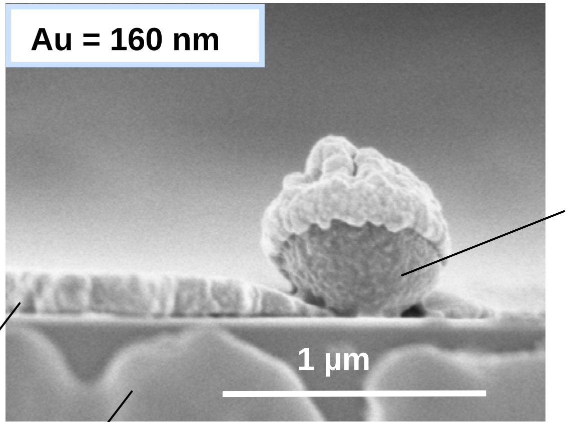


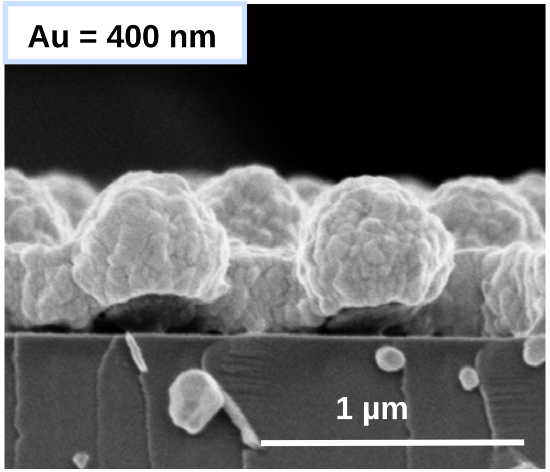
8 min

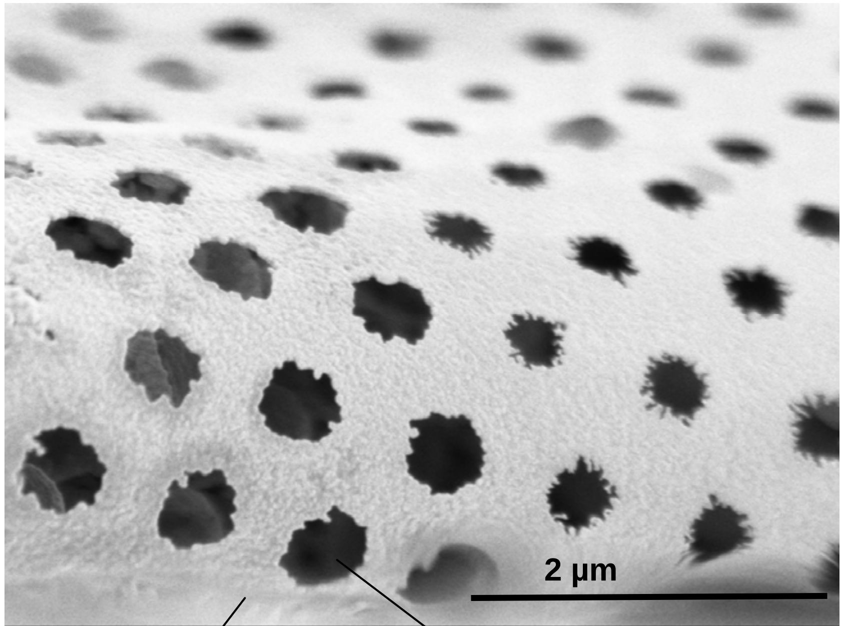
- Reactive Ion Etching process allows diameter

