Nanofabrication on Non-flat Irregular Surfaces

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（这个物理所讲习班每年暑期都开，一周时间，免费包教材不包住宿。有学分）

Canada's Largest Engineering School

- **6,554** Undergraduate Students
- **700** International Students
- **1,829** Graduate Students
- **286** Faculty
- **202** Staff

- North America's first (now the world's largest) co-operative education program
- Canada's most innovative university for 21 consecutive years (*Maclean's*)

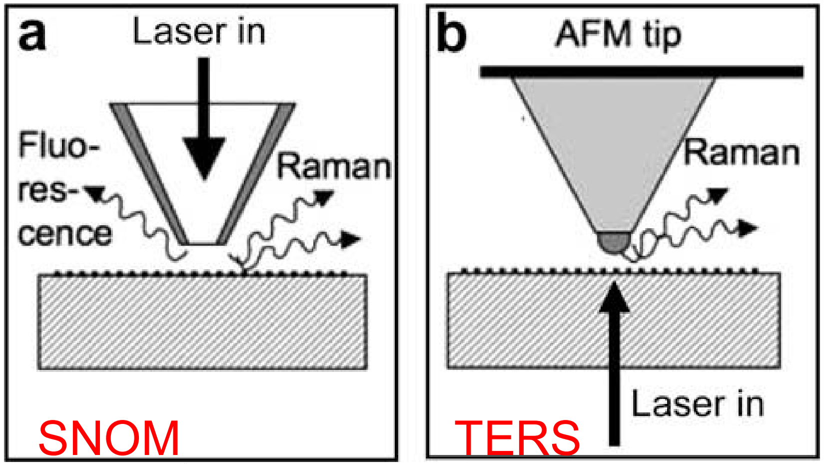
WATERLOO
ENGINEERING
ELECTRICAL AND COMPUTER ENGINEERING

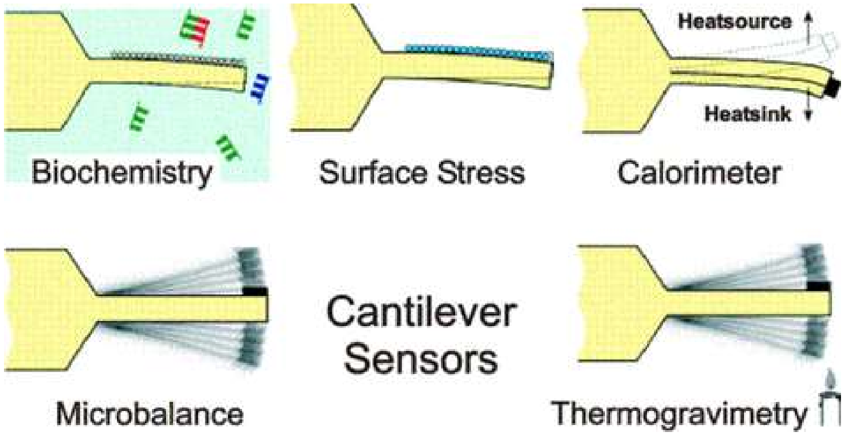
Outline

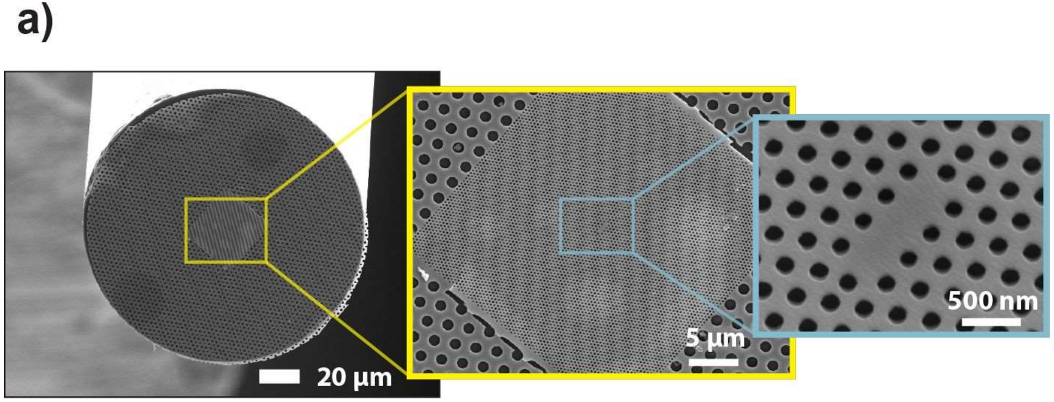
- Motivation and introduction.
- E-beam lithography using evaporated resist.
- E-beam lithography using ice resist.
- E-beam lithography using mono-layer polymer brush resist.
- E-beam lithography using self-assembled mono-layer (SAM) resist.

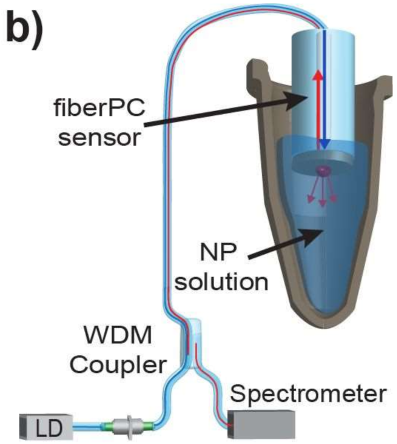
Motivation: why patterning on non-flat surface

- Nanofabrication on non-planar surfaces may find applications in: (AFM) tip-enhanced Raman spectroscopy (TERS) for chemical analysis, mass-(bio)sensor using a cantilever, and lab-on-fiber technology.



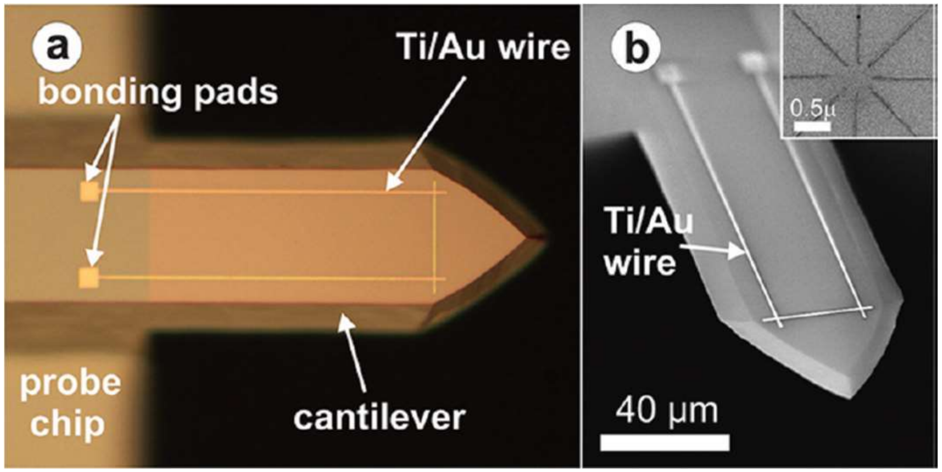


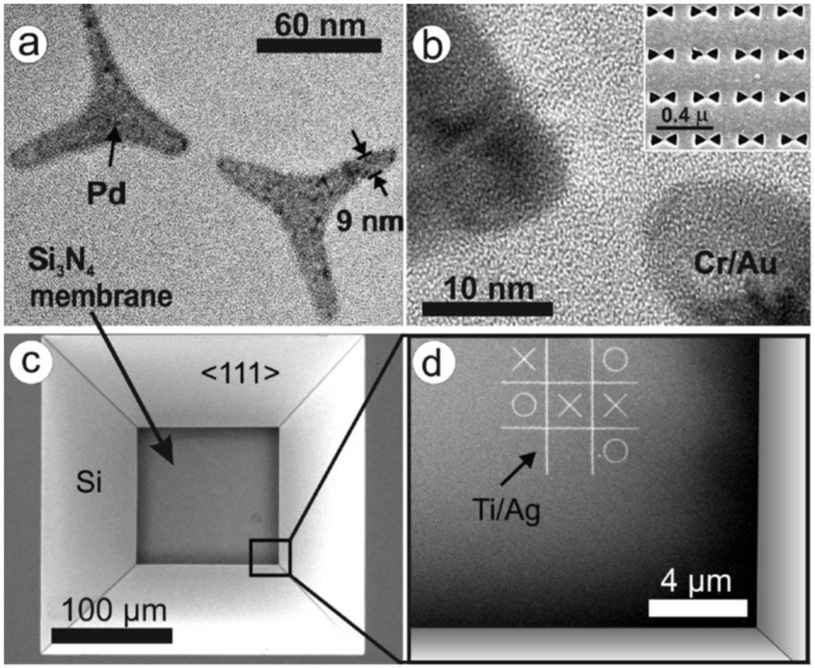




Yeo B S, Stadler J, Schmid T, Zenobi R and Zhang W H, Chem. Phys. Lett., 472, 1–13 (2009).
Consales M, Ricciardi A, Crescitelli A, Esposito E, Cutolo A and Cusano A, ACS Nano, 6, 3163–70 (2012).

Motivation: why patterning on non-flat surface



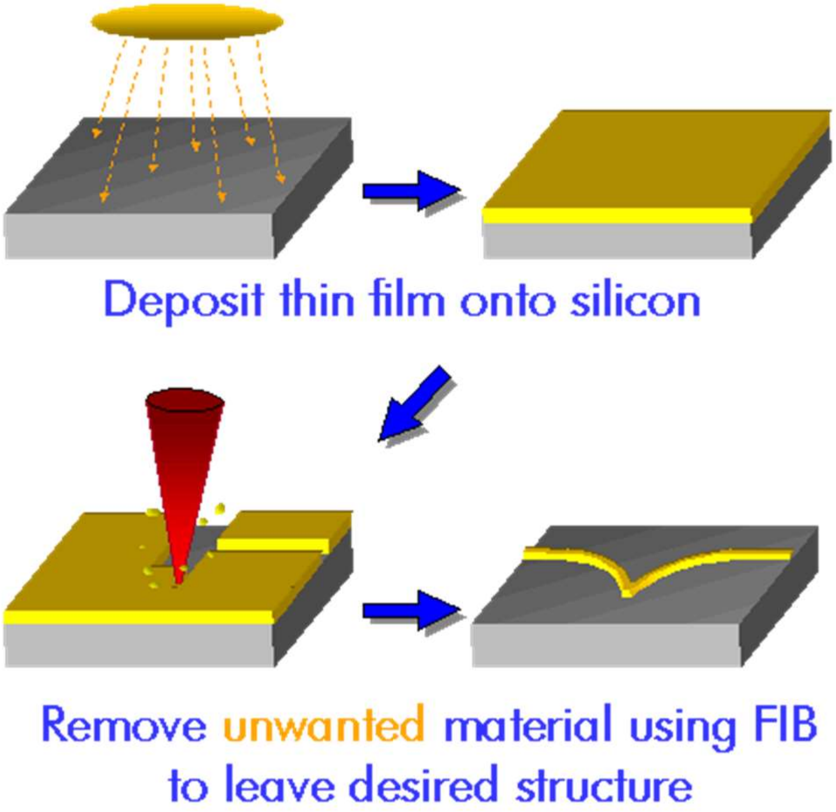


- Cantilevers with added metal structures have been used to study fundamental quantum mechanical systems, such as Bose−Einstein condensates and mesoscopic persistent currents.
- Silicon nitride membranes find important applications as platforms for plasmonic nanostructures and nanopores, but are too fragile to withstand ultrasonication, which is usually used with polymeric resists to assist lift-off.

A. Han, et al., Nano Lett., 12, 1018 (2012).

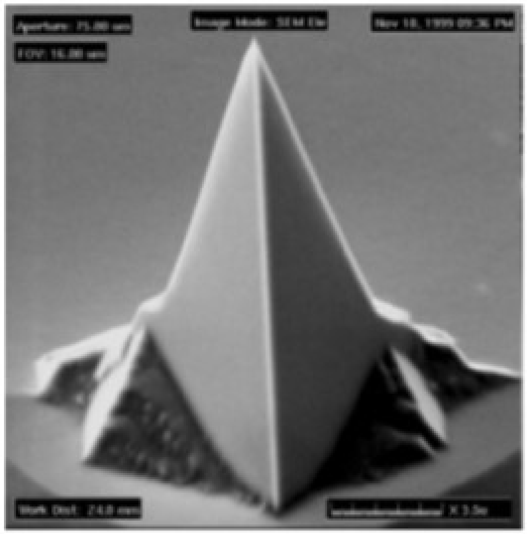
The most popular method: focused ion beam (FIB)

- FIB milling on any surface.

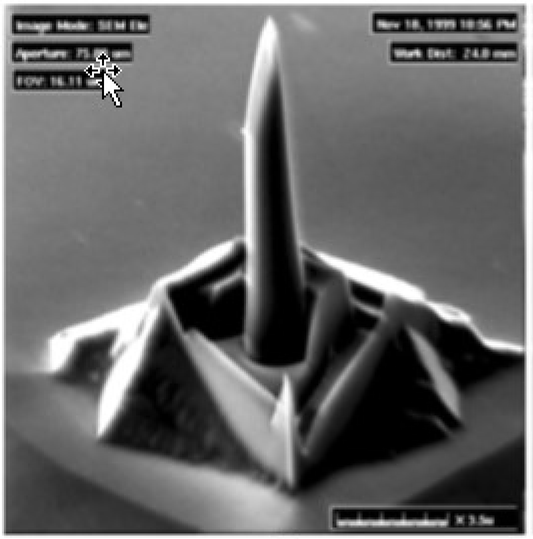


Deposit thin film onto silicon

Remove unwanted material using FIB to leave desired structure



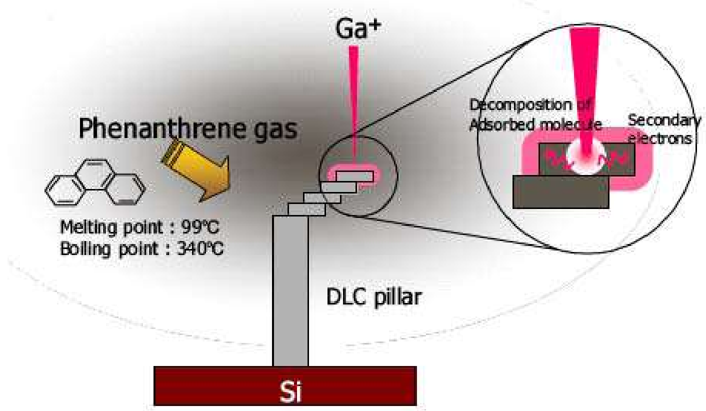
(a)

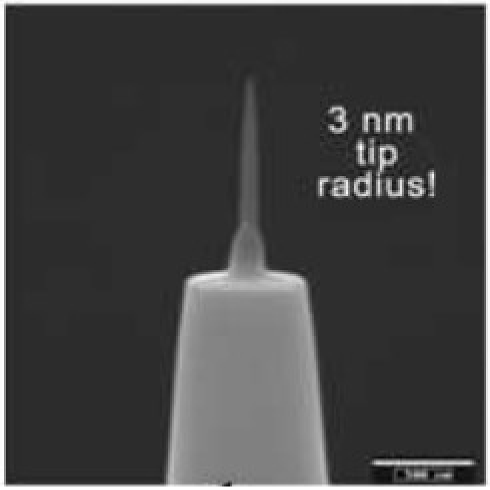


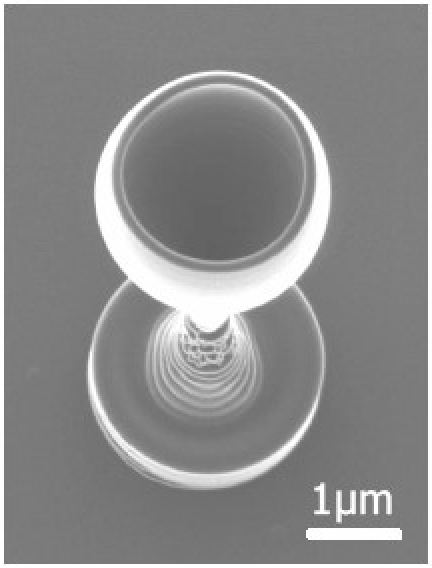
(b)

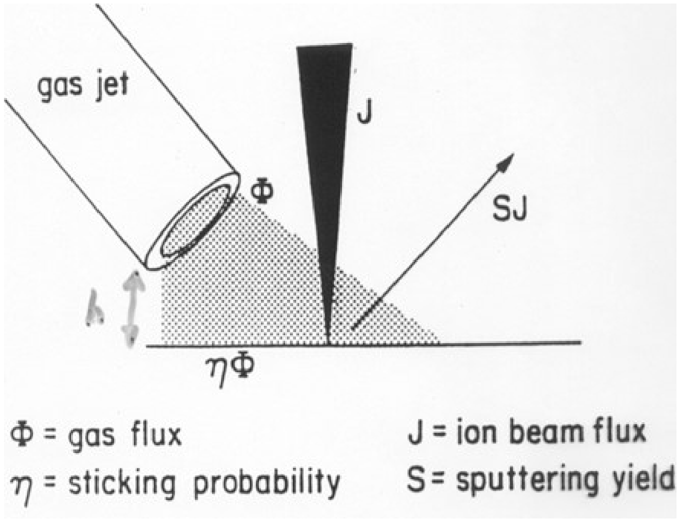
The most popular method: focused ion beam (FIB)

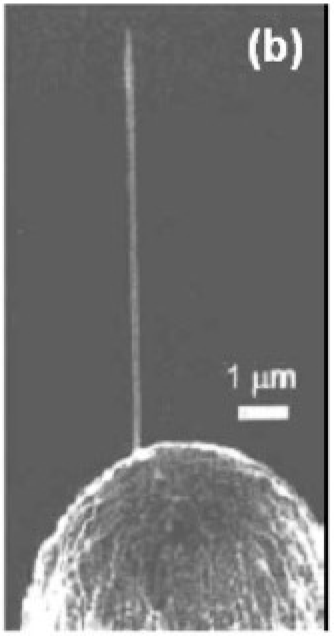
- Focused ion (or electron) beam induced deposition, on any surface.











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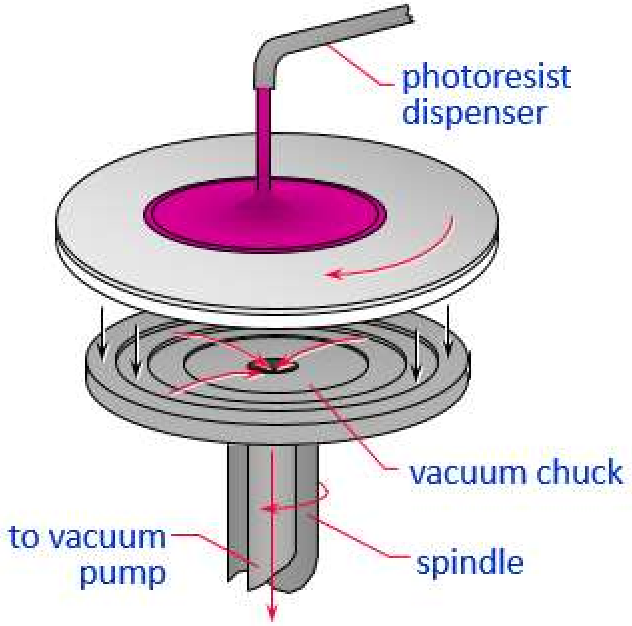
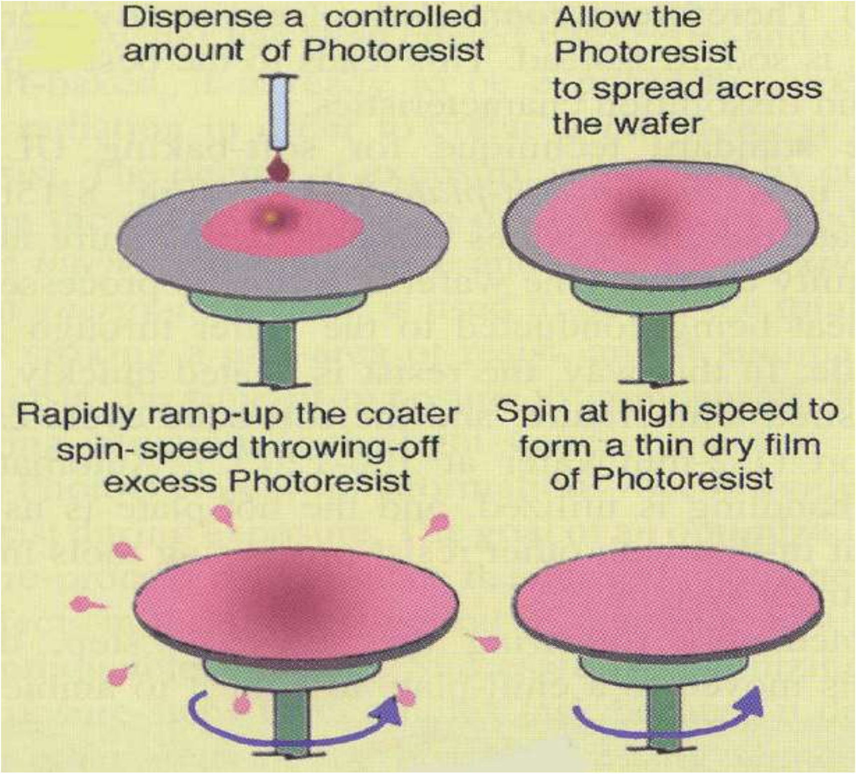
Electron beam lithography (EBL) for patterning irregular surfaces

- FIB is very versatile, but has its own limits: very slow, very high cost, Ga contamination, substrate damage, works well for milling trenches/holes but not protruded structures…..
- EBL doesn't have those issues, yet need an e-beam resist.
- Unfortunately, the most popular resist coating method, spin-coating, can attain uniform thin resist only on flat wafers.