Synthesis of Si NWs by metal assisted chemical etching

Covering by metal sputtering

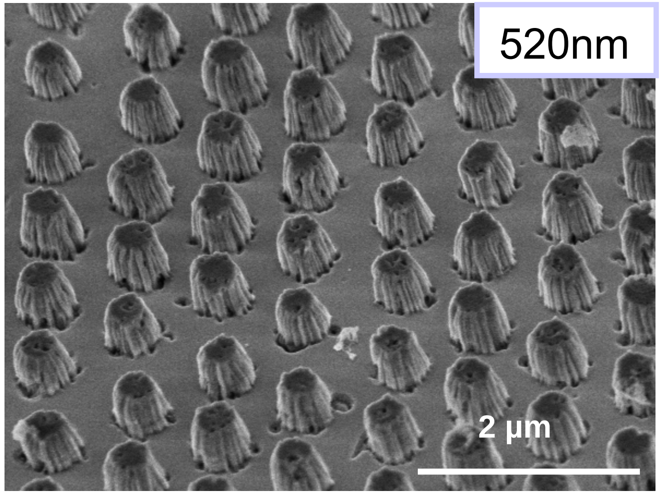


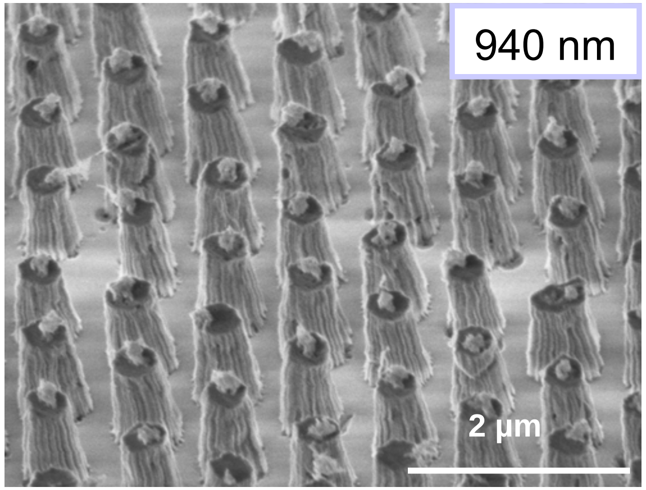




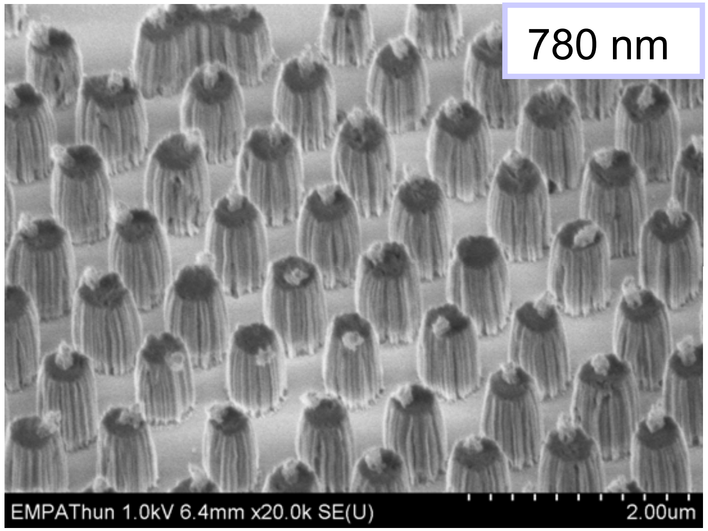
Synthesis of Si NWs by metal assisted chemical etching

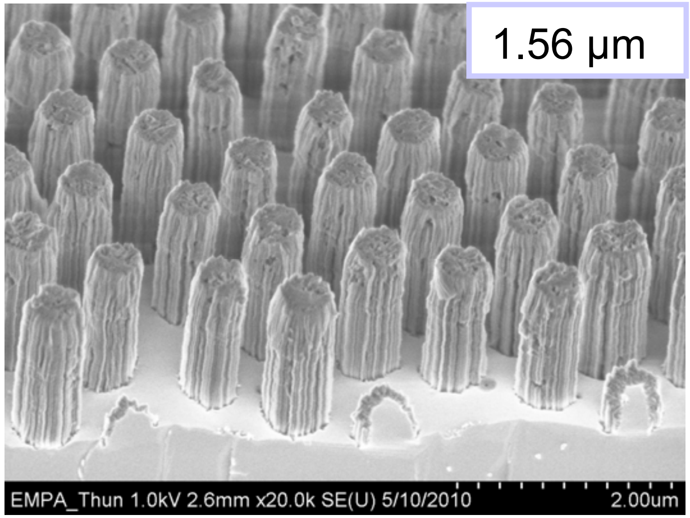
Control of the length





time

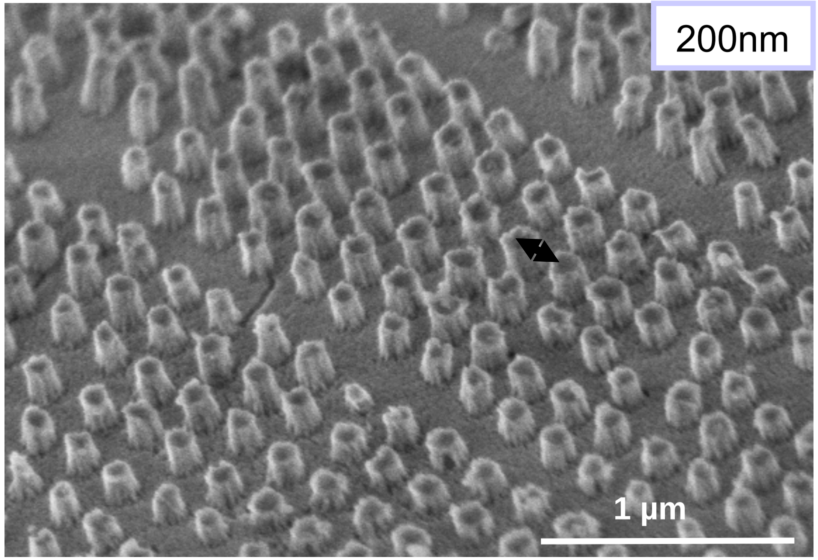




Synthesis of Si NWs by metal assisted chemical etching

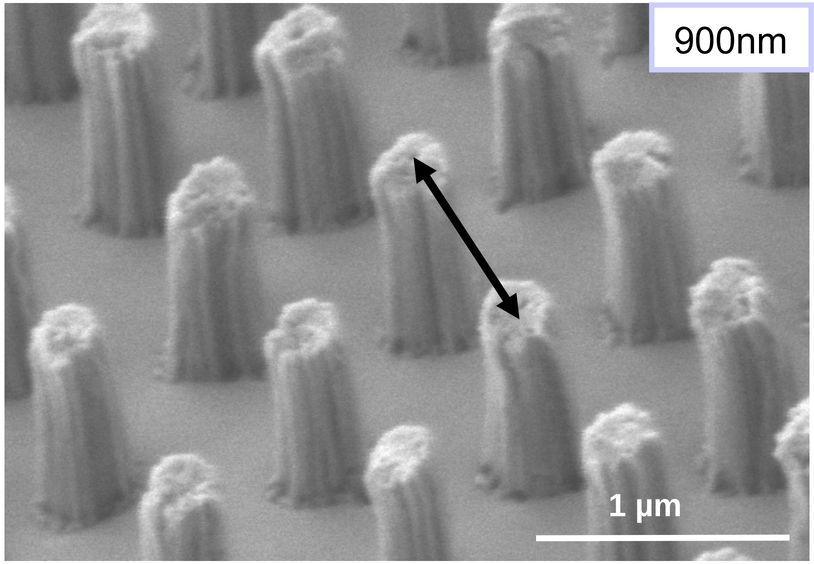
Control of the interdistance and diameter