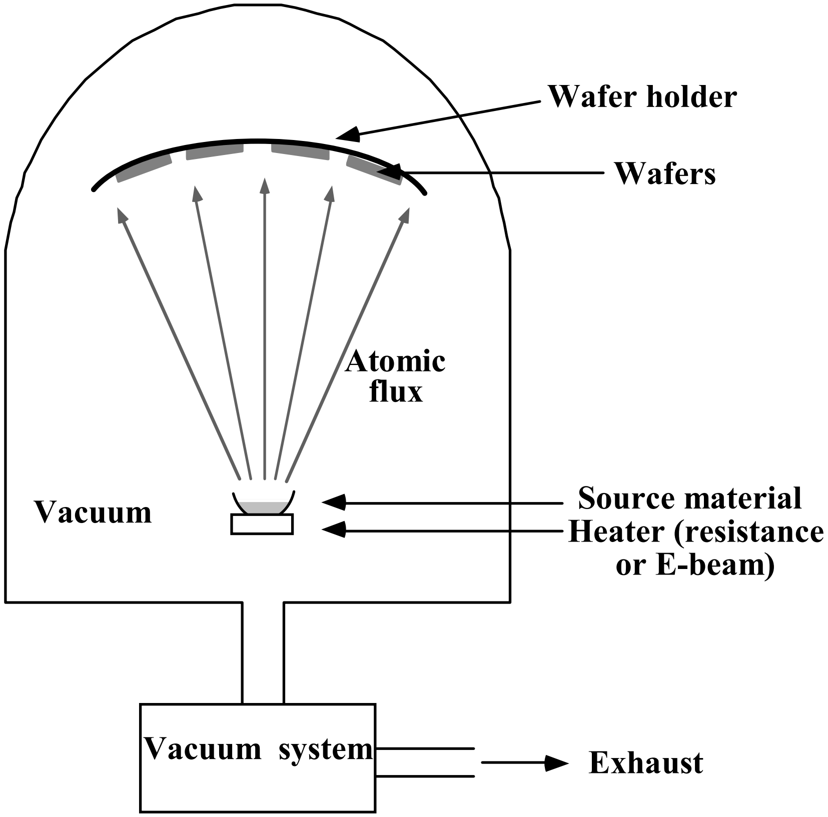


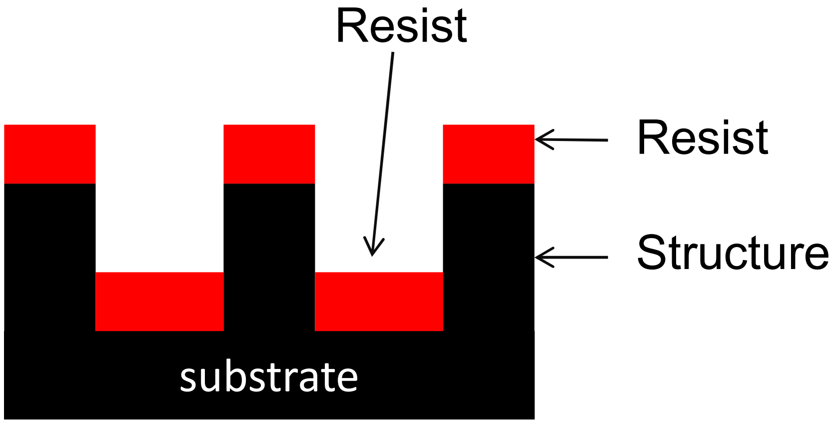
Outline

- Motivation and introduction.
- E-beam lithography using evaporated resist
  - ➢ Metal halide (AlF_3, NaCl, …)
  - ➢ Non-polymeric sterol
  - ➢ Polymer (polystyrene)
- E-beam lithography using ice resist.
- E-beam lithography using mono-layer polymer brush resist.
- E-beam lithography using self-assembled mono-layer (SAM) resist.

Electron beam lithography (EBL) using evaporated resist

- In evaporation, source material is heated in high vacuum chamber ($P < 10^{-5}$ Torr), hence the name vacuum deposition.
- Heating is done by resistive or e-beam sources.
- High vacuum is required to minimize collisions of source atoms with background species.
- It is a line-of-sight deposition, can coat any surface.

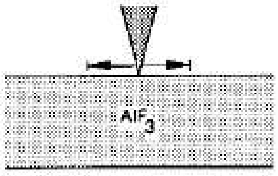




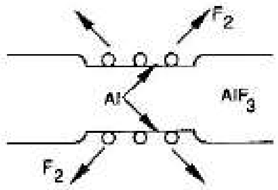
High-Res. Positive Inorganic Resists

(Coated by evaporation)

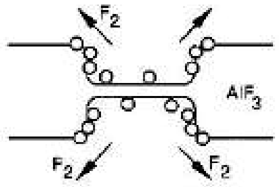
Resist	Minimum linewidth	Typical aspect ratio	Deposition	Dose at 100 keV to expose 500-nm-thick layer (C/cm^2)	Mechanism of exposure
PMMA	8–10 nm	45	Spinning	5×10^{-4}	Bond breaking
NaCl	1.5 nm	>40	Sublimation 40-Å grain	10^2–10^3	Dissociation of Cl_2 Diffusion of Na
LiF	1.5 nm	>40	Sublimation 50-Å grain	10^{-1}–10^{-2}	Dissociation of F_2 Diffusion of Li
MgF_2	1.5 nm	>40	Sublimation 50-Å grain	1–10^{-1}	Dissociation of F_2
AlF_2	1.5 nm	>40	Amorphous	1–10	Dissociation of F_2 Diffusion of Al
KCl	1.5 nm	>40	Deposition 50-Å grain	1–10	Dissociation of Cl_2 Diffusion of K
Metal–alumina	1.5 nm	>40	Cut thin-film slabs	1×10^7 (2000 Å thick)	



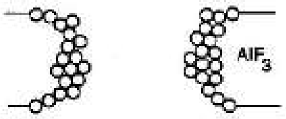
(a) BEGINNING OF EXPOSURE



(b) AFTER SOME IRRADIATION



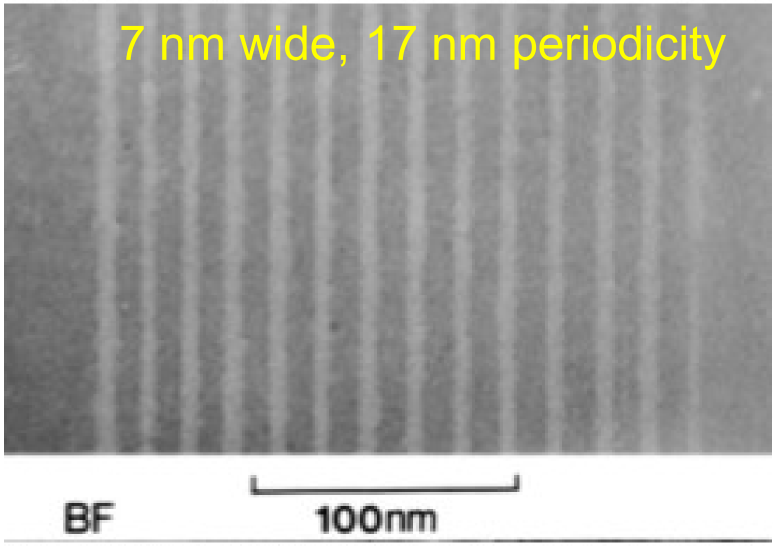
(c) MORE IRRADIATION



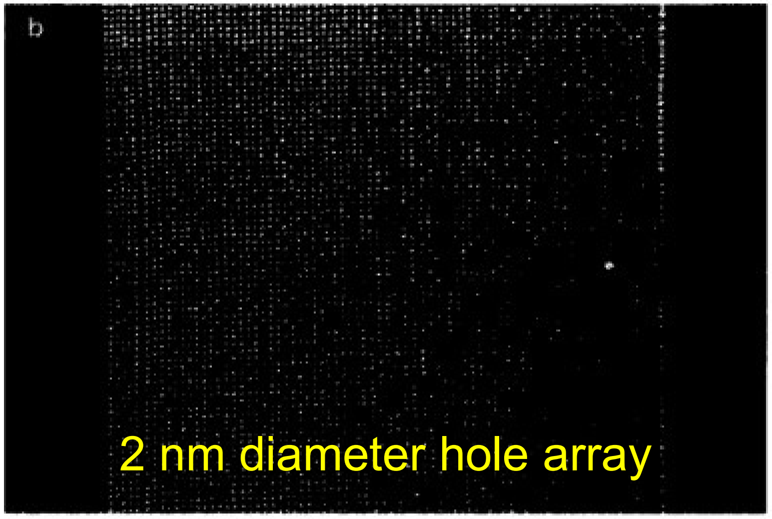
Self-development of metal halides by e-beam

Ultra-high resolution of evaporated AlF_3 resist

- Extremely high resolution, since only primary (not secondary) high energy beam is responsible for exposure.
- However, extremely low sensitivity, need long writing time.
- It is good for narrow trench/hole exposure, not good for area exposure.



7 nm wide, 17 nm periodicity



2 nm diameter hole array

FIG. 4. Array of holes etched into 80 nm thick AlF_3 with 100 keV electrons. (a) 4 nm average diameter holes on 8 nm centers and (b) 2 nm average diameter holes on 4 nm centers (from Ref. 14).

Microelectronic Engineering, 9, 557 (1989).
J. Vac. Sci. Technol. B, 3, 367 (1985).

Evaporated dry QSR-5 resist

- QSR-5 is a negative resist consisting of non-polymeric sterol molecules, commercialized by Quantiscript Inc.
- It is a “dry” resist coated by thermal evaporation.
- Develop by MEK (methyl ethyl ketone), sensitivity 5630 μC/cm^2 at 20 keV exposure.

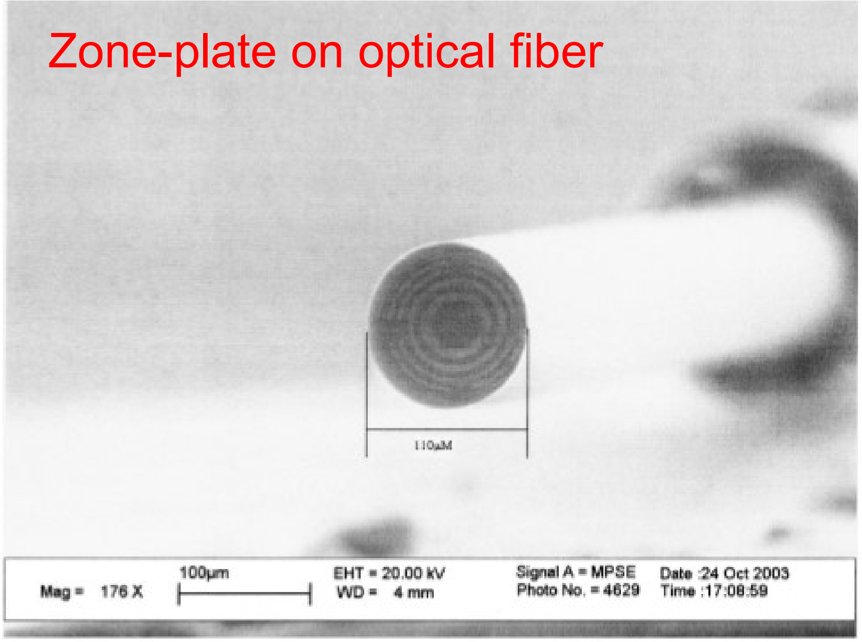

Zone-plate on optical fiber

FIG. 1. Top view of the fabricated zone plate on the tip of the fiber.

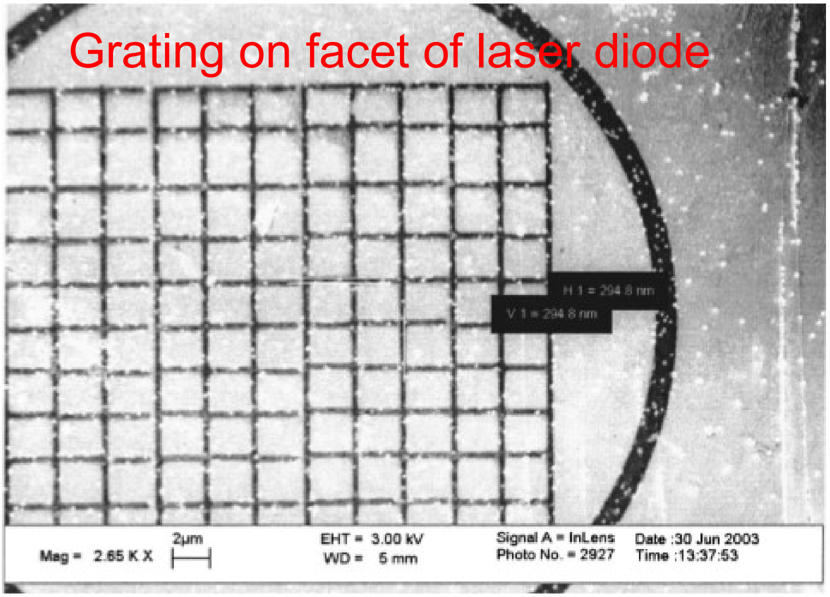

Grating on facet of laser diode

FIG. 4. Diffractive element fabricated on the facet of laser diode with linewidth of approx. 225 nm.

Microelectronic Engineering, 103, 123 (2013); J. Vac. Sci. Technol. A, 22, 743 (2004).

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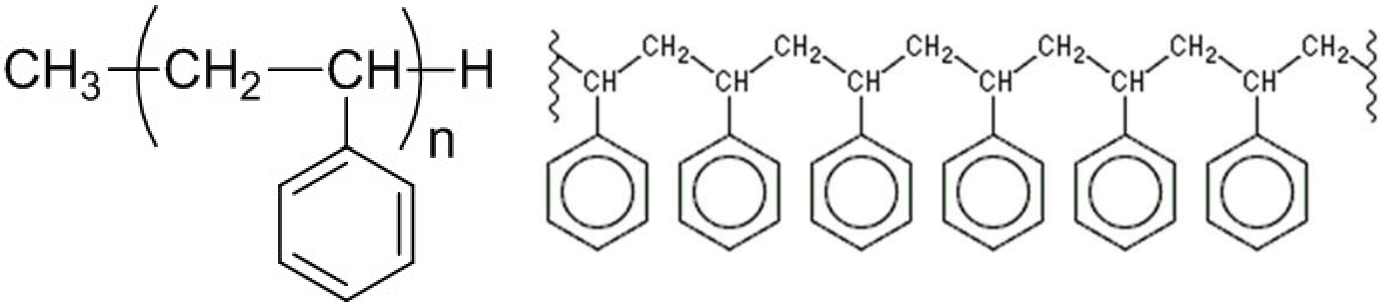
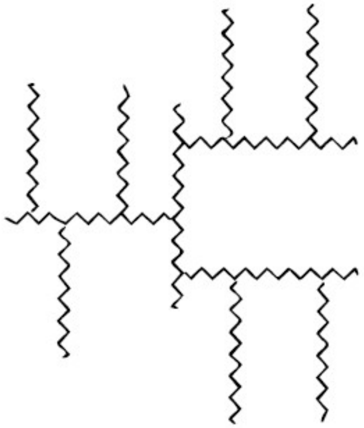
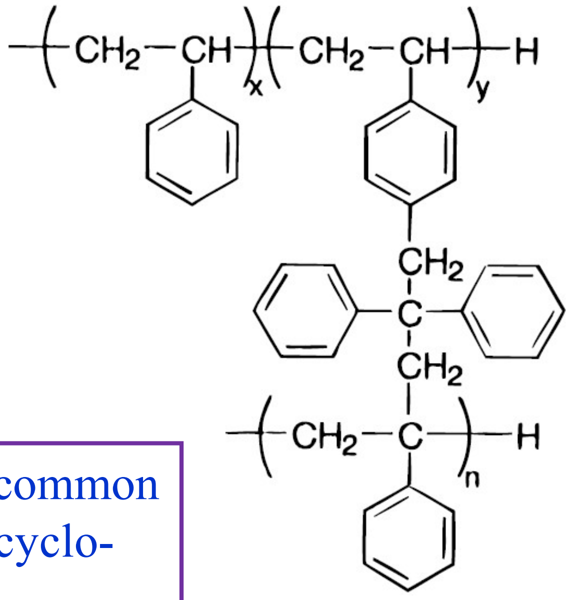
Evaporated polystyrene resist

Linear polystyrene



Cross-linked by e-beam exposure, schematic and chemical structure



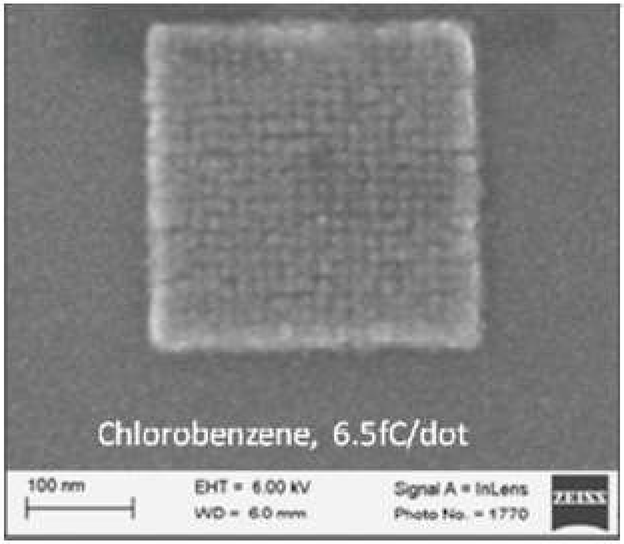


- Linear polystyrene (PS) can be dissolved by many common solvents: acetone, tetrahydrofuran, chlorobenzene, cyclohexane, xylene, anisole, and so on.
- Cross-linked PS is insoluble in those solvents.
- Thus ALL those solvents can be used as developer for PS.

Motivation: why polystyrene?

Polystyrene (PS) is a negative resist, and it offers:

- Very high resolution for low molecular weight PS.
- Very high sensitivity for high molecular weight PS.
- Low cost and unlimited shelf life.
- Easy development – any solvents that dissolves unexposed PS can be used.
- High dry etching resistance for etching mask, similar to ZEP.



PS dot array with 15 nm pitch

Moreover, it can be:

- Coated on any surface (flat or not) by thermal evaporation.

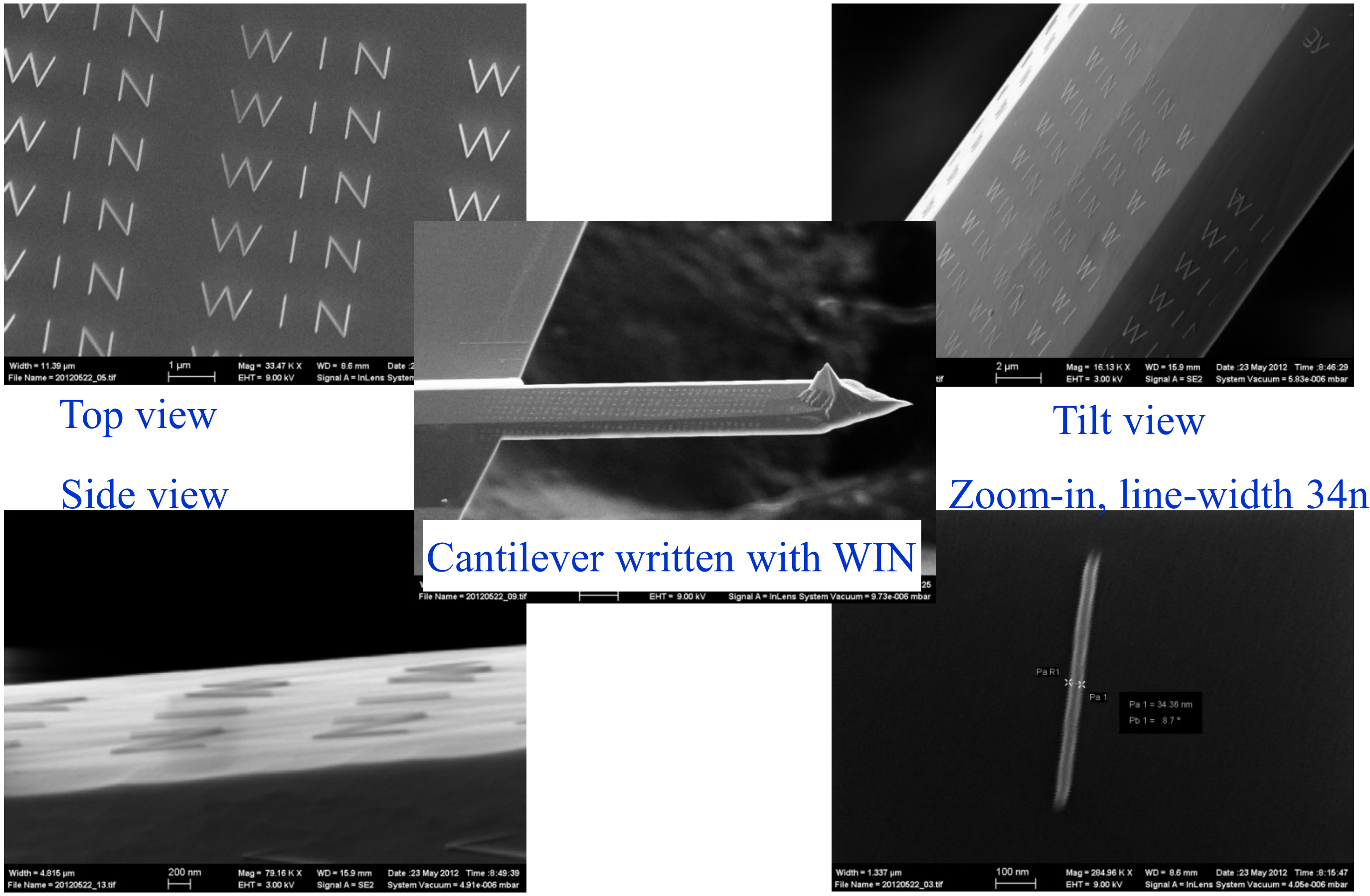
Ma and Cui, Nanoscale Research Letters, 6, 446 (2011). Con and Cui, Microelectronic Engineering, 98, 254-257(2012).
Dey and Cui, Nanotechnol., 24, 245302 (2013). Dey and Cui, J. Vac. Sci. Technol. B, 32, 06F507 (2014).

Polystyrene can be thermally evaporated

- Evaporated resist can be coated on any surface, such as on an AFM tip or optical fiber.
- However, most resists (e.g. PMMA) decomposes at temperature needed for evaporation.
- Nevertheless, we are able to thermally evaporate low molecular weight polystyrene.

Electron beam lithography on an AFM cantilever using evaporated polystyrene resist.

“WIN” = Waterloo Institute for Nanotechnology



Top view

Side view

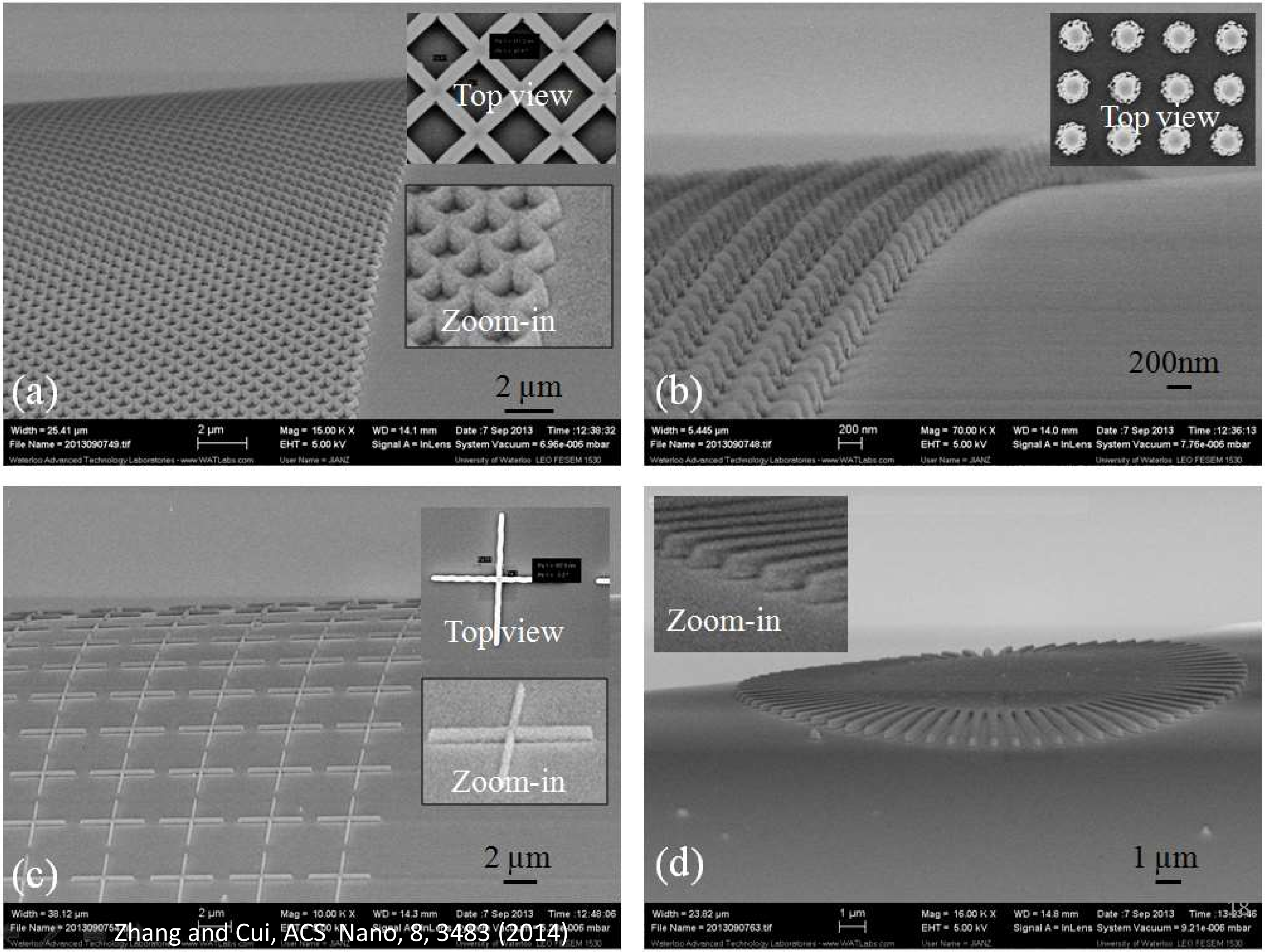
Cantilever written with WIN

Tilt view

Zoom-in, line-width 34nm

Zhang and Cui, ACS Nano, 8, 3483 (2014). Jian Zhang/张鉴 is currently professor at Hangzhou Dianzi University/杭电

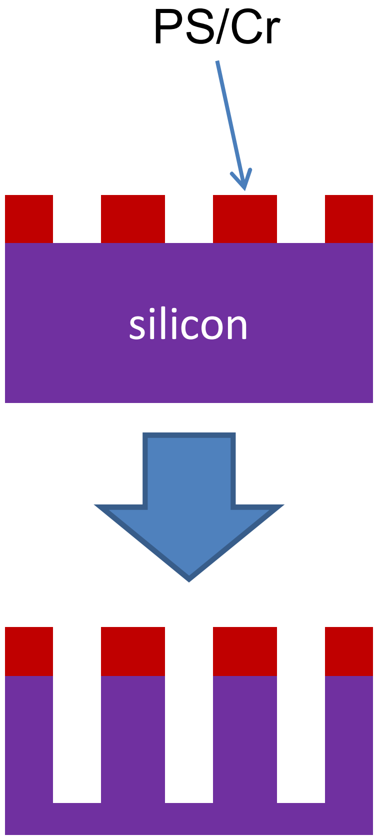
Nanofabrication on optical fiber using evaporated polystyrene resist

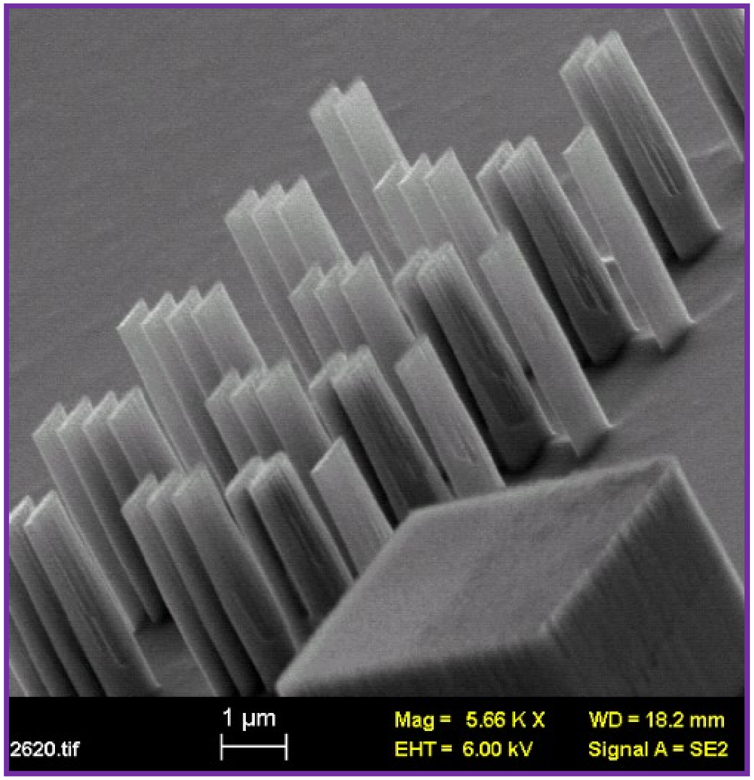


Zhang and Cui, ACS Nano, 8, 3483 (2014)

Nano-composite resist Cr/polystyrene by co-evaporation

- Polymer resist is not good mask for pattern transfer to the substrate, with typical etching selectivity to silicon on the order of 1:1.
- Metal is ideal etch mask, with selectivity on the order of 1:100 (that is, etches silicon 100× faster than metal).
- Nano-composite resist Cr/PS can be patterned directly by electron beam lithography, with very high resistance to etching as it contains metal.
- This can be realized for polystyrene since it can be evaporated, like most metals (Cr, Al…).





Silicon structures with 100nm width and 3µm height. (if using pure polystyrene resist, can etch at most 400nm height)

ICP-RIE (reactive ion etching) using SF_6 and C_4H_8 gas. Resist consists of 7% Cr.

Con and Cui, Nanotechnology, 25, 175301 (2014)

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  - ➢ Water (H_2O) ice
  - ➢ Organic ice
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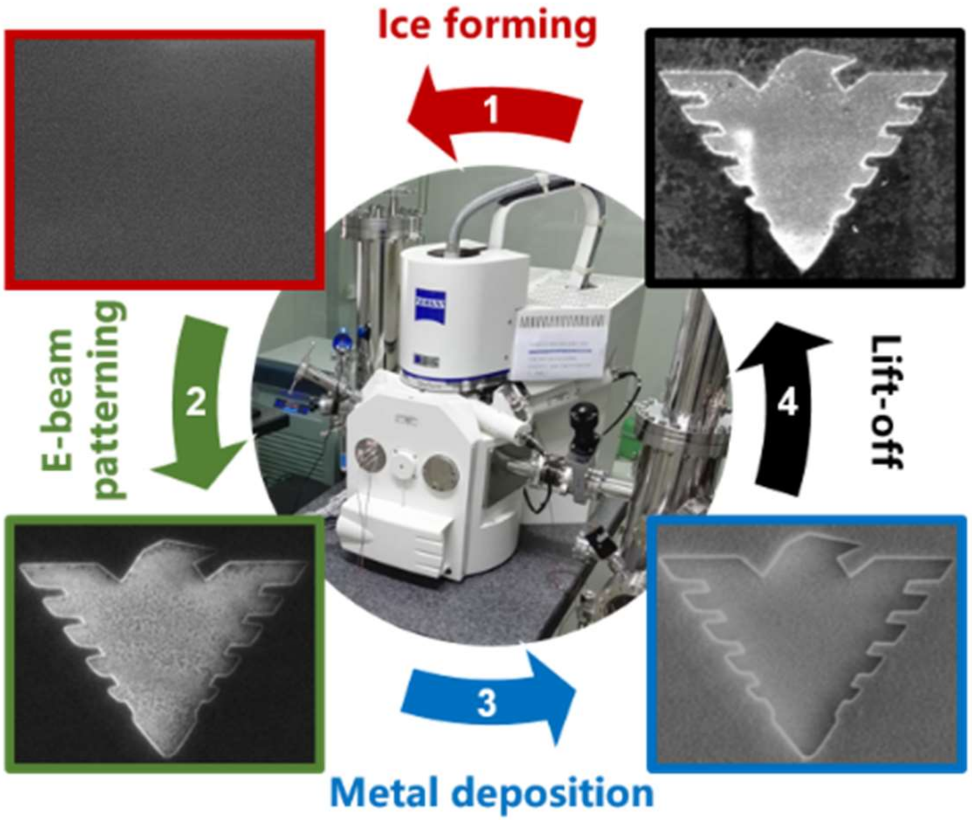
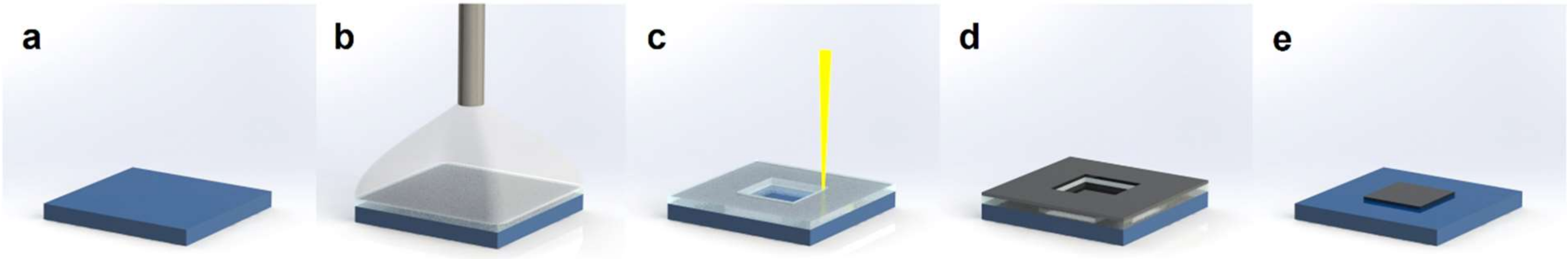
Electron-beam lithography using ice (iEBL)



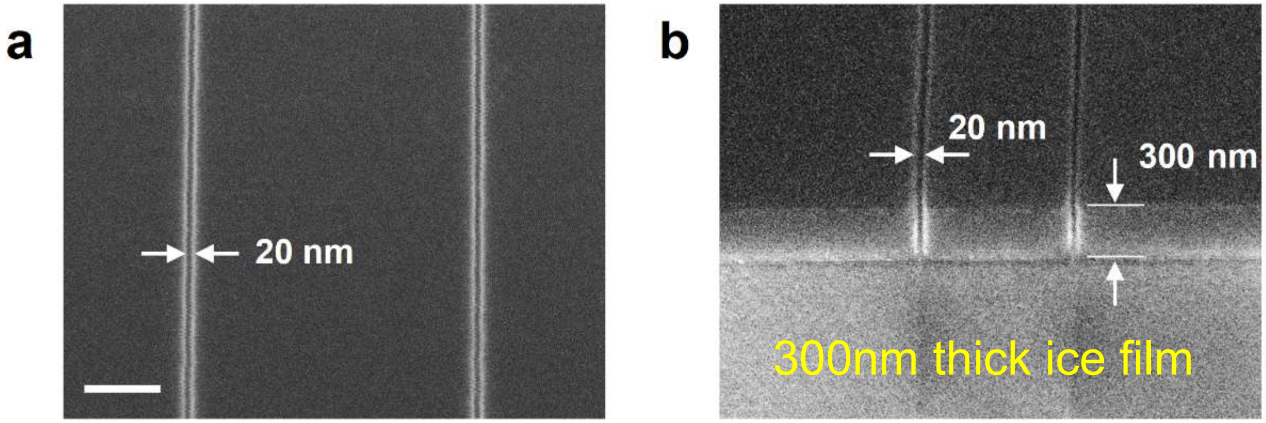
- Water ice is vaporized by e-beam exposure, thus it is positive resist.
- No need of development – it is self-developing.
- Metal evaporation needs to be done at low temperature (ice not melted).
- Lift-off can be done by soaking into water.



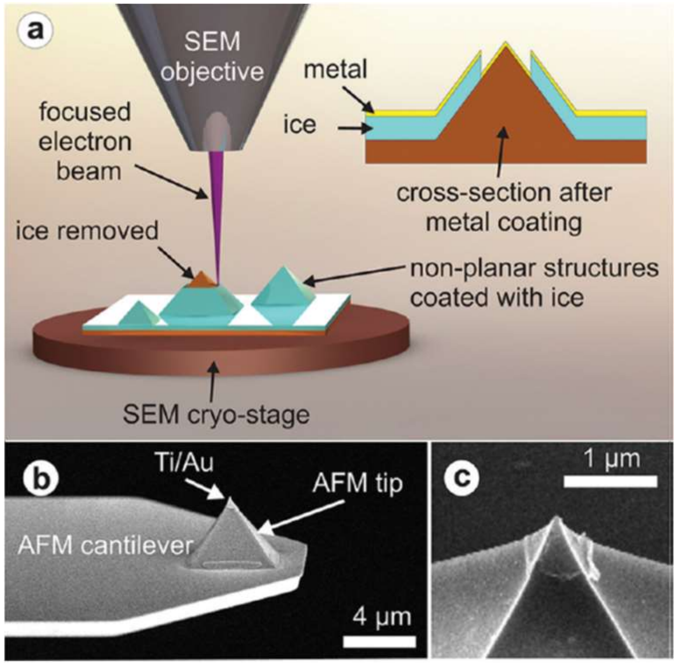
Refrigeration Condensation Exposure Metallization Lift-off

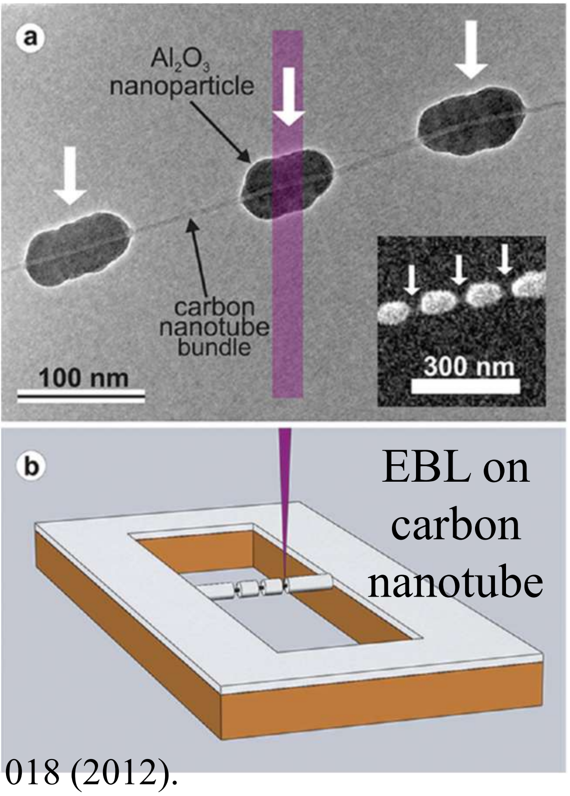
Hong, Zhao and Qiu, Nano Letts, 2018

Ice lithography on irregular surface



300nm thick ice film



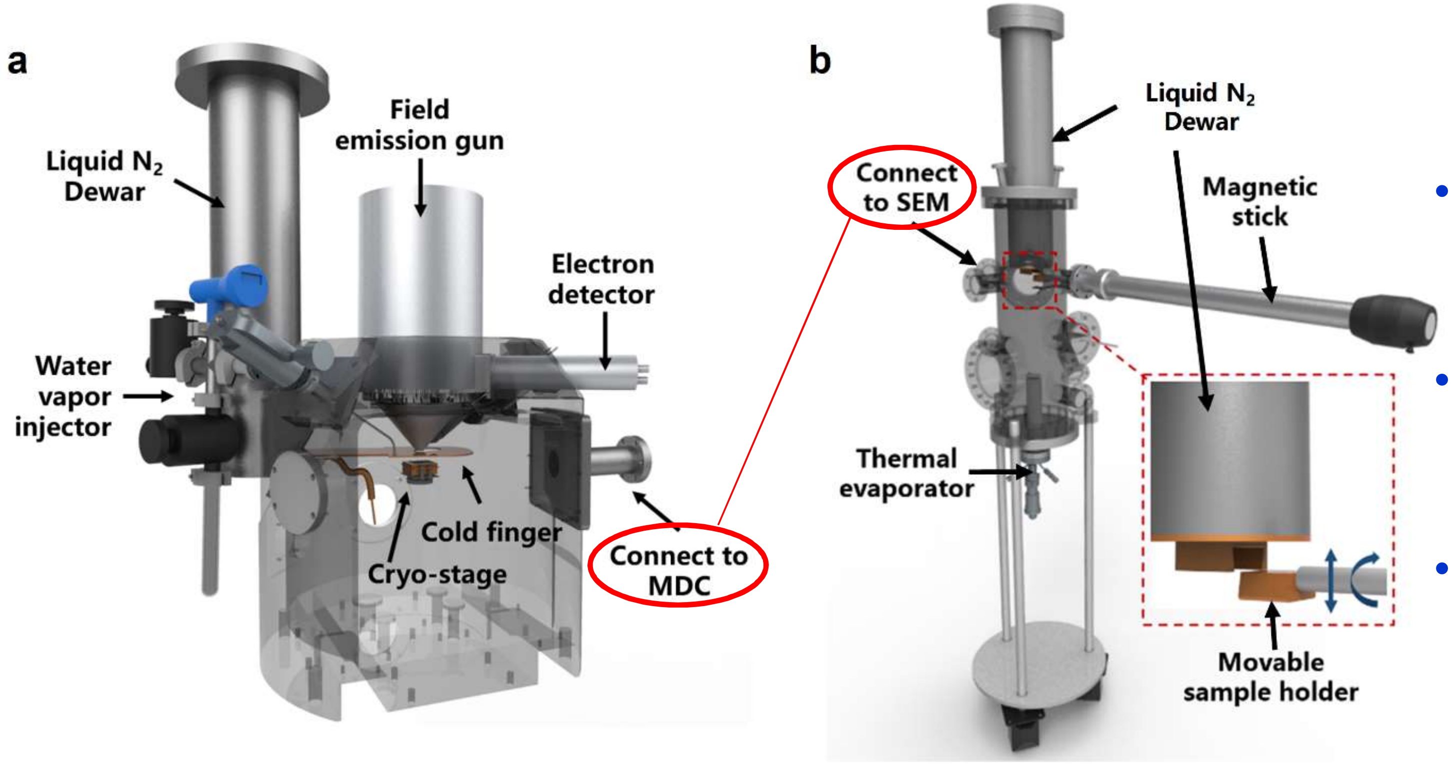


EBL on carbon nanotube

Hong, Zhao and Qiu, Nano Letts, 2018.