Spatial density: 6.25 x 10^9 cm^{-2}



SiNWs diameter : 70 nm
Length : 100 nm

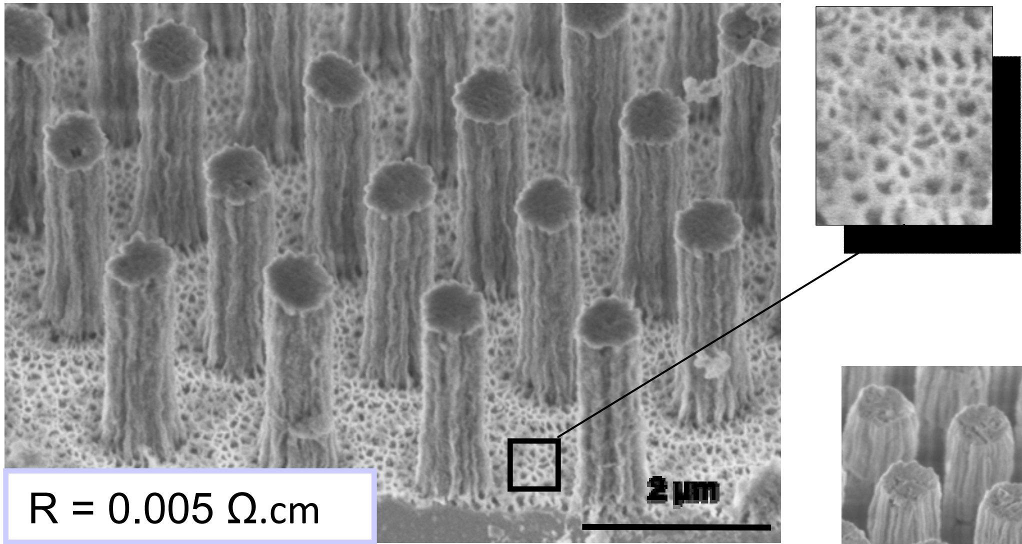
Spatial density: 2.5 x 10^8 cm^{-2}



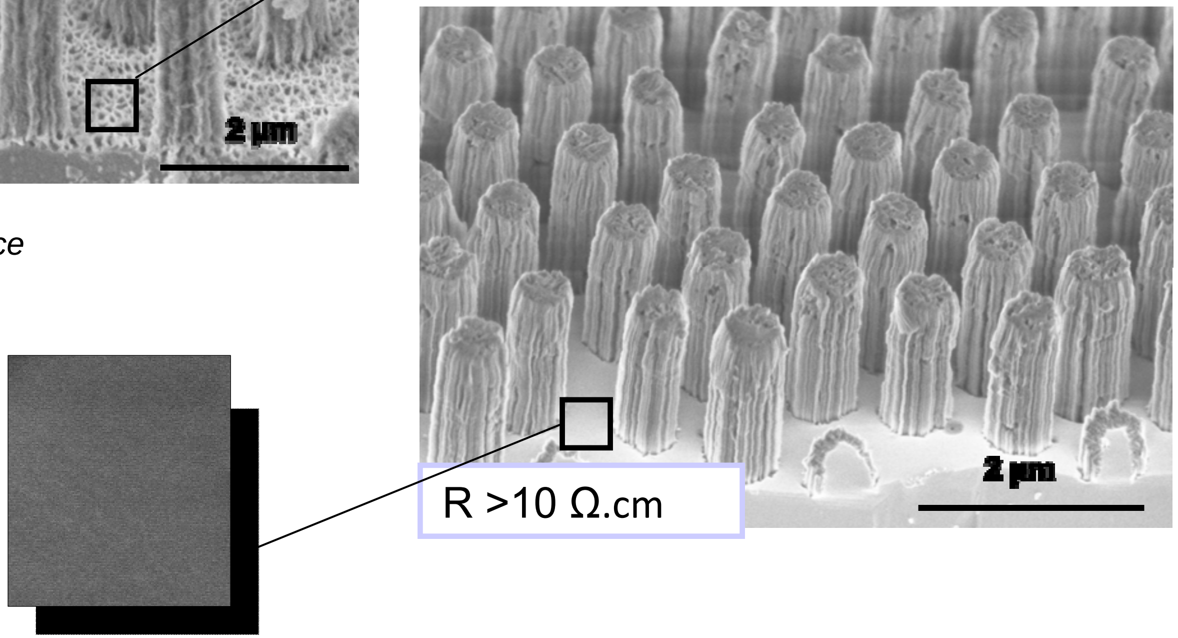
SiNWs diameter : 400 nm
Length : 0.90 µm

Synthesis of Si NWs by metal assisted chemical etching

Control of the porosity



High roughness of the wafer surface



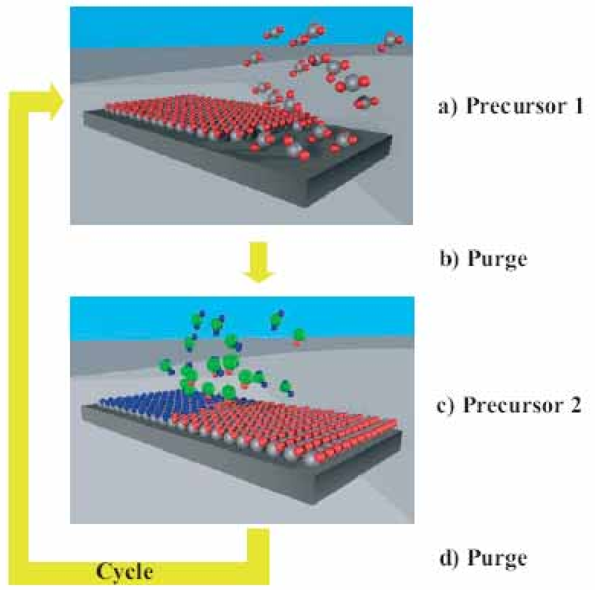
Atomic layer deposition

1) ALD is a thin film deposition technique

2) Sequential use of a gas phase (chemical process)

3) The majority of ALD reactions use two chemicals (precursors)

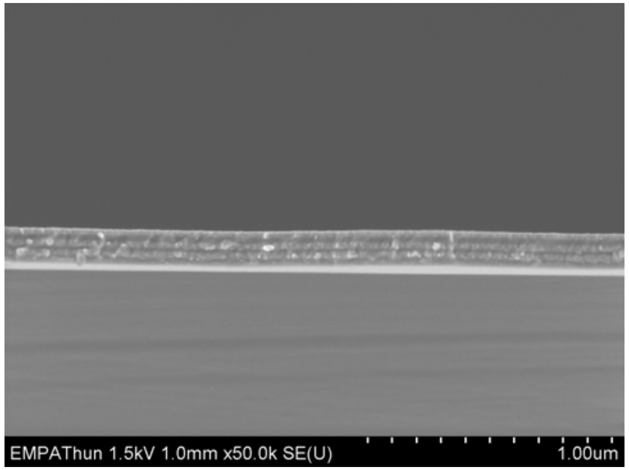
4) Precursors react with a surface one-at-a-time in a sequential manner

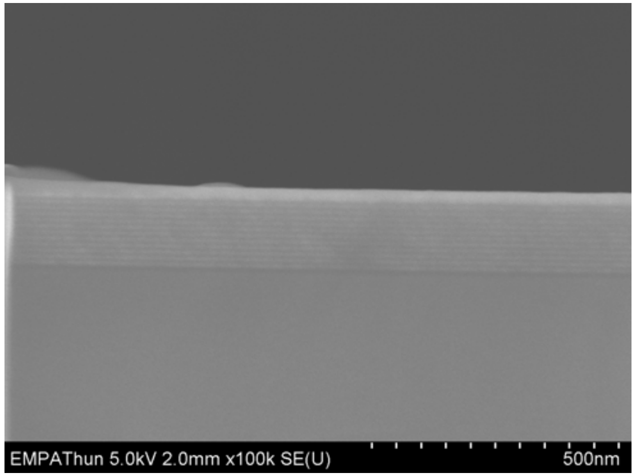


5) By exposing the precursors to the growth surface repeatedly, a thin film is deposited