Han, Nano Lett. 12, 1018 (2012).

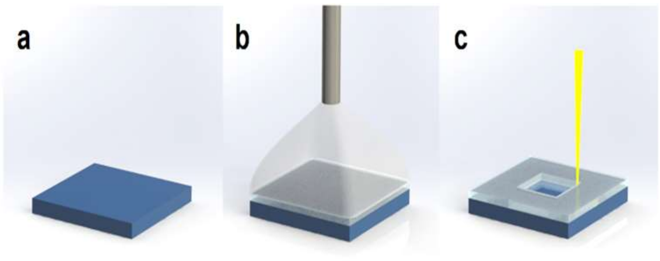
iEBL instrument (maintain low temperature)

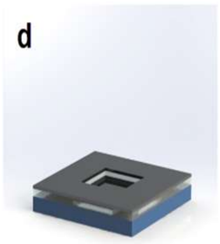


- EBL chamber and MDC chamber is connected
- They are maintained at high vacuum.
- Sample stage cooled by liquid N_2.

Modified Zeiss Sigma SEM

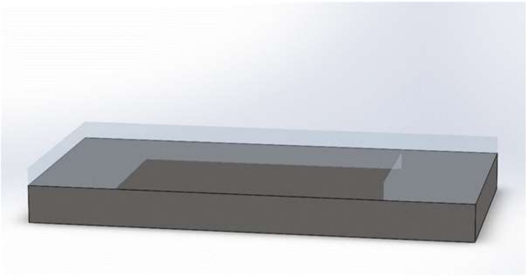
Metal deposition chamber (MDC)





Hong, Zhao and Qiu, Nano Letts, 2018

3D nanofabrication by stacking layered structures



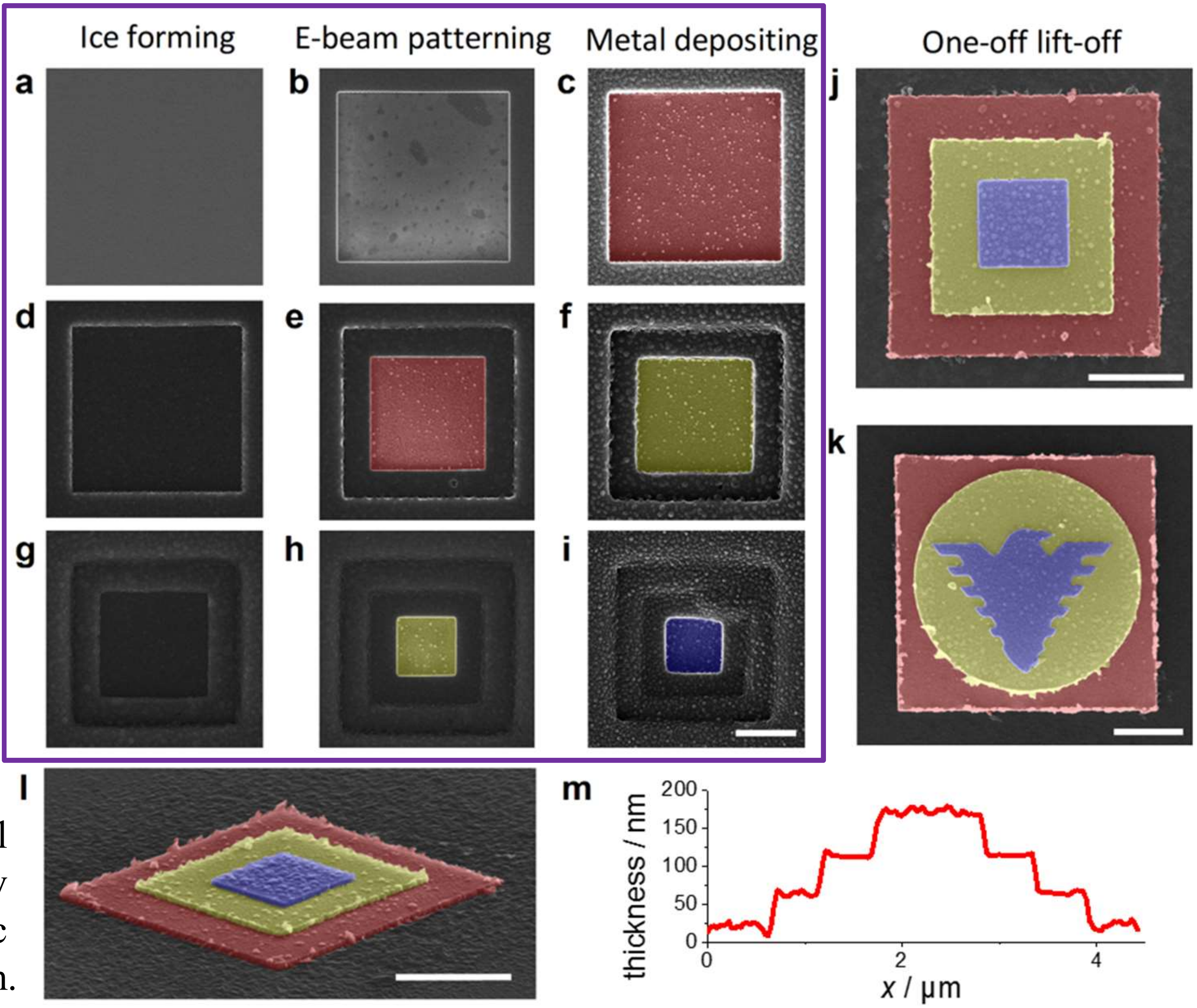


Figure 2. 3D pyramidal nanostructure fabricated by iEBL and *in situ* cryogenic SEM imaging. Scale bar, 1um.

Hong, Zhao and Qiu, Nano Letts, 2018

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    - Water (H_2O) ice
    - Organic ice
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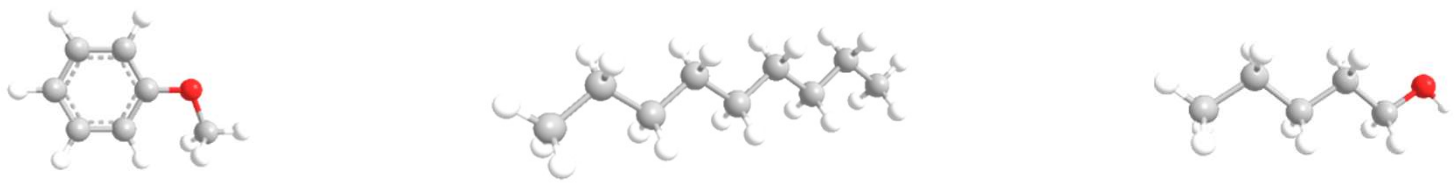
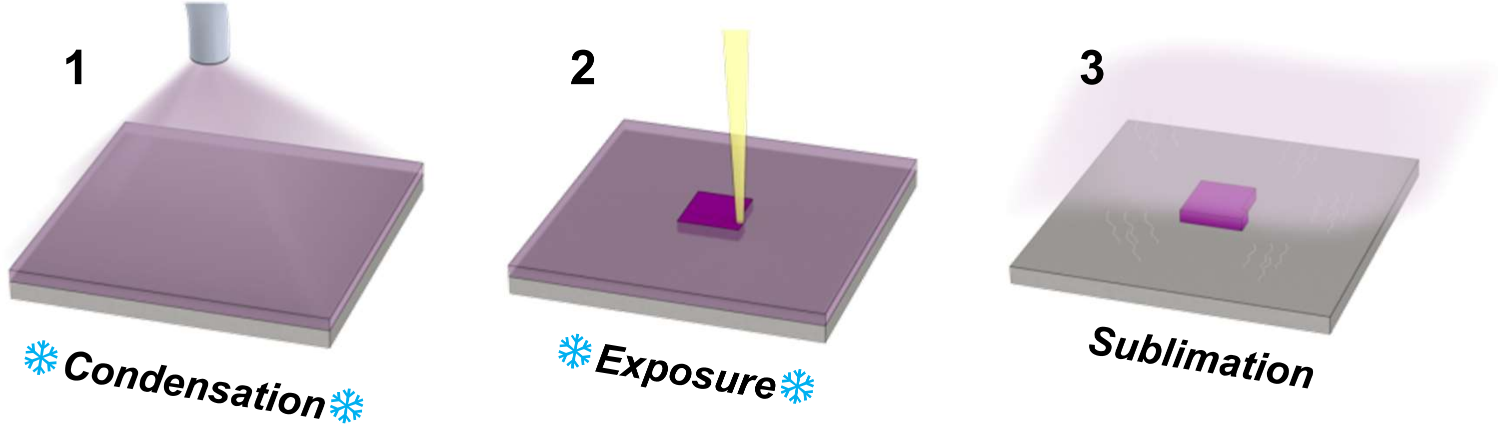
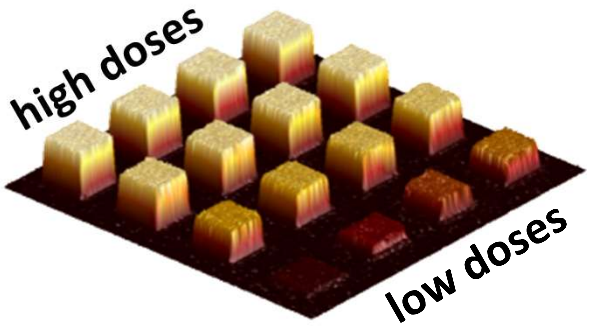
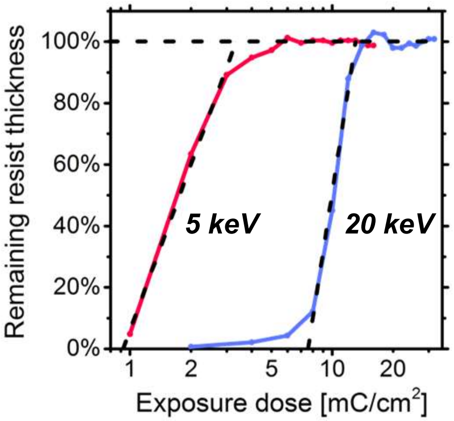
Organic ice lithography with negative tone

anisole C_7H_8O **nonane** C_9H_{20} **1-pentanol** $C_5H_{12}O$





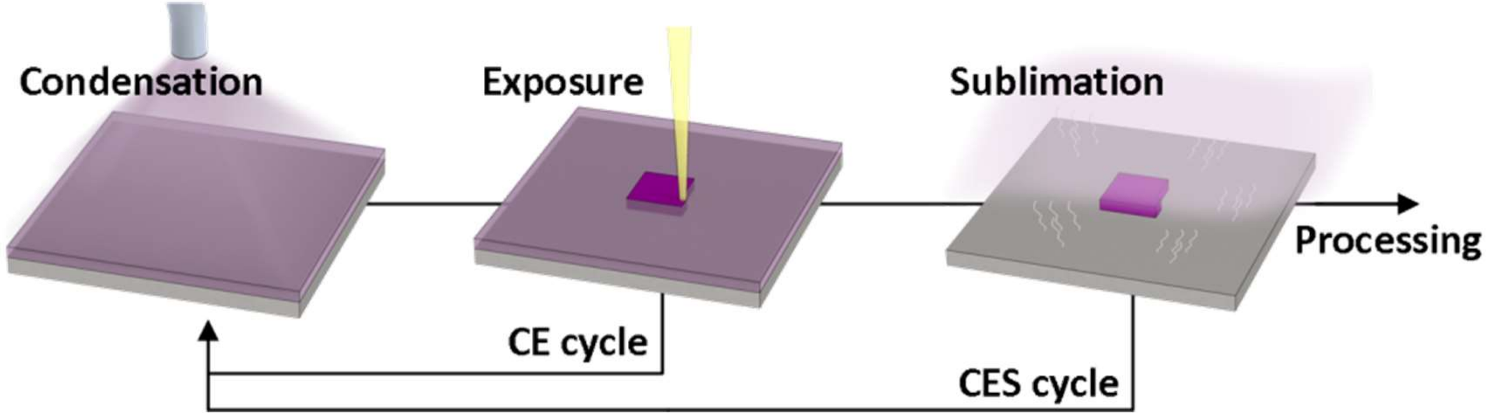




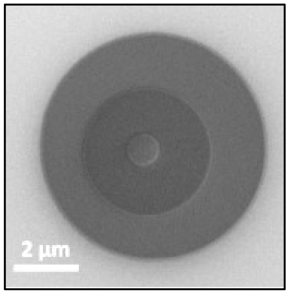
- **Nonane (C_9H_{20})**
- **Deposition:** -145 °C
- **Critical dose:** 3-12 mC/cm^2
- **Contrast:** 1.8-4.3

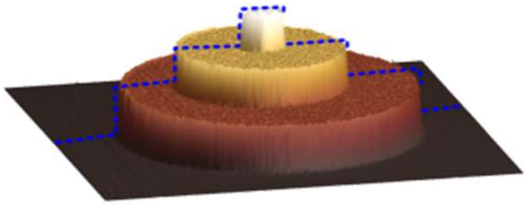
Tiddi and Pan, Nano Letts, 17 (12), 7886 (2017)

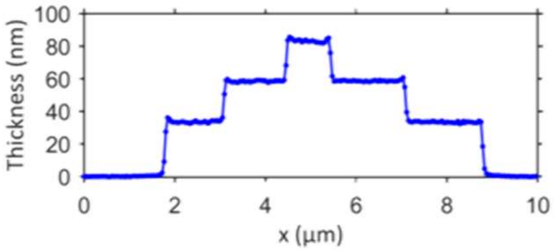
3D multilayer lithography



After three iterations:



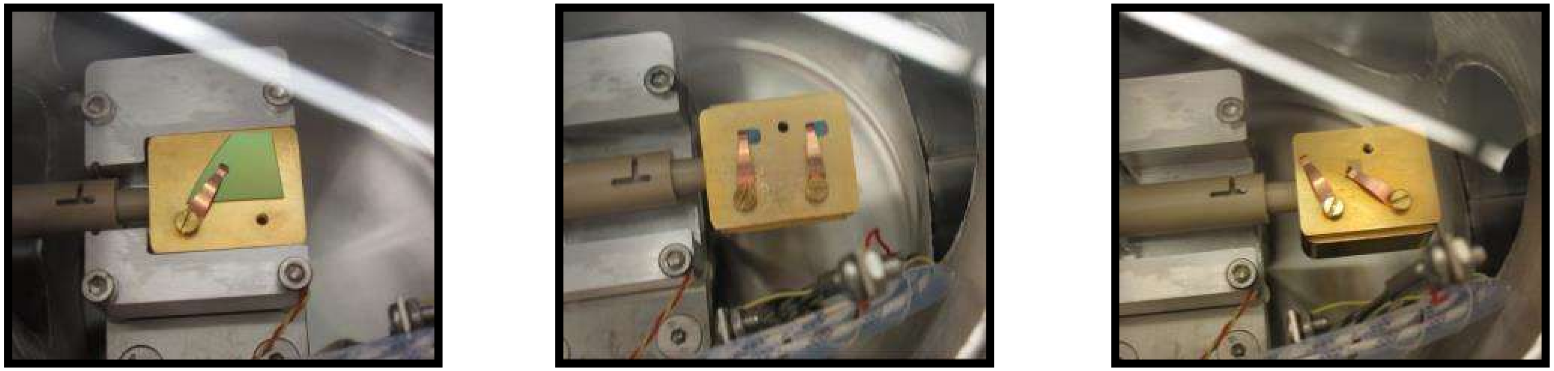


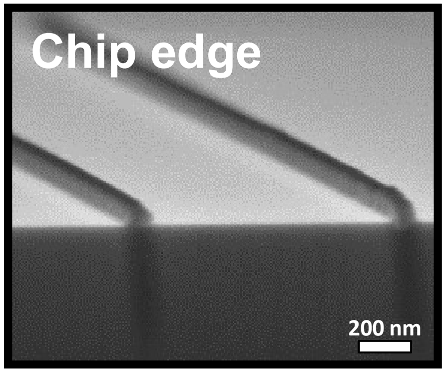


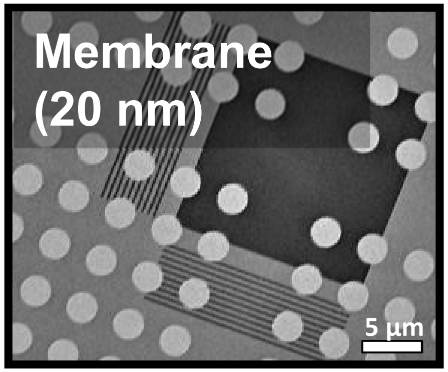
- Water H_2O cannot be cross-linked, can only be vaporized, thus positive tone.
- Organic molecule can be polymerized/cross-linked by electron beam exposure, thus can show negative tone.