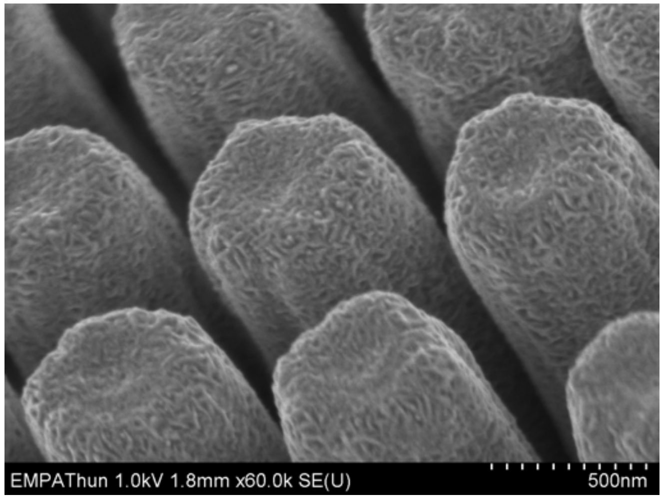
Atomic layer deposition

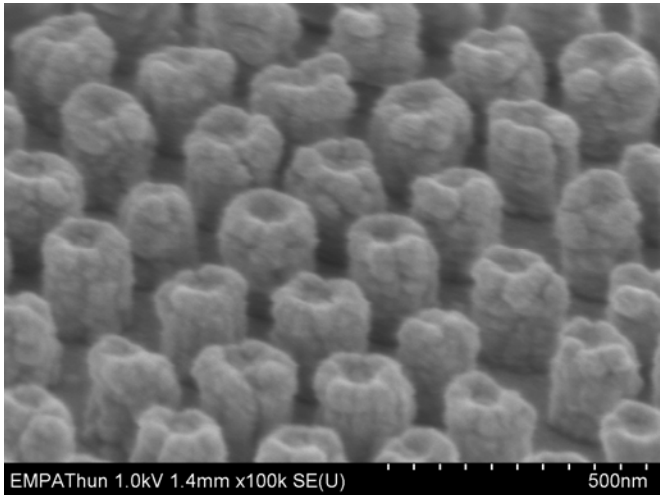
Multilayer of ZnO/Al2O3





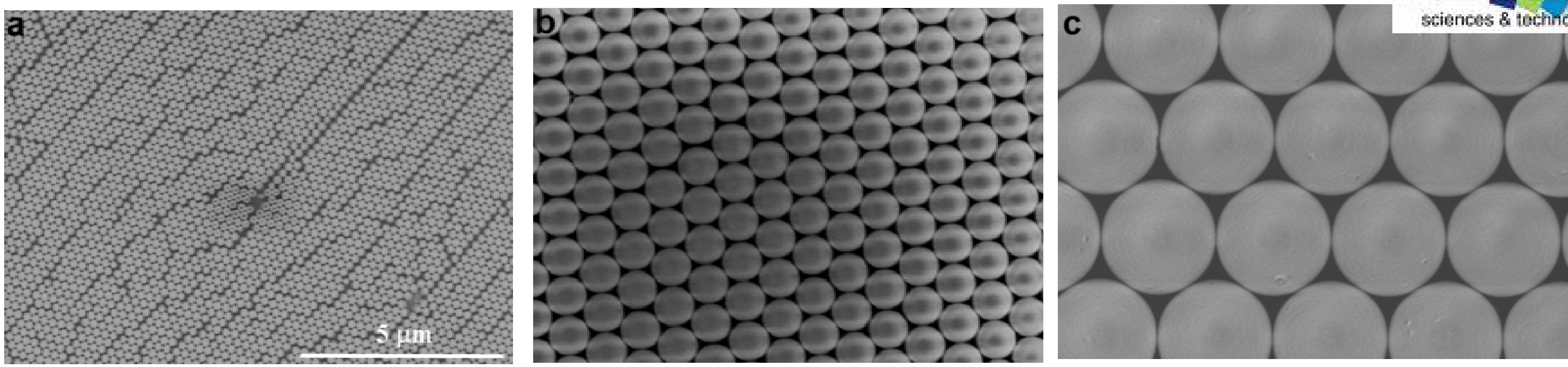
Change the roughness of Si NWs



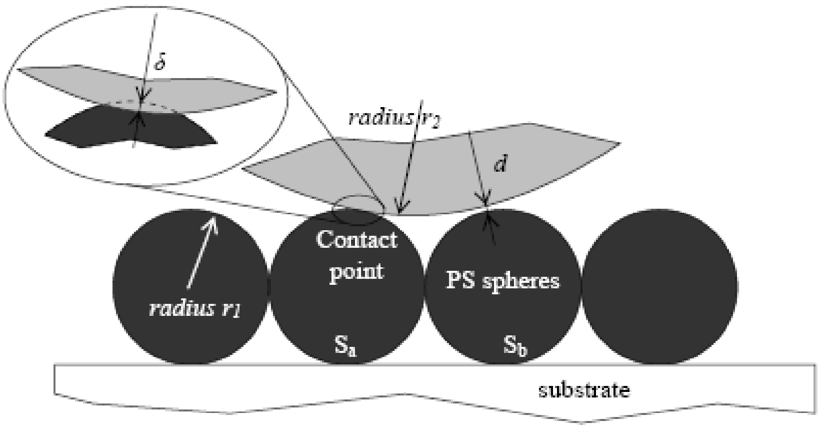


Reducing the adhesion between surfaces

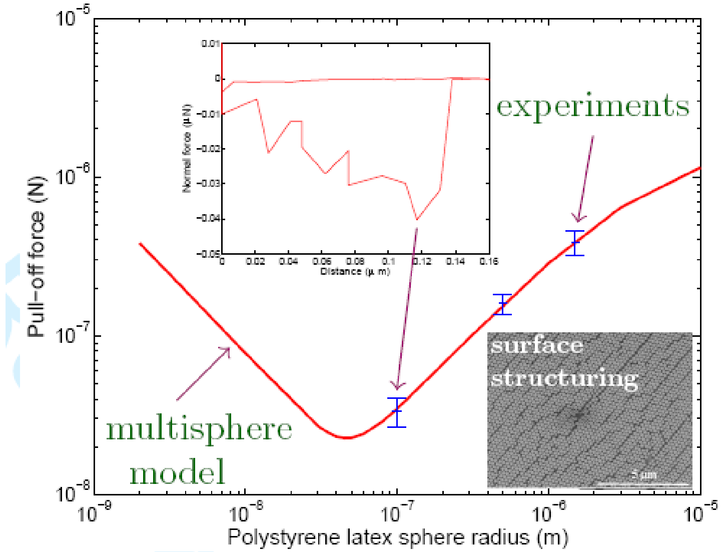
➲ Structuring by PS



Force measurements obtained by AFM compared to a multisphere van der Waals force model



The model suggests the existence of an optimal value of the sphere radius which minimizes the adhesion



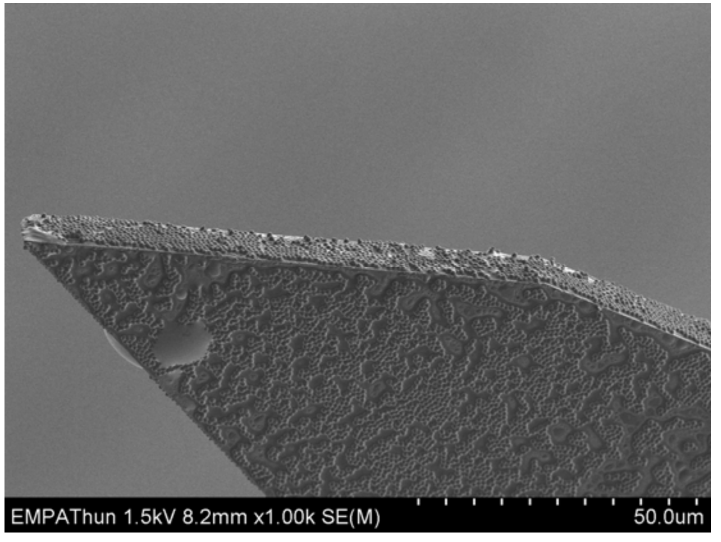
Dejeu et al. ***ACS Applied Materials & Interfaces,*** *2010*

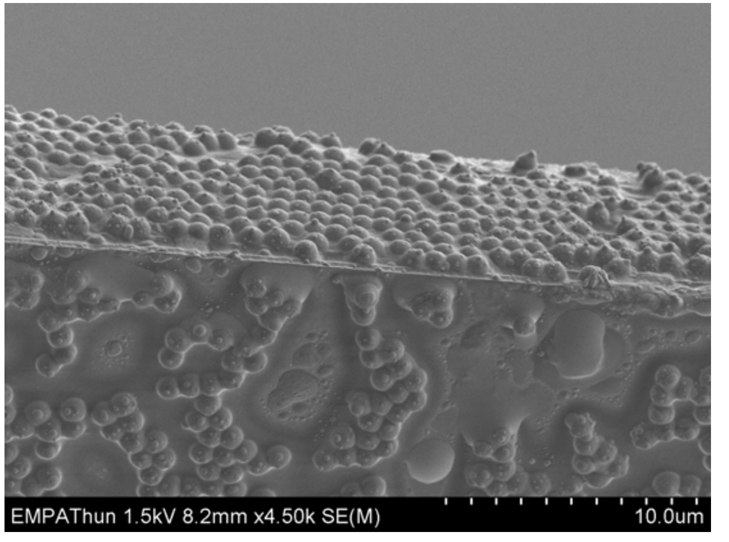
Structuring of Grippers/Surface

- Objectives
  - Natural lithography
    - Structuring of large substrates
      - Different structures
      - Different materials
  - Natural lithography
    - Structuring of grippers
      - Some limitations
  - FIB (Focused ion beam) and FEB (Focused electron beam)
    - Structuring of grippers
      - Local and flexible gripper structuring
      - Limited surface area

Natural lithography for the grippers structuring

- **Grippers Structuring (CNRS, Besançon)**
  - ↳ **Polystyrene spheres**





- **Not localized technique: Deposition of PS spheres on all the gripper**
  - ↳ **Grippers structuring by FEBID and FIB**

Structuring of Grippers/Surface

Objectives

- Natural lithography
  - Structuring of large substrates
    - Different structures
    - Different materials

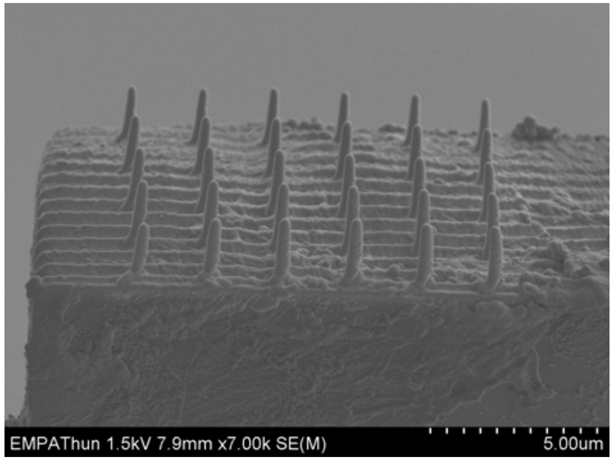
- Natural lithography
  - Structuring of grippers
    - Some limitations

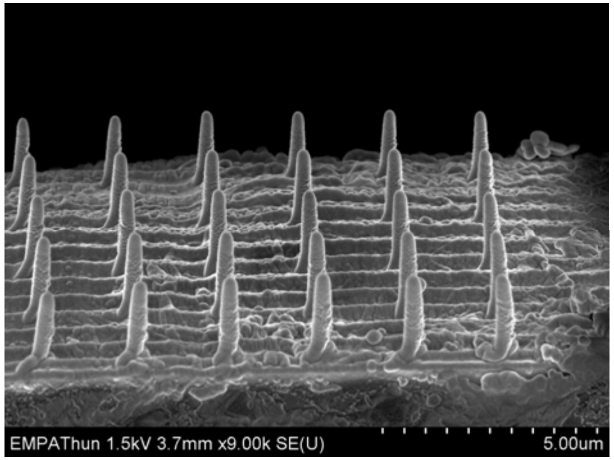
- FIB (Focused ion beam) and FEB (Focused electron beam)
  - Structuring of grippers
    - Local and flexible gripper structuring
    - Limited surface area

Grippers structuring by FEBID

- **Focused Electron Beam Induced Deposition (FEBID)**
  - **Local decomposition of precursor molecules**
    - **Deposition of metals/Carbon at the desired positions, which can be controlled by the position of the beam irradiation**

- **Au plots on CNRS grippers by EBID technique**

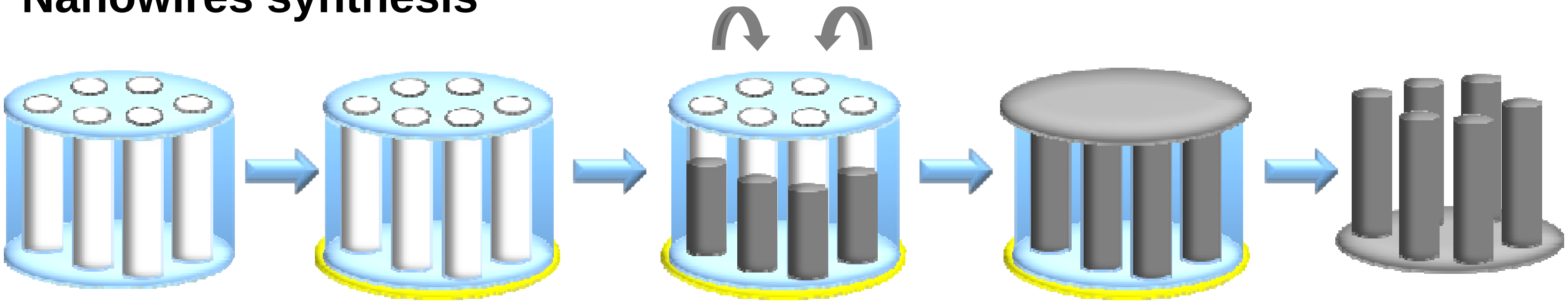




- **Should be tested for manipulation of microspheres (CNRS)**

Structuring of grippers with metallic NWs attachment

➢ Nanowires synthesis



⇨electrochemical deposition:

❶ Al_2O_3 porous membrane
pore size = 200 nm, thickness = 60 μm

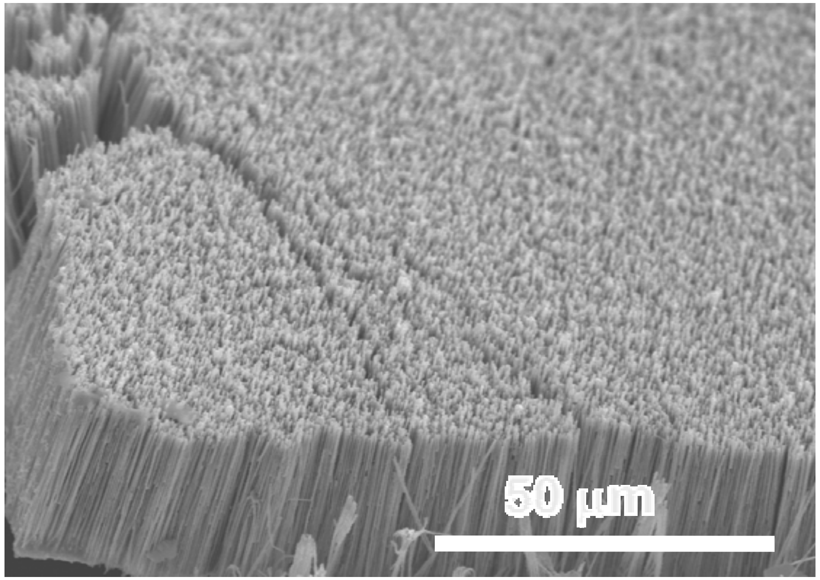
❷ gold layer evaporated onto one side
PVD, thickness = 200 nm