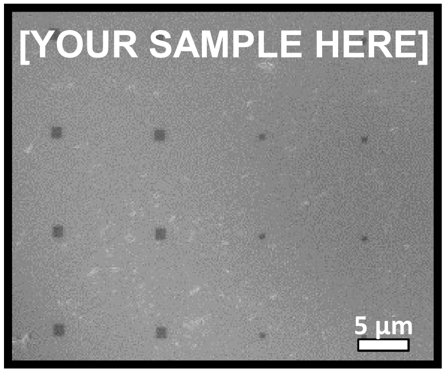
Tiddi and Pan, Nano Letts, 17 (12), 7886 (2017)

Ice lithography on irregular surfaces



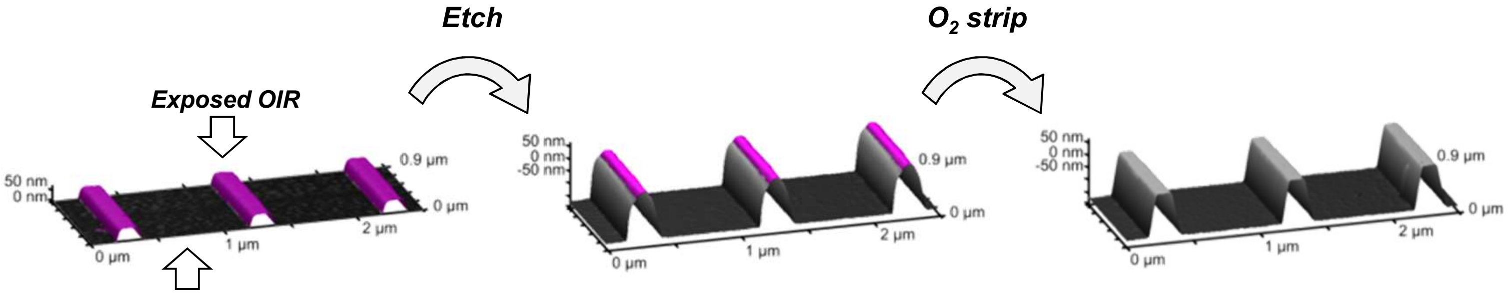






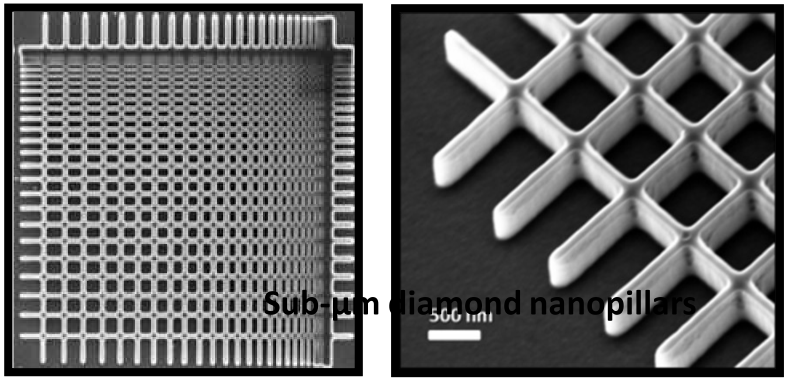
Pattern into Si, SiO_2 and diamond by RIE

Unlike water ice (positive tone), exposed organic ice resist (negative tone, cross-linked upon exposure) is stable at room temperature or etching temperature.

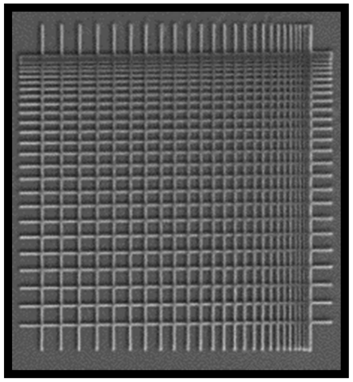


Substrate (Si)

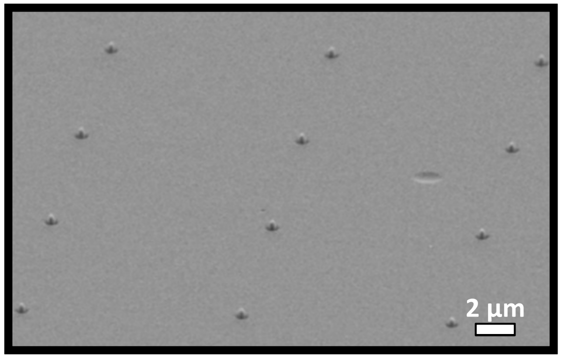
Si etch



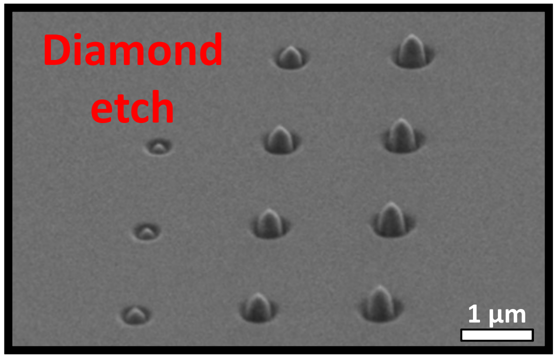
SiO_2 etch



Sub-µm diamond nanopillars

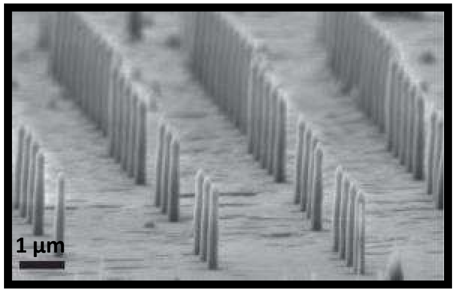


Diamond etch



Single photon sources

(Babinec et al., 2010)



Lê, Rasmussen, Pan, DTU

SiO$_2$ as e-beam resist

Thermal oxide (SiO$_2$) can be grown on Si surface of any shape/pre-structure.

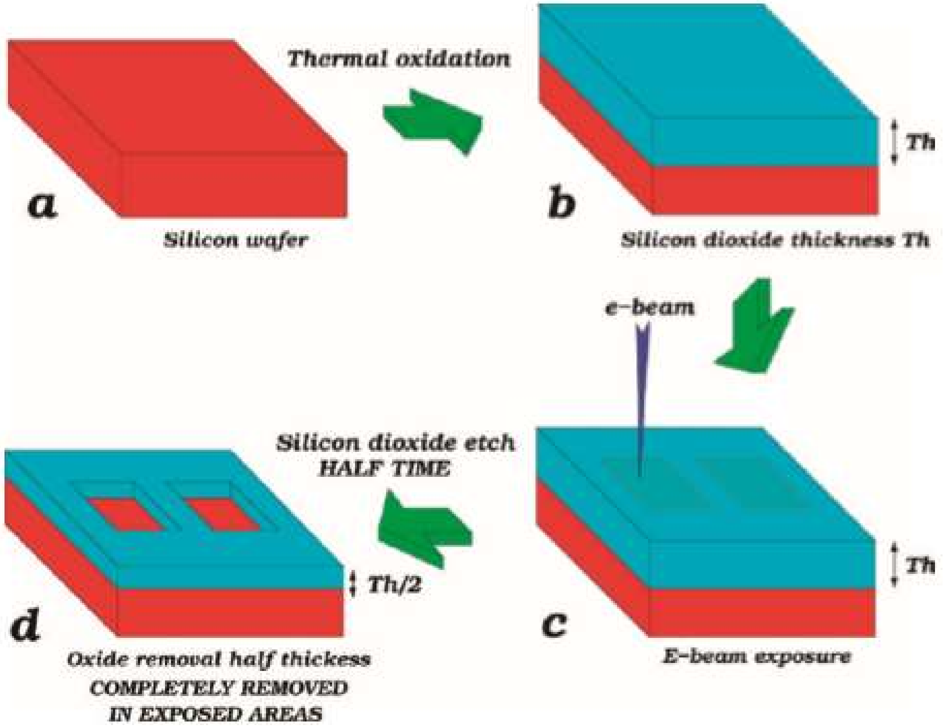


Figure 1. Schematic view of the electron beam stimulated oxide etching technique. (a) The starting substrate is a silicon wafer ⟨100⟩ oriented. (b) A silicon dioxide layer with a thickness T_h is grown by thermal oxidation. (c) Electron beam lithography is used for exposing delimited areas with a suitable dose. (d) Silicon dioxide wet etch (buffered HF) is performed for half of the time required for removing the whole SiO$_2$ layer (d). The final thickness in unexposed areas is $T_h/2$, meanwhile the SiO$_2$ is completely removed from exposed areas.

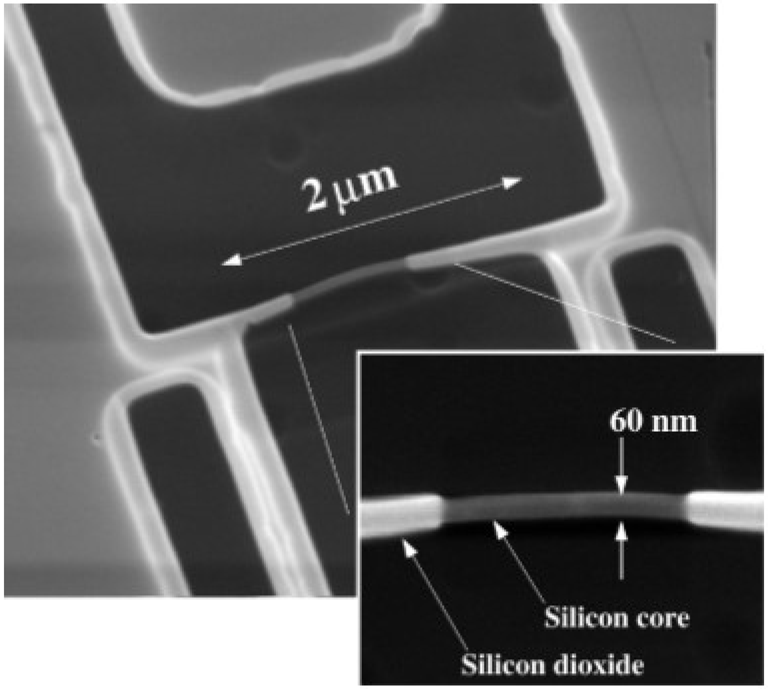


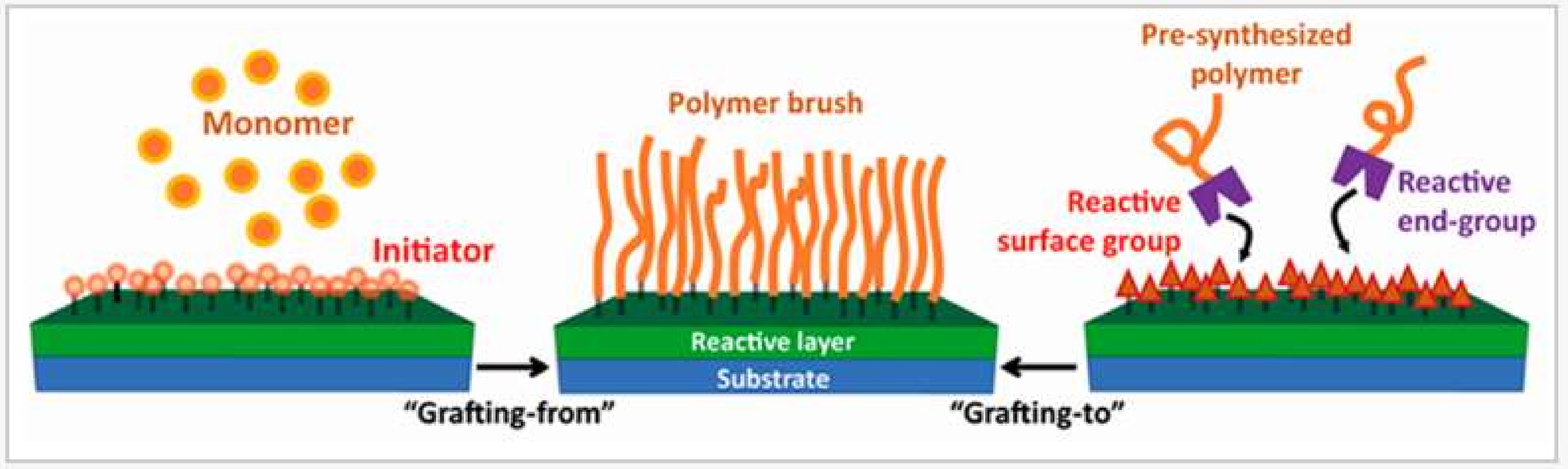
Figure 4. SEM images of a silicon nanowire positioned between silicon leads for routing the electrical signal to large area pads with a window in the oxide surrounding the nanowire. The inset shows a particular of the SiNW central region: the silicon core has been stripped from the SiO$_2$ that still surrounds all the rest of the nanowire.

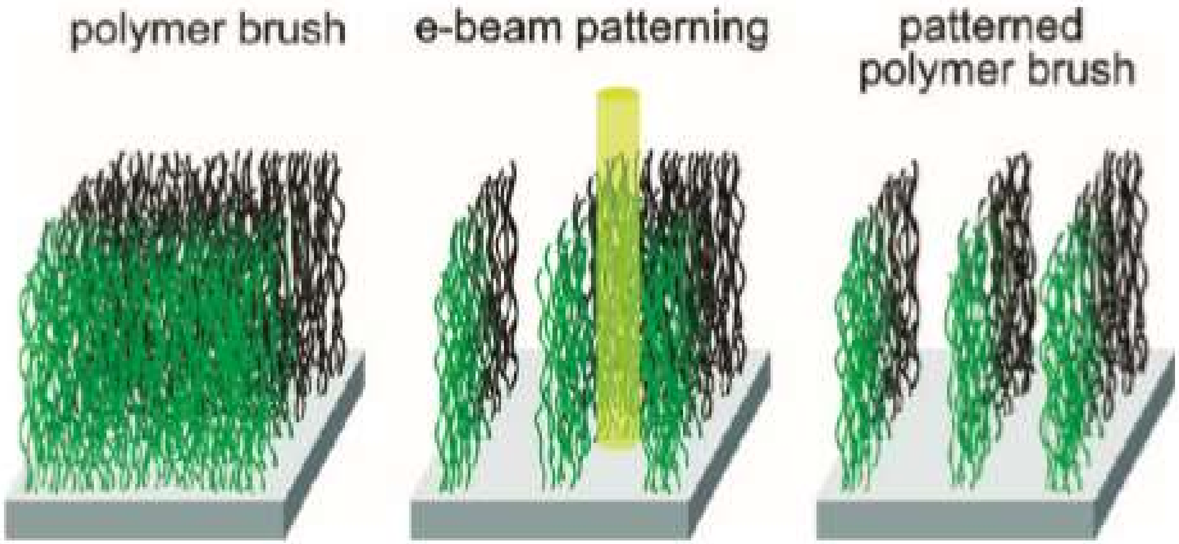
Nano Letters, 12, 1096 (2012).

Outline

- Motivation and introduction.
- E-beam lithography using evaporated resist
    - Metal halide (AlF_3, NaCl, …)
    - Non-polymeric sterol
    - Polymer (polystyrene)
- E-beam lithography using ice resist
    - Water (H_2O) ice
    - Organic ice
- E-beam lithography using mono-layer polymer brush resist
    - PMMA brush, positive and negative tone
    - Polystyrene brush, positive and negative tone
- E-beam lithography using self-assembled mono-layer (SAM) resist.

Mono-layer polymer brush as beam resist

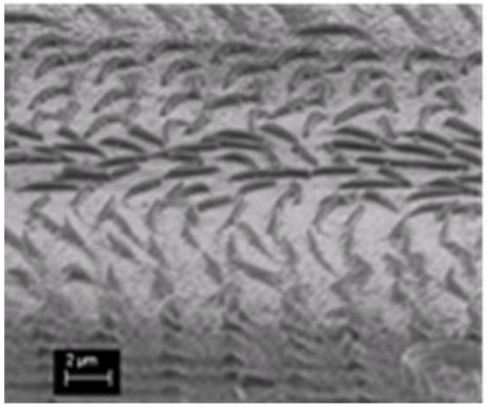




Polymers, 7, 1346 (2015);

Advantages of polymer brush resist

- Low cost process, as it employs only spin or dip-coating.
- The resist can be simple polymer such as PMMA, most popular, cheapest and easy to use.
- Monolayer chemically/firmly bonded to the sub-layer, thus no pattern collapse due to capillary force.
- Thicker than self-assembled monolayer resist (see next section), thus easier for pattern transfer.
- No “edge-bead effect” that exists when spin-coating a resist, thus suitable for patterning on ultra-small (or irregular) substrate.

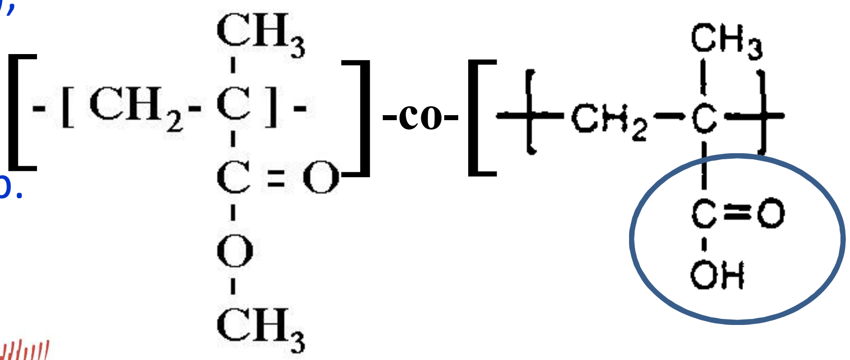


Collapsed polystyrene pillars

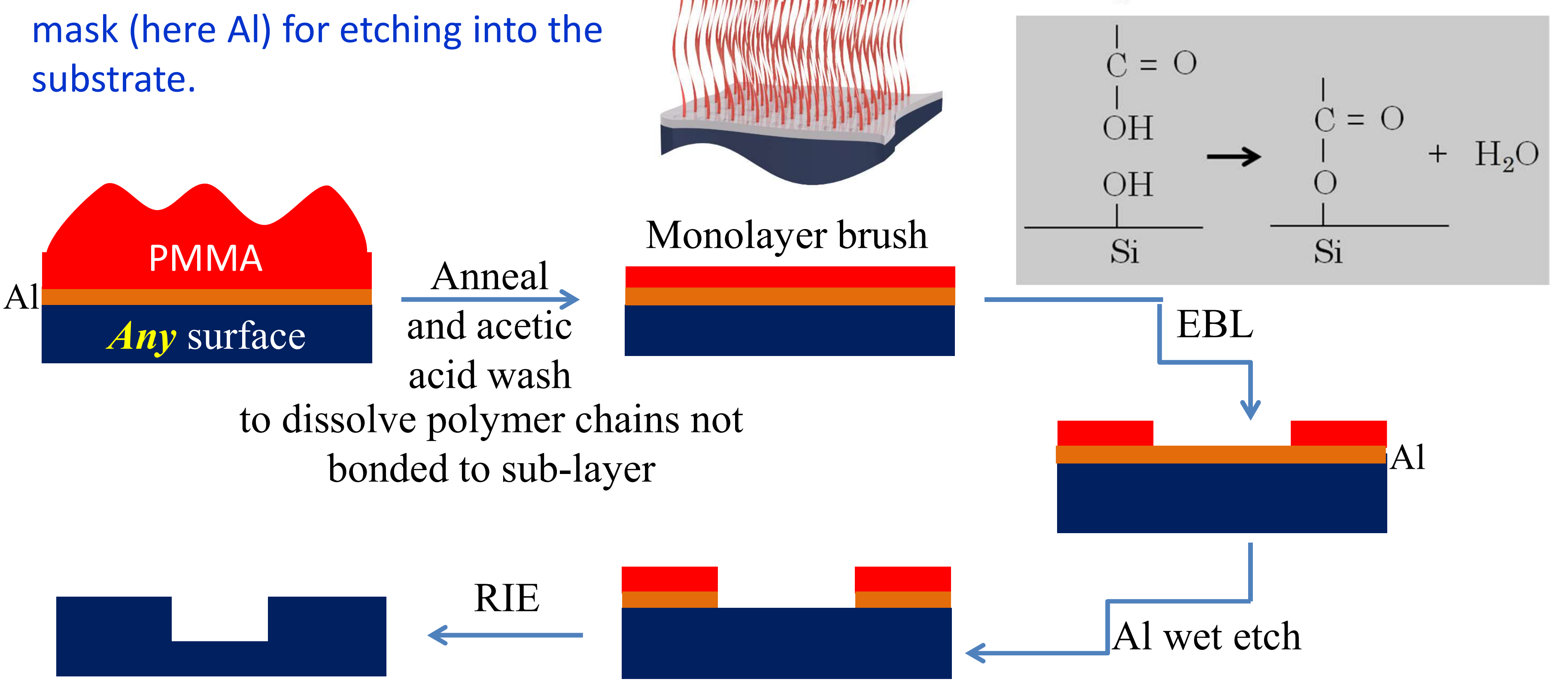
E-beam lithography using PMMA monolayer brush resist

- The PMMA contains 1.6% MAA ((meth)acrylic acid), to further promote the grafting process as MAA contains the desirable –COOH group.
- The -COOH group chemically bonds with -OH group.
- Brush is too thin for pattern transfer.
- Thus we need an intermediate hard-mask (here Al) for etching into the substrate.