❸ 0.05 M Ag_2SO_4 + 2.3 M KSCN aq
pH = 6.0-6.5

❹ electrochemical deposition
-250 mV until pores are filled

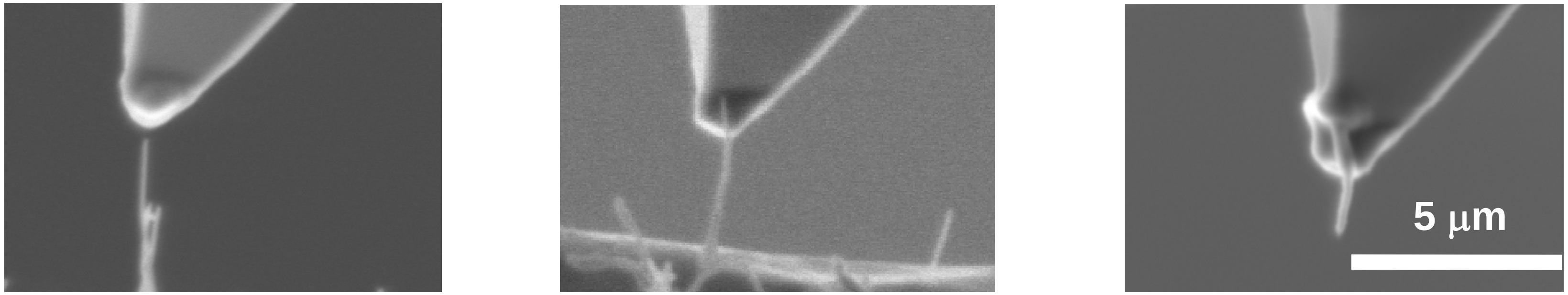
❺ dissolution of the Al_2O_3 membrane
5 wt% NaOH solution, 50°C, 30 minutes

⇨freestanding nanowires

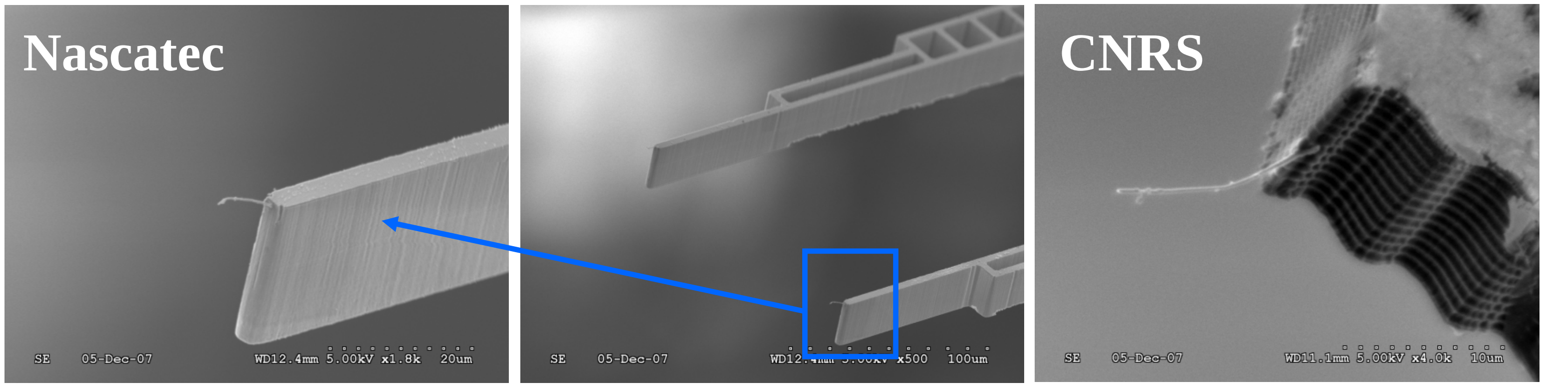


Structuring of grippers with metallic NWs attachment

⇨ **approach** ⇨ **fixation** ⇨ **withdrawal**



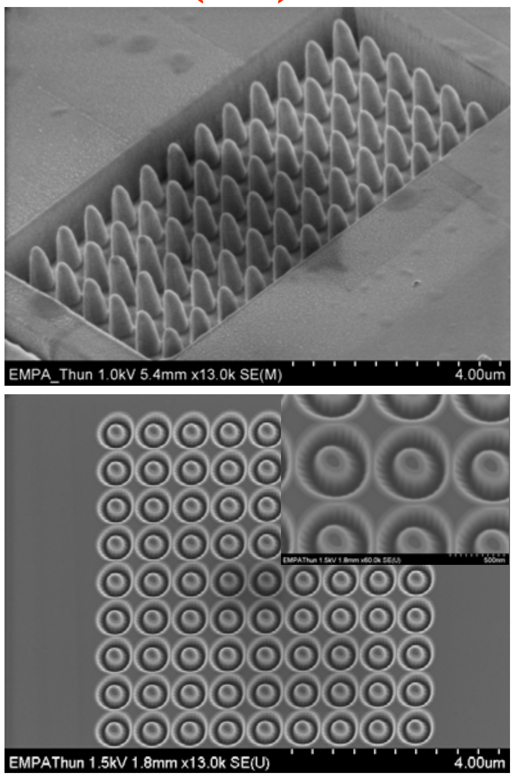
- **Nanosoldering by FEBID**
  - **Soldering micro/nanostructures with each other**
    - **Ag NWs on a piezoelectric slip-stick robot arm**
    - **CNRS or Nascatech microgrippers on a x,y,z piezo stage**
    - **Bond using the electron beam and gases present in the SEM chamber**