PMMA-CO-PMAA

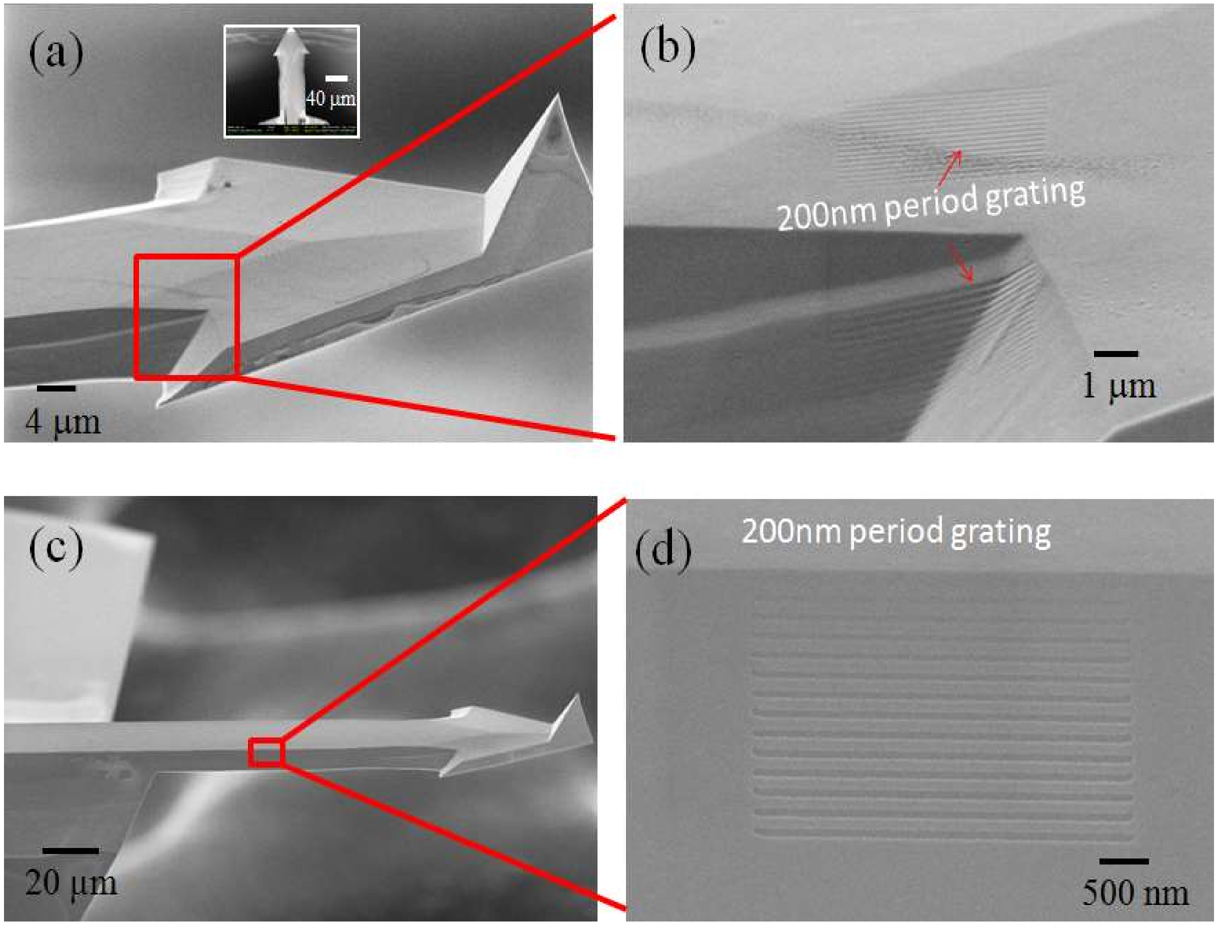


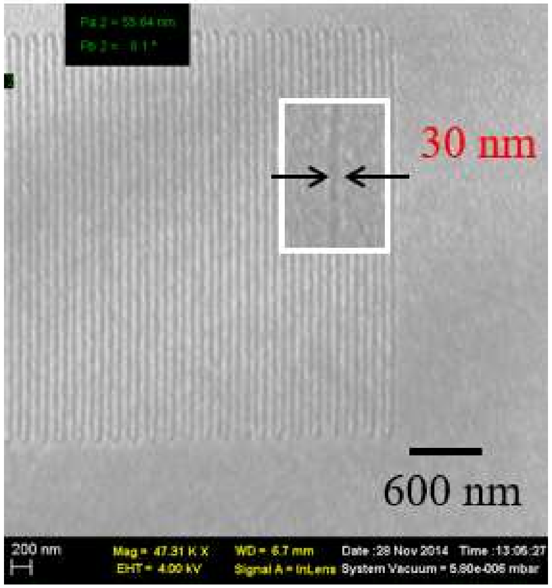
$$\left[-[CH_2-\underset{\underset{\underset{CH_3}{|}}{\underset{O}{|}}}{\overset{CH_3}{\underset{C=O}{\overset{|}{\underset{|}{C}}}}}]-\right]\text{-co-}\left[-CH_2-\overset{CH_3}{\underset{\underset{OH}{|}}{\underset{C=O}{\overset{|}{\underset{|}{C}}}}}-\right]$$



Dey and Cui, Advanced Materials - Interfaces, 4, 1600780 (2016)

High resolution grating etched into AFM cantilever (silicon)

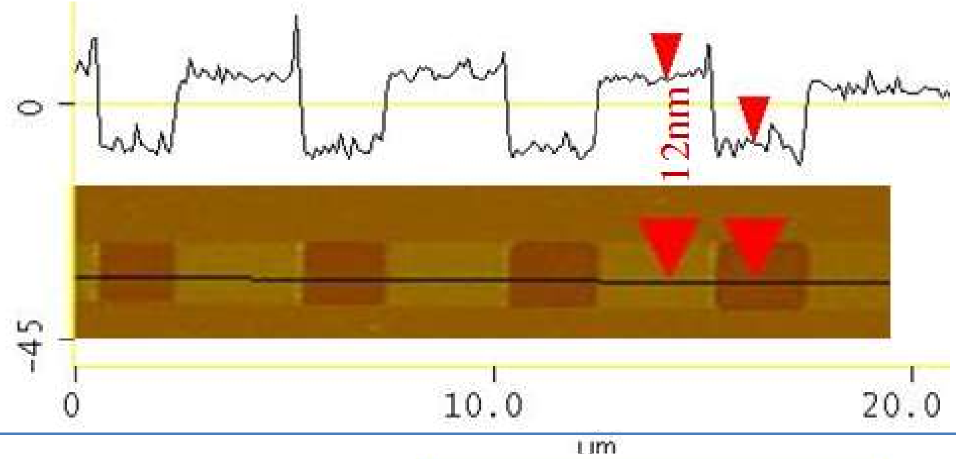




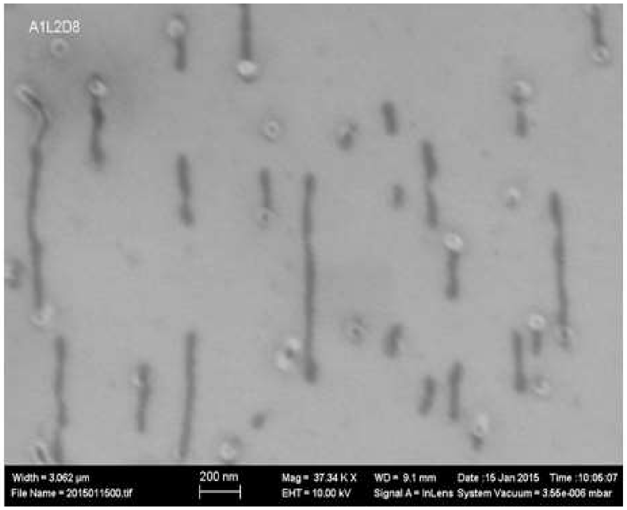
Dey and Cui, Advanced Materials - Interfaces, 4, 1600780 (2016)

Brush thickness, and as adhesion layer

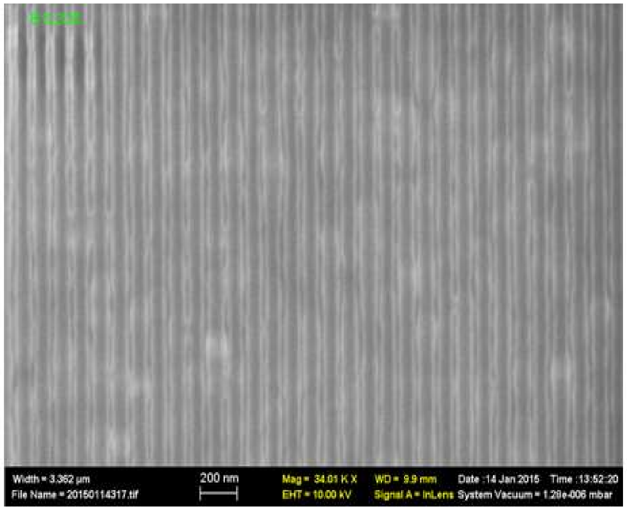
AFM image showed the mono-layer grafted brush has a height of 12 nm, PMMA, 34 kg/mol. (Here the squares were exposed and then developed)



Another application of PMMA brush monolayer: enhance resist adhesion



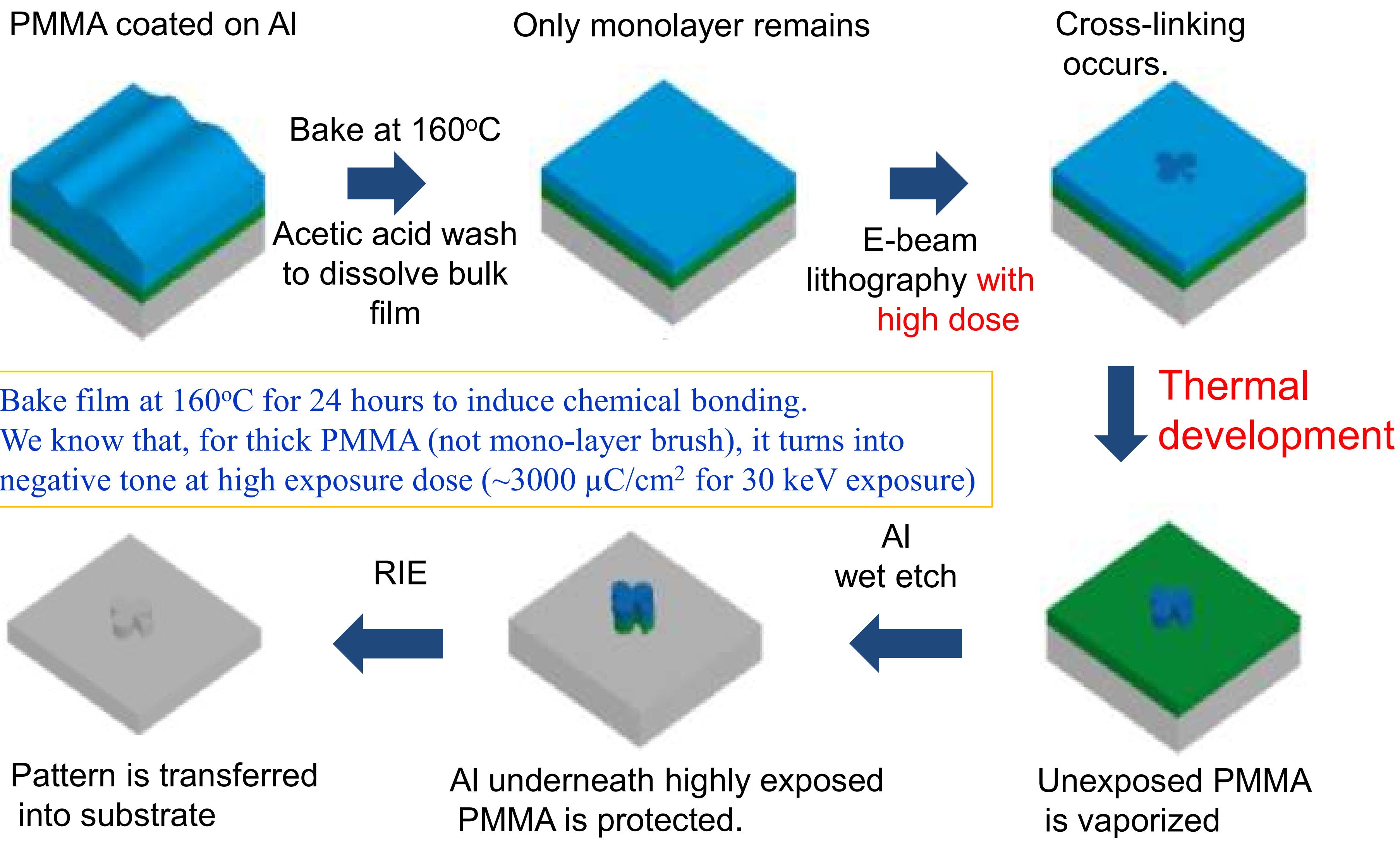
No brush, poor adhesion, almost all lines peeled off.



With brush, well defined pattern

Viscomi and Cui, JVST B, 33, 06FD06 (2015}.

PMMA brush as negative tone resist



PMMA coated on Al

Bake at 160^{o}C

Acetic acid wash to dissolve bulk film

Only monolayer remains

E-beam lithography with high dose

Cross-linking occurs.

Thermal development

Unexposed PMMA is vaporized

Al wet etch

Al underneath highly exposed PMMA is protected.

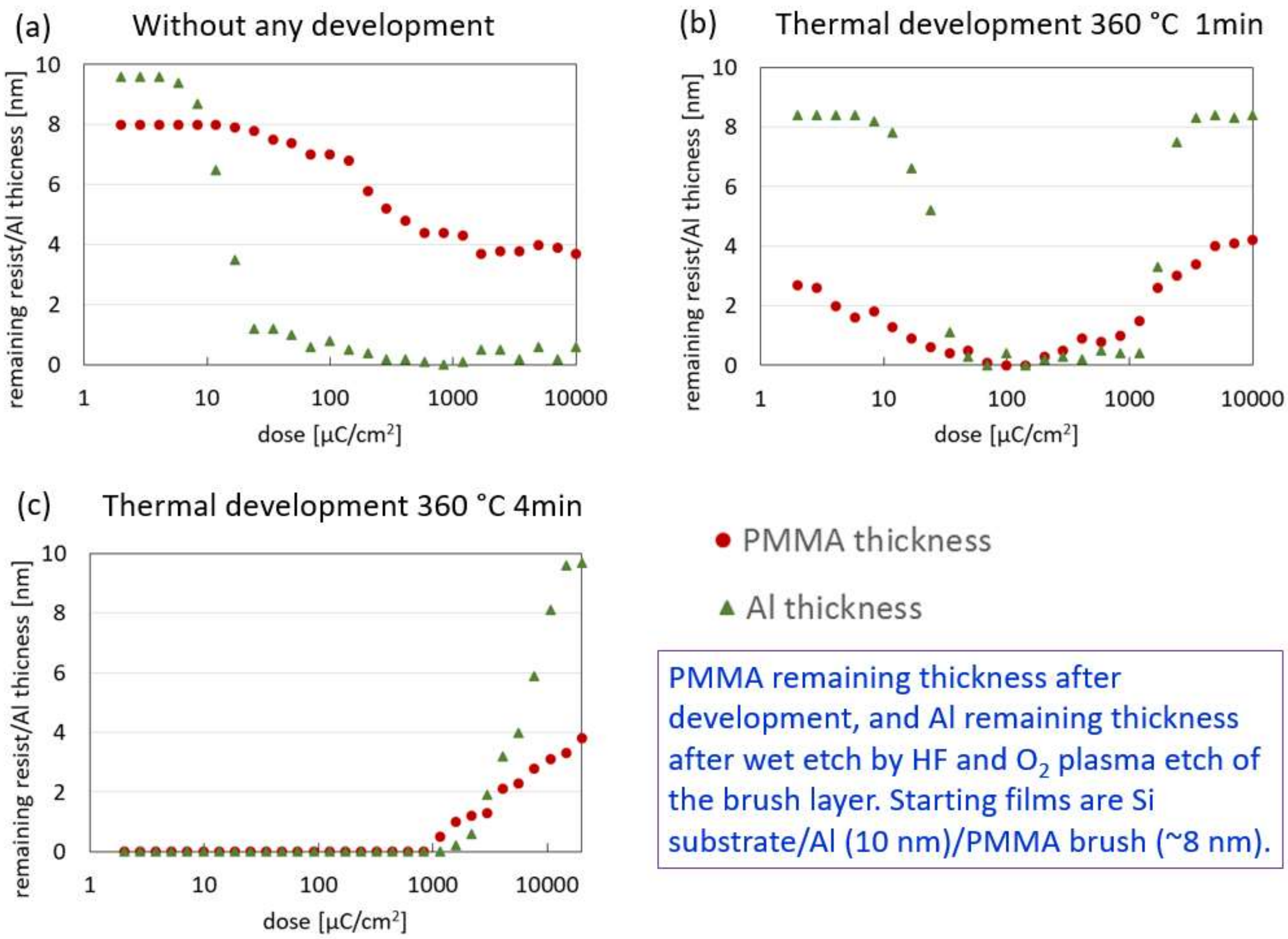
RIE

Pattern is transferred into substrate

- Bake film at 160^{o}C for 24 hours to induce chemical bonding.
- We know that, for thick PMMA (not mono-layer brush), it turns into negative tone at high exposure dose (~3000 $\mu C/cm^2$ for 30 keV exposure)

Yamada and Cui, Langmuir, 33, 13790 (2017). Con and Cui, Advances in Nano Research, 1, 105-109 (2013).

Contrast curve for thermal-developed PMMA brush

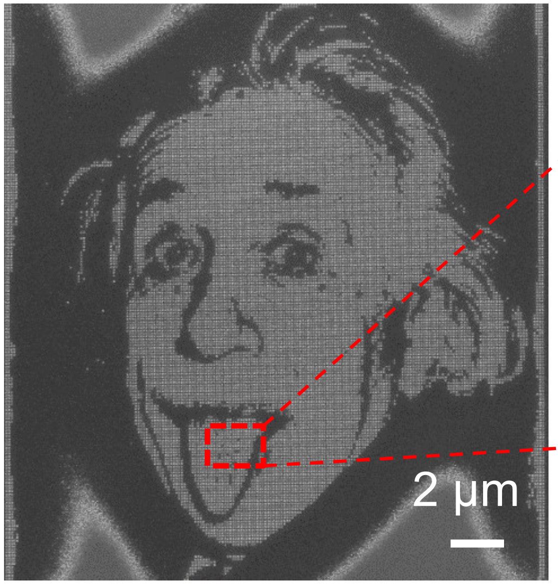


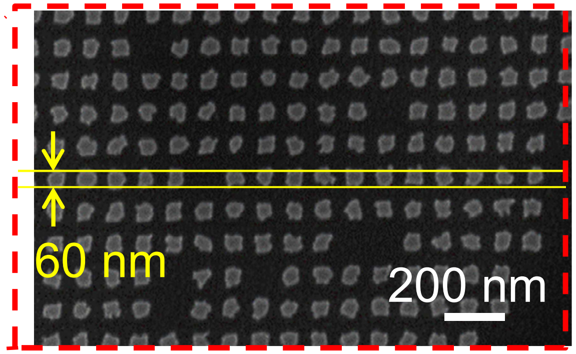
PMMA remaining thickness after development, and Al remaining thickness after wet etch by HF and O_2 plasma etch of the brush layer. Starting films are Si substrate/Al (10 nm)/PMMA brush (~8 nm).

- Exposed brush failed to protect underneath Al layer from wet etch (positive tone).
- After thermal development, heavily exposed PMMA brush recovered the resistance to wet etch (negative tone).
- 1-min 360 °C not enough to fully vaporize lightly exposed and unexposed PMMA.

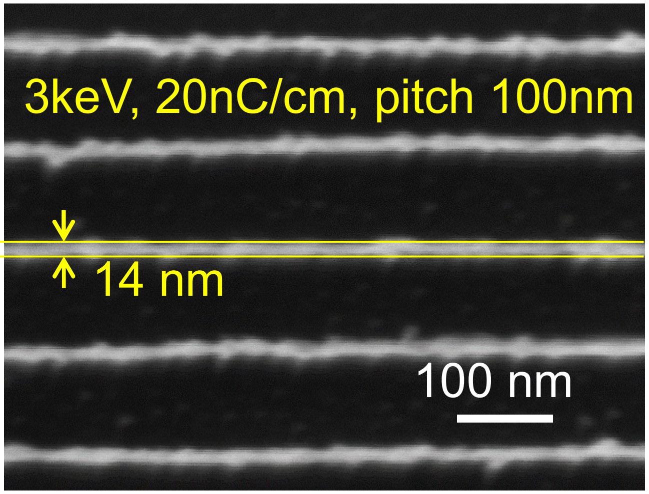
Yamada and Cui, Langmuir, 33, 13790 (2017).

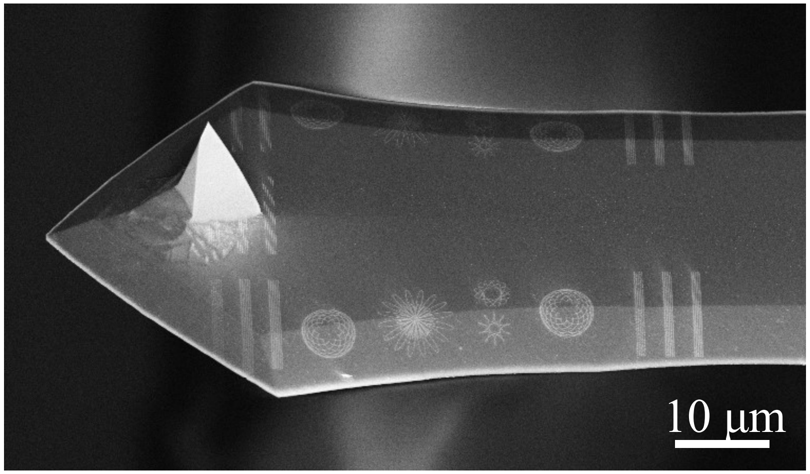
Pattern Si using PMMA brush with negative tone



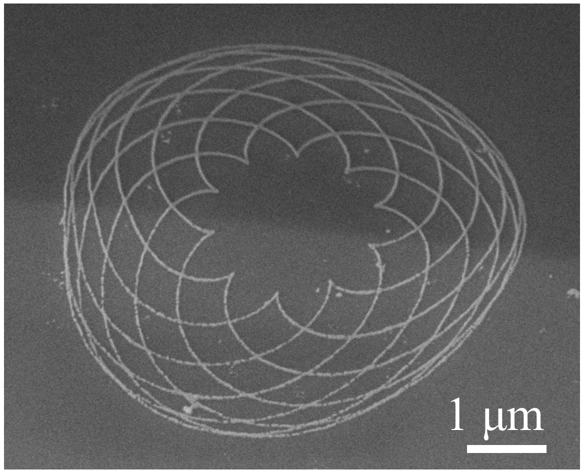


20 keV, 40,000 μC/cm², 60 nm pixel size.

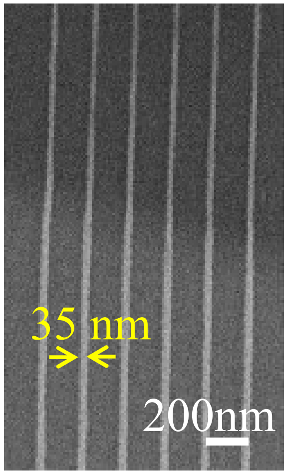




Patterns on an AFM cantilever



20keV, 400nC/cm



20keV, 400nC/cm

Yamada and Cui, Langmuir, 33, 13790 (2017).

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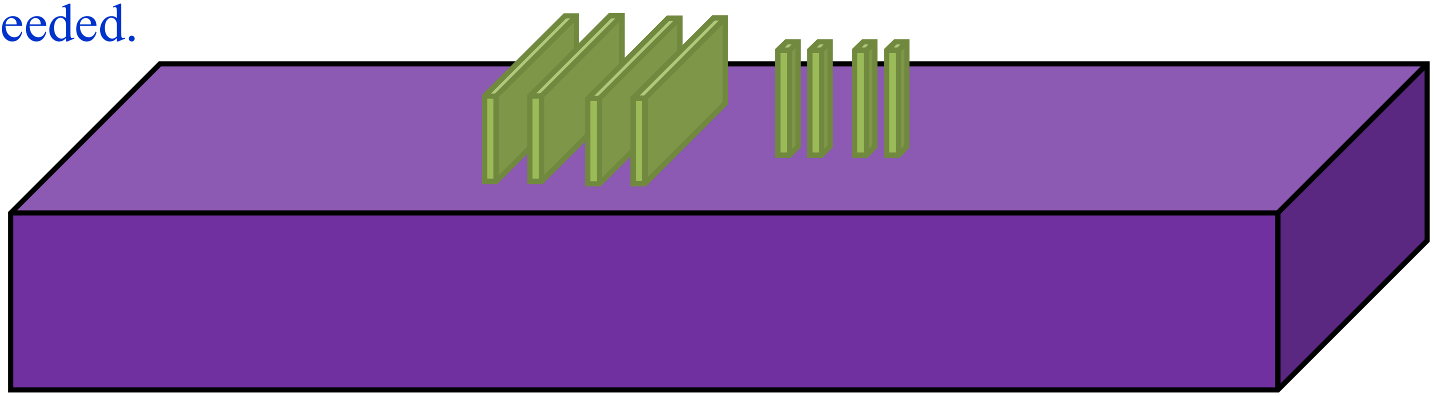
Polystyrene brush as e-beam resist

- To etch protruded structures, negative tone resist should be used.
- PMMA brush is positive tone (unless if very heavily exposed).
- whereas polystyrene is naturally a negative resist with tunable exposure properties. It is thus more preferred when negative tone is needed.

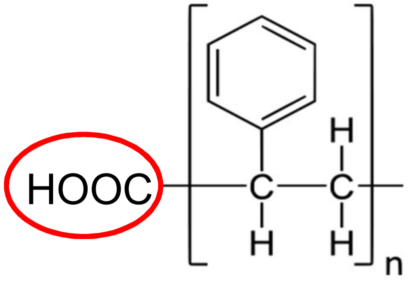


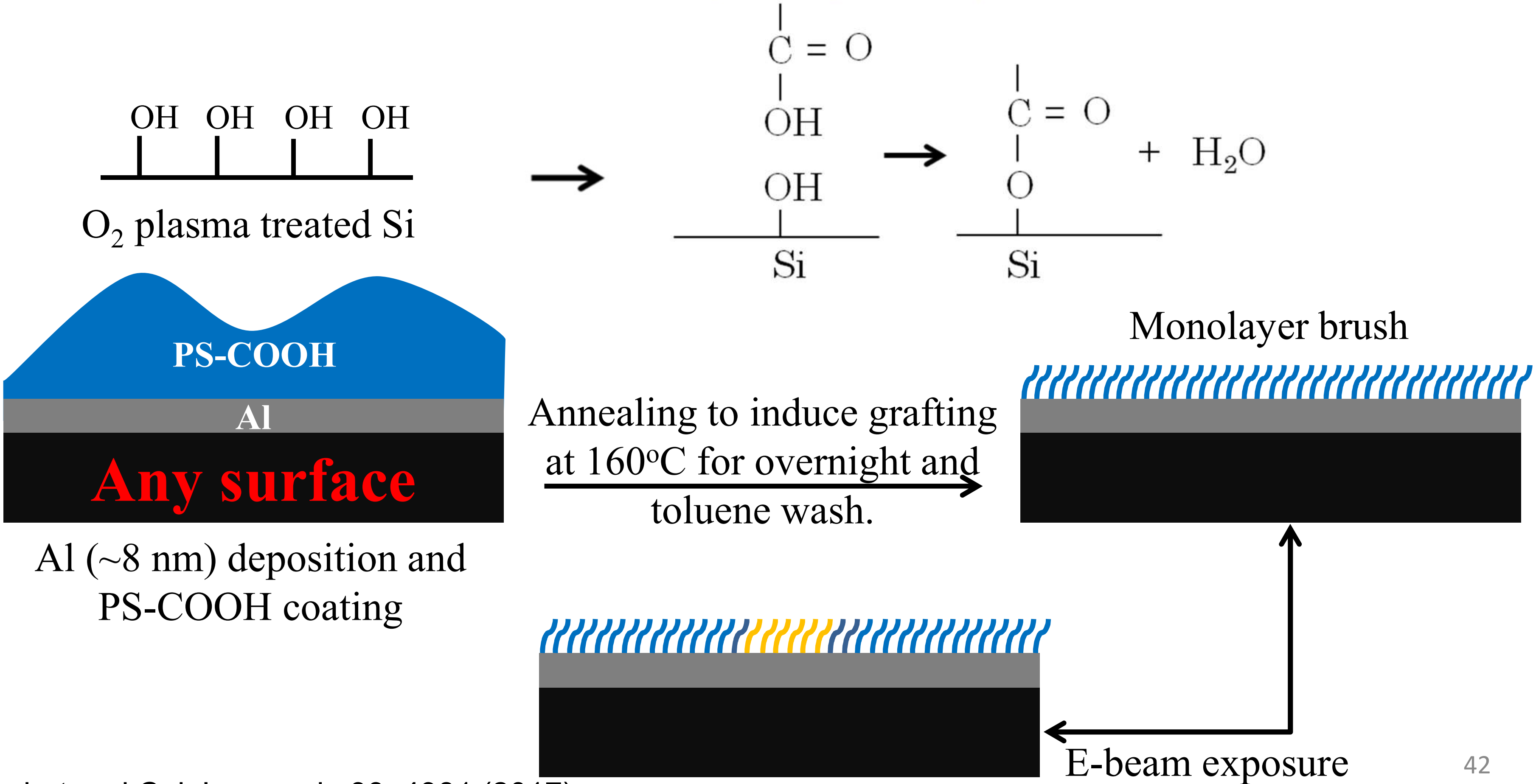
- Polystyrene (PS) can be dissolved by many common solvents. But cross-linked PS is insoluble in those solvents. Thus all those solvents can be used as developer for PS.
- But none of those solvents can dissolve PS *brush* that is chemically/firmly bonded to substrate, thus they cannot develop PS *brush* resist.
- Need unconventional method for PS brush resist development.

Ferhat and Cui, Langmuir, 33, 4981 (2017).

Polystyrene brush grafting and exposure

- Process similar to grafting PMMA brush.
- The –COOH group promotes the grafting process since it chemically bonds with -OH group. Mw 13 kg/mol.
- Dissolved in toluene for spin-coating or dip-coating.





Al (~8 nm) deposition and PS-COOH coating

Annealing to induce grafting at 160°C for overnight and toluene wash.

E-beam exposure

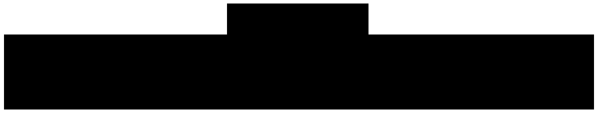
Ferhat and Cui, Langmuir, 33, 4981 (2017).

Development and pattern transfer

Negative tone

Thermal development @300ºC

Al wet etch (1:500 = HF:H_2O)



RIE

Positive tone

“Development” by etching Al in HF (1:25 = HF:H_2O)

RIE

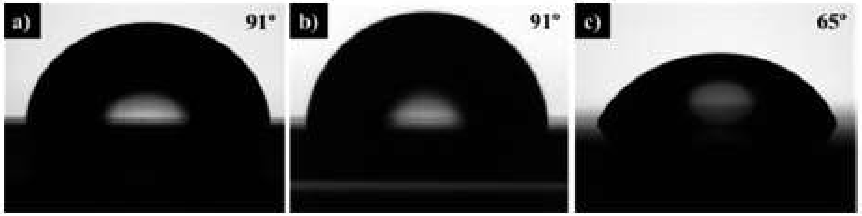
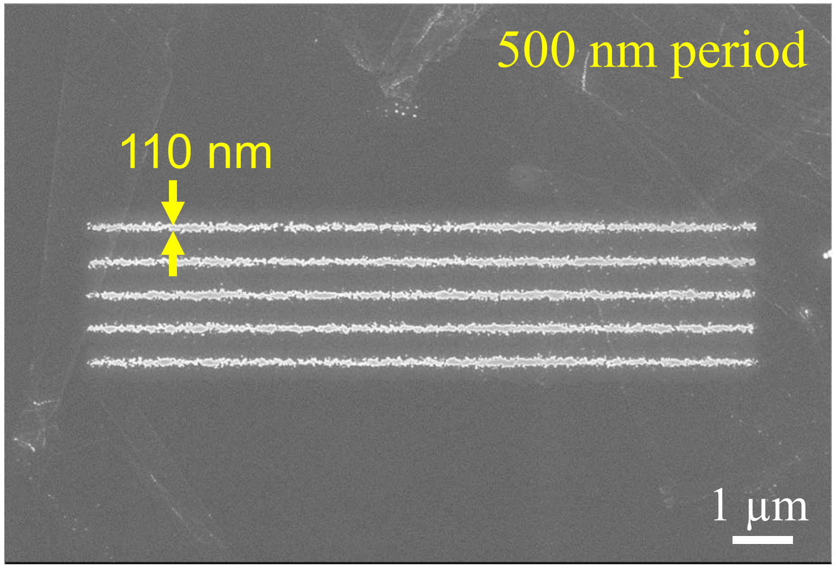


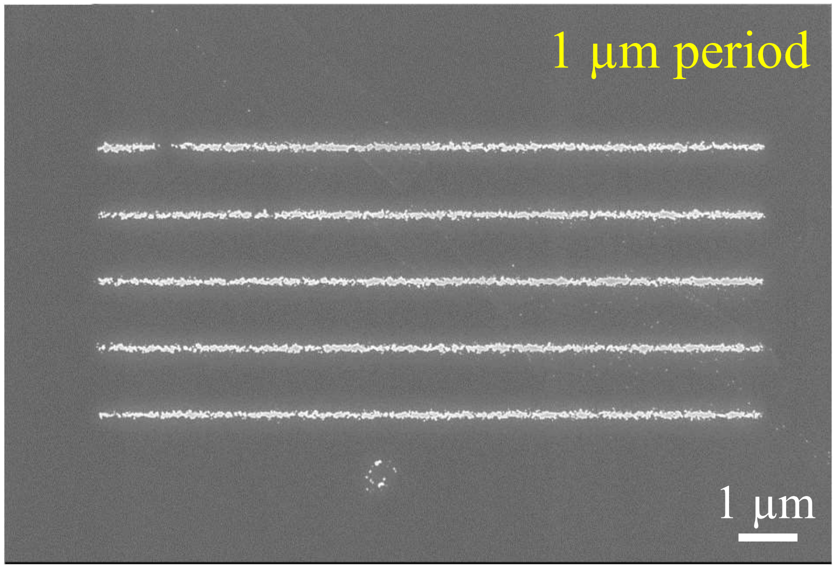
Figure 6. Contact angle measurement of (a) thick PS film, (b) non-cross-linked PS brush, and (c) cross-linked PS brush.

- Development has been done thermally for negative tone;
- Or by HF wet etching of Al for positive tone (exposed PS is more hydrophilic (smaller water contact angle), to facilitate aqueous HF etching of underneath Al).

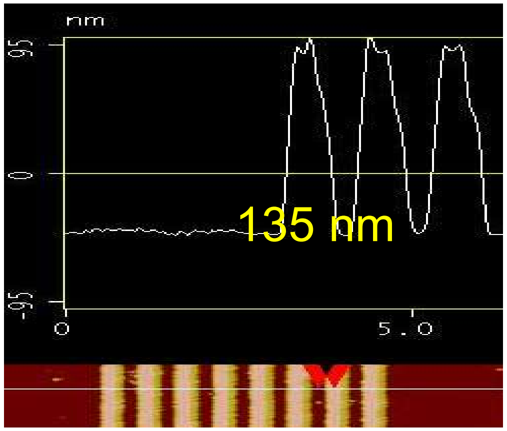
Ferhat and Cui, Langmuir, 33, 4981 (2017). Con and Cui, Advances in Nano Research, 1, 105 (2013).

Grating etched into silicon (negative tone)



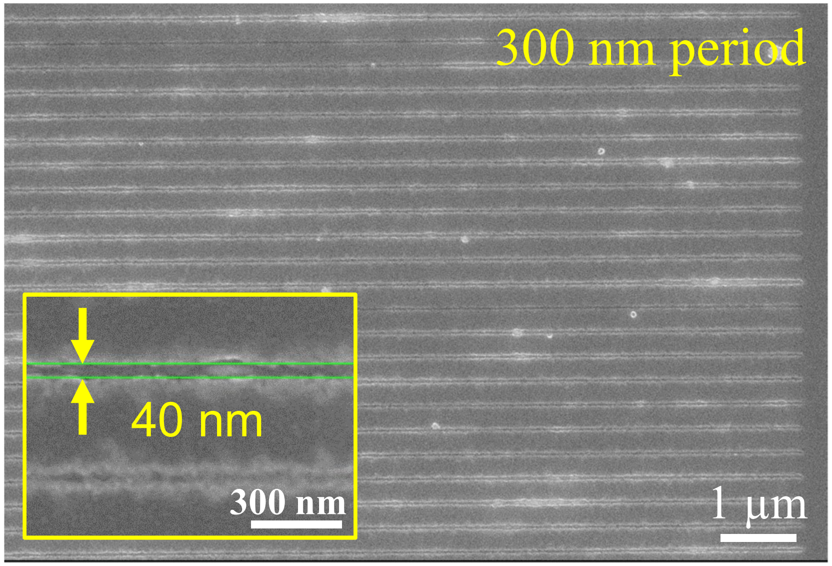


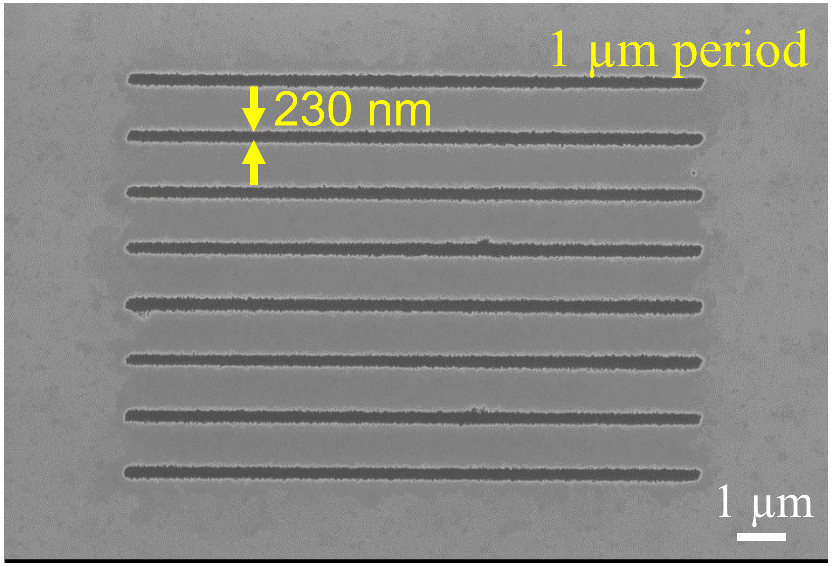
- 500 nm and 1 µm period grating, exposed at 5 keV.
- 110 nm-wide lines (protruded lines) were obtained.
- The pattern was transferred into silicon using RIE.



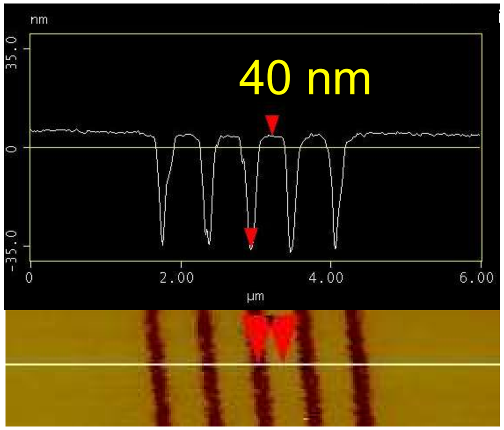
Ferhat and Cui, Langmuir, 33, 4981 (2017).