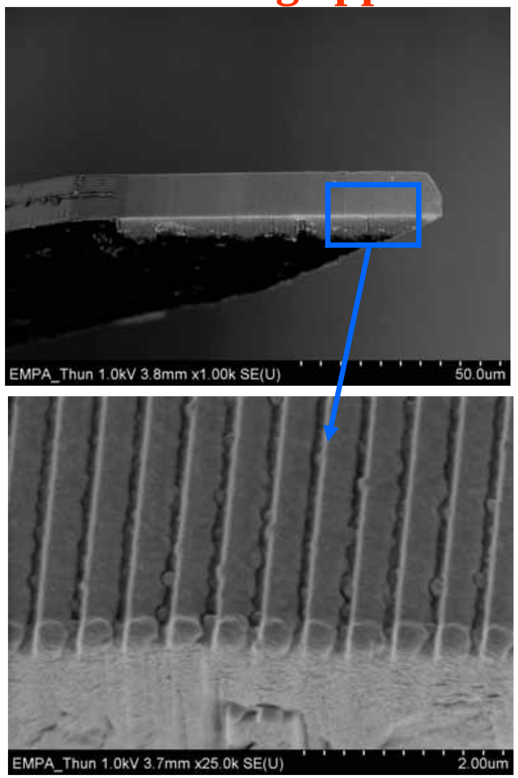
Structuring of Grippers by FIB

- **Focused ion beam (FIB)**
  - **Mainly based on liquid Gallium sources**
  - **Ability to remove or add locally material (metals or insulators) at sub-100 nm dimensions**

Arrays of pillars onto Si(100) wafer

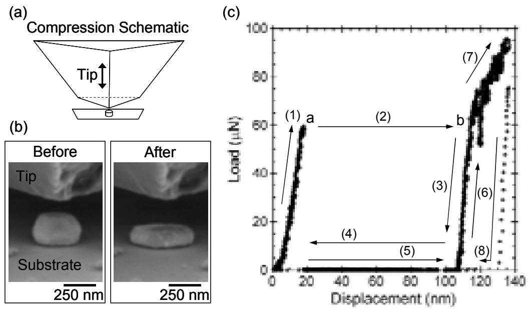


Gold steps onto CNRS grippers



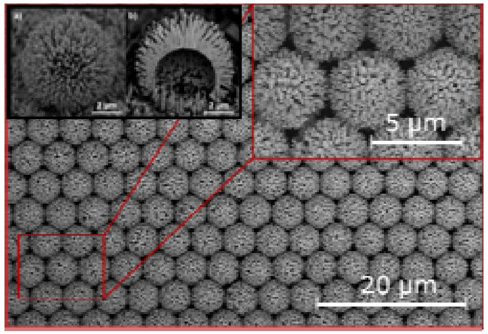
Applications de ces Nanostructures

Propriétés mécaniques



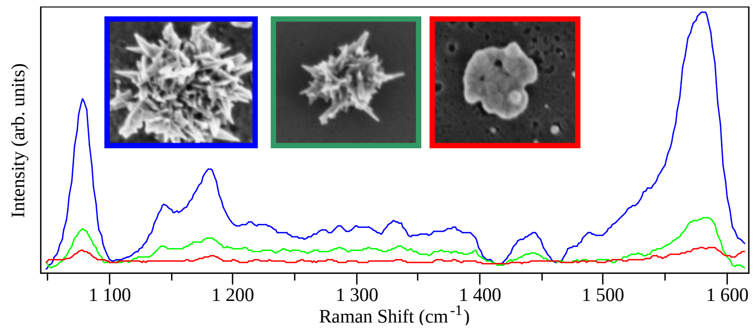
Nanotech. 21, 2010, 055701

Photovoltaic applications

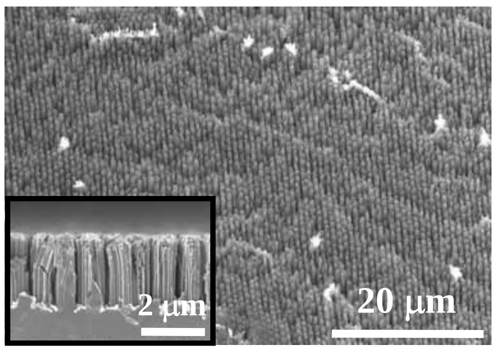


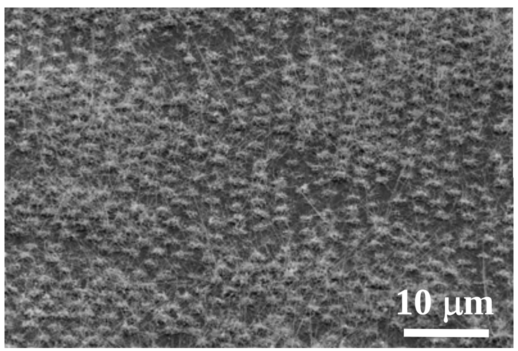
Adv. Marter. 22, 2010, 1

SERS applications



Silicon NWs by etching and growth





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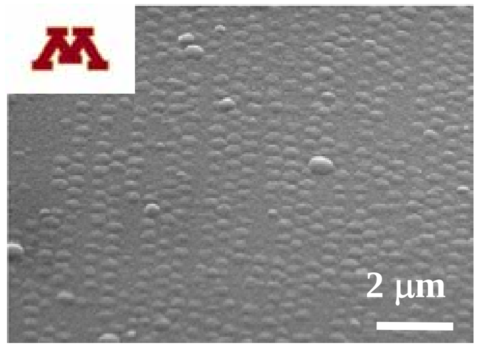
Composites Si/SiC



Conclusion and Perspectives

- **Natural lithography: Three methods for fabrication of metallic nanodots on Si substrates**
  - ↳ **Different size, shape, density and crystallinity**
  - ↳ **Different materials (Au, Ag, Si, ZnO…)**
    - ➲ **Study of pull-off forces in Progress**

- **Gripper structuring by FEBID**

- **Gripper structuring with metallic NWs**

- **Gripper structuring by FIB**

  ➲ **Study these grippers for nanomanipulation inside the SEM and/or for microgripping**

Après…..

Un poste CR 15/02
Laboratoire d'affectation : Institut Européen des Membranes
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Membranes inorganiques non oxydes multifonctionnelles pour des applications «énergie»

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