Grating etched into silicon (positive tone)



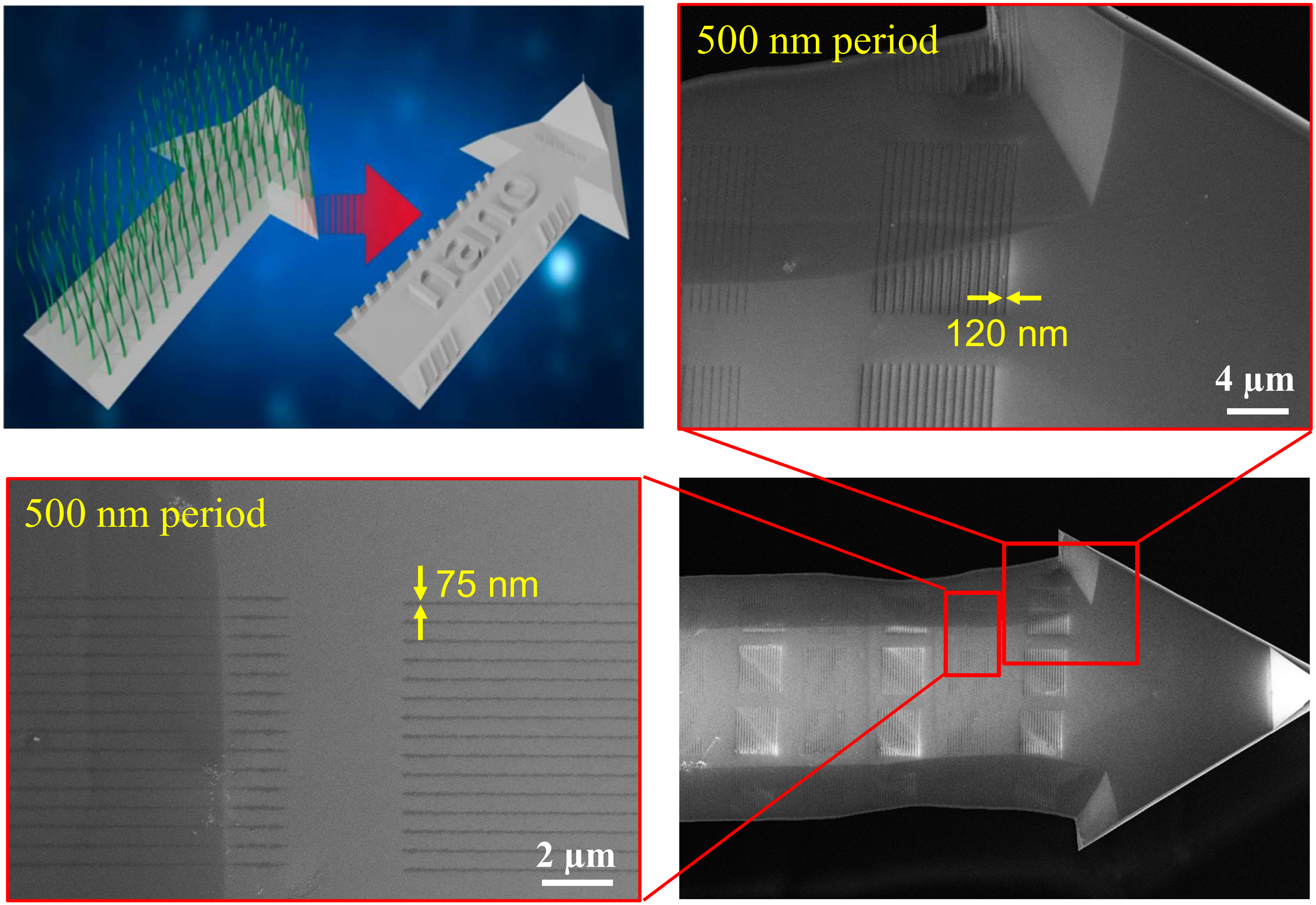


- 300 nm and 1 µm period grating, exposed at 5 keV.
- 40 nm and 230 nm wide lines (recessed trench) were obtained.
- The pattern was transferred into silicon using RIE.



Ferhat and Cui, Langmuir, 33, 4981 (2017).

Grating etched into AFM cantilever (positive tone)



Ferhat and Cui, Langmuir, 33, 4981 (2017).

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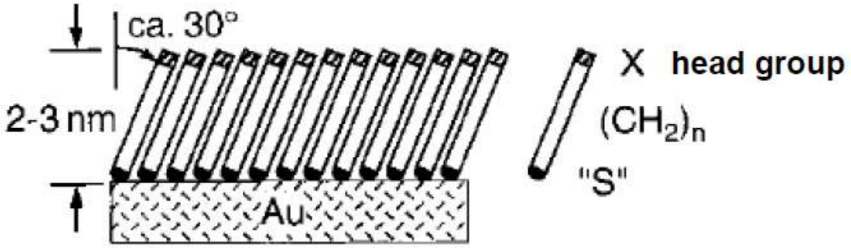
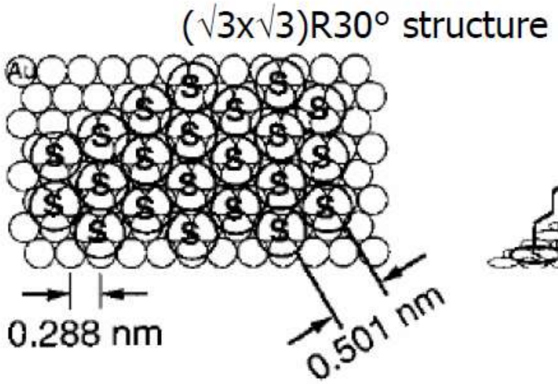
Self-assembling, classical –SH and Au bonding

- Definition: spontaneous organization of molecules (objects) into stable, well-defined structures by non-covalent forces.
- Driving force: thermodynamic equilibrium.
- Biological 3D self assembly: folding of proteins, formation of DNA helix…

$$X(CH_2)_nSH + Au^0 \longrightarrow X(CH_2)_nS^-Au^I + 1/2\ H_2$$

Self assembled monolayer (SAM)





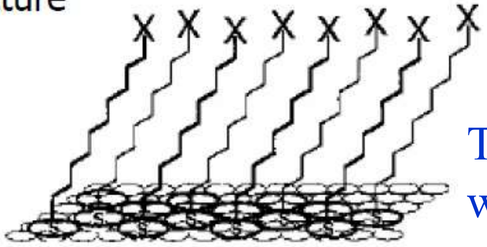
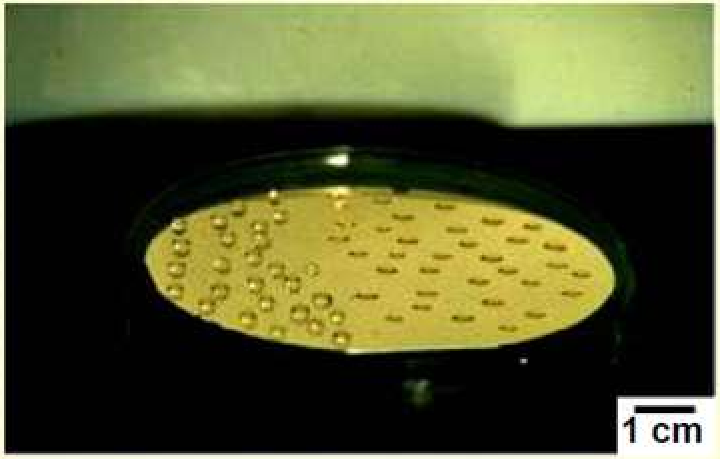


Image of Gold Surface Patterned with Hydrophobic and Hydrophilic SAMs

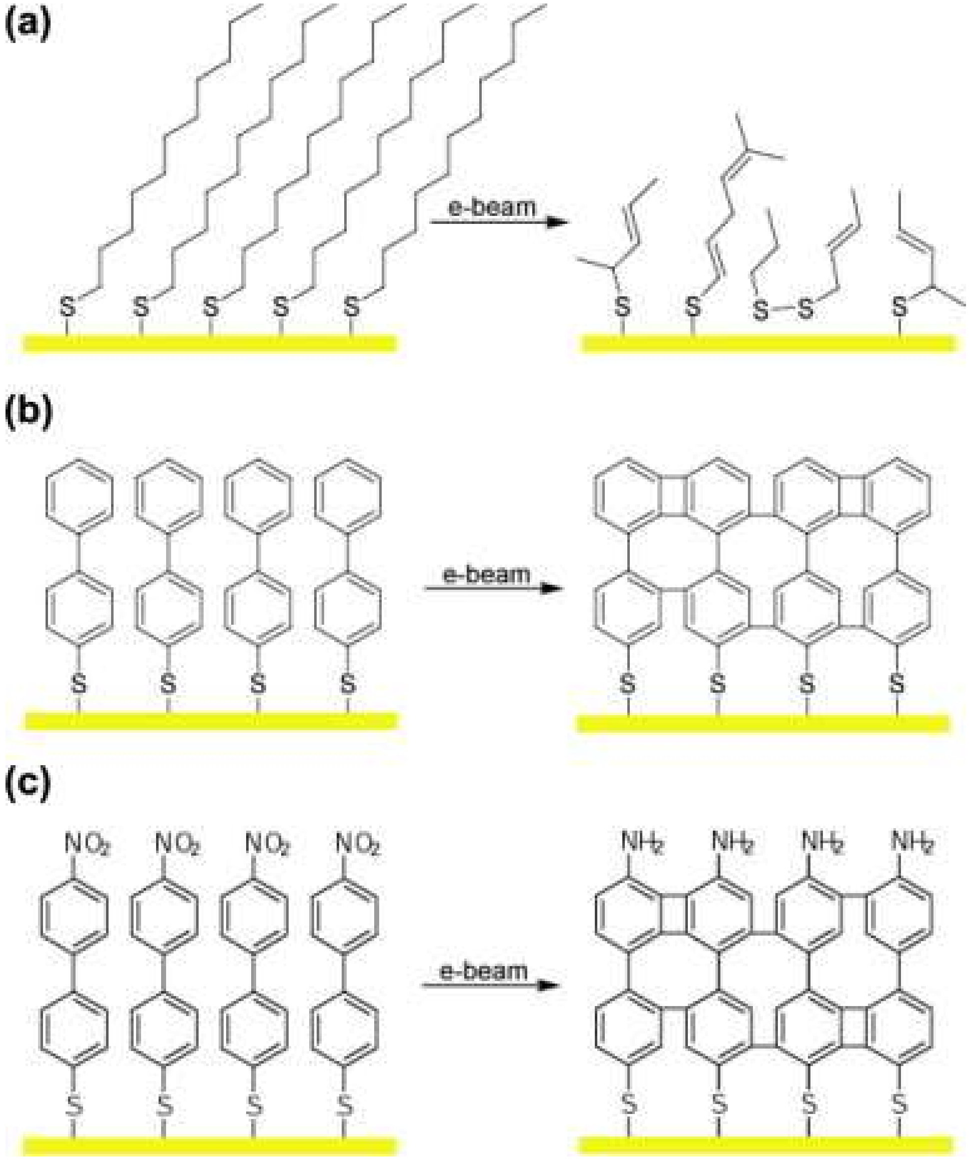


These mono-layers allow control over wettability, adhesion, chemical reactivity, electrical conduction, and mass transport to underlying metal

Thiol (-SH) also binds to Ag, but Ag surface not as stable as Au.

Laibinis and Whitesides, JACS, 113, 152 (1991); Xia and Whitesides, Angew. Chem., Int. Ed. 37, 550 (1998).

Electron interaction with SAM resist

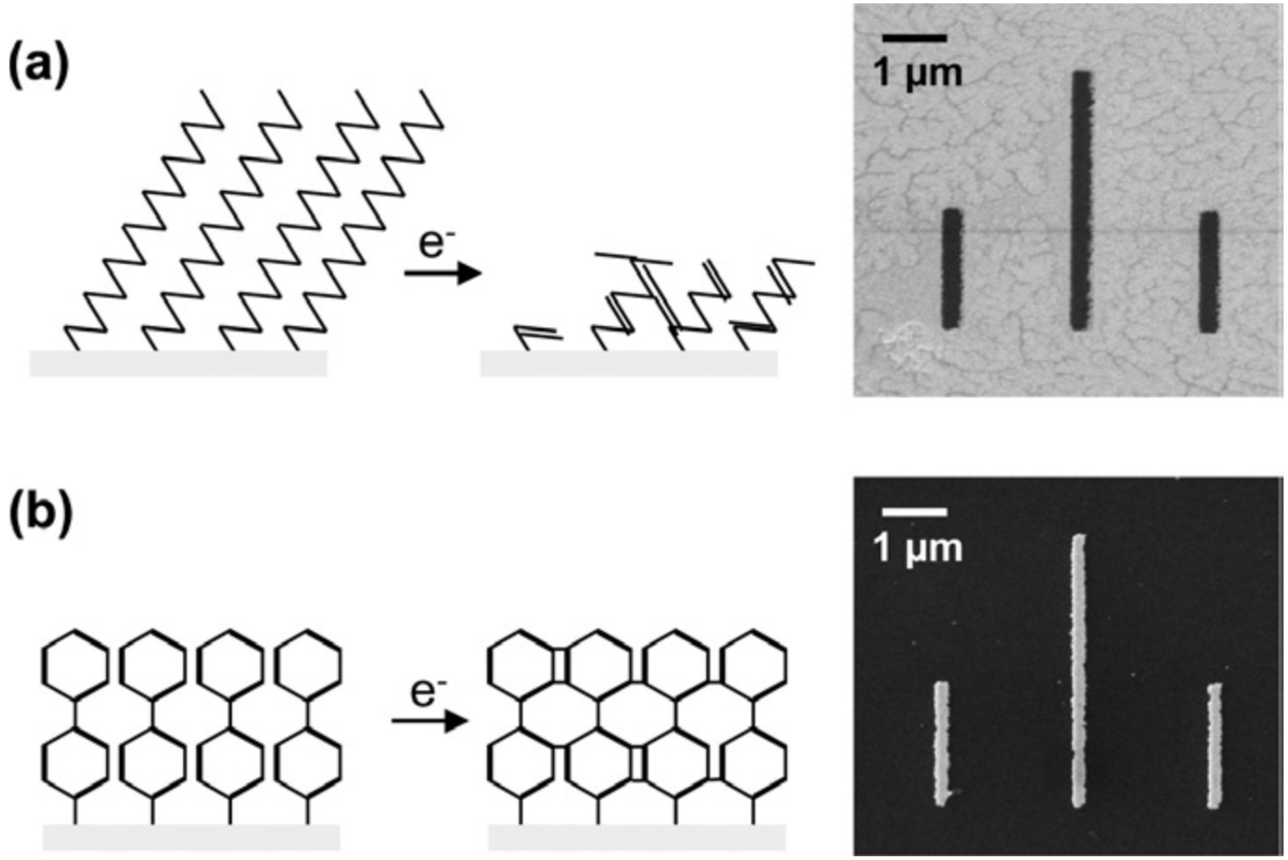


SAM – self assembled monolayer

- In *aliphatic* SAMs, electrons induce a cleavage of C–H bonds forming C = C double bonds. Positive tone.
- In *aromatic* SAMs, first C–H cleavage, followed by crosslinking between neighboring phenyl units. Negative tone.
- In aromatic SAM terminated with nitro groups, again C–H cleavage occurs; but liberated H-atoms locally reduce the -NO_2 to -NH_2 group, which can be further chemically modified by electrophilic agents.

Progress in Surface Science, 87, 108 (2012)

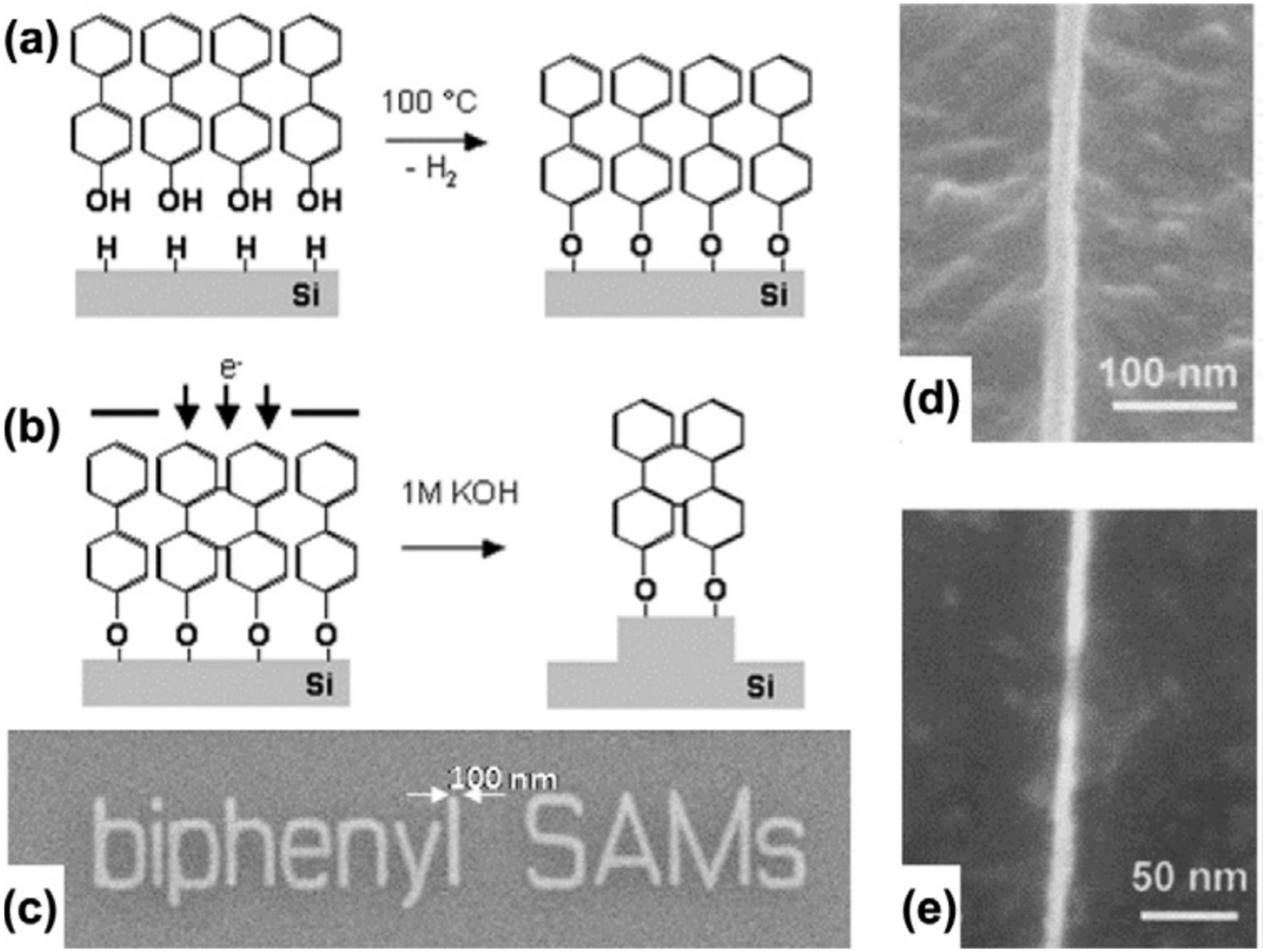
Patterning Au using SAM resist



a) Etched lines in a gold film; hexadecanethiol (HDT) was employed as a positive resist SAM.
b) Gold lines on silicon; biphenylthiol (BPT) was employed as a negative resist.

Progress in Surface Science, 87, 108 (2012)

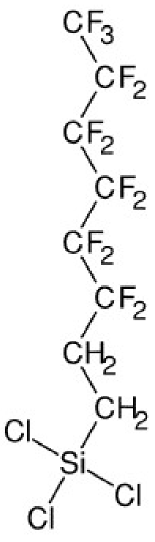
Patterning Si using SAM resist



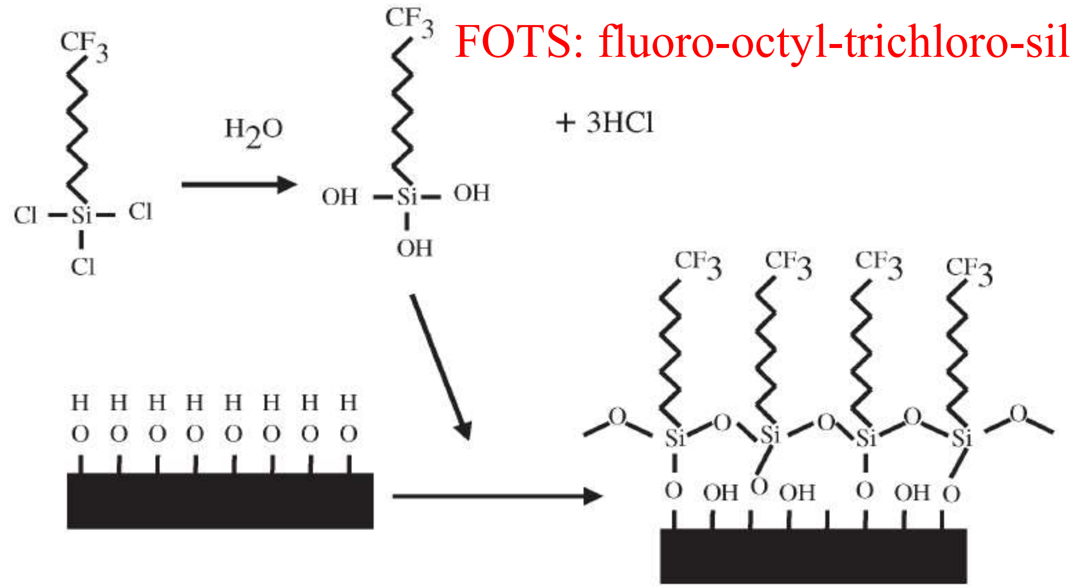
Application of hydroxybiphenyl SAMs as e-beam resists for patterning silicon

Progress in Surface Science, 87, 108 (2012)

EBL using FOTS SAM resist



FOTS



FOTS: fluoro-octyl-trichloro-silane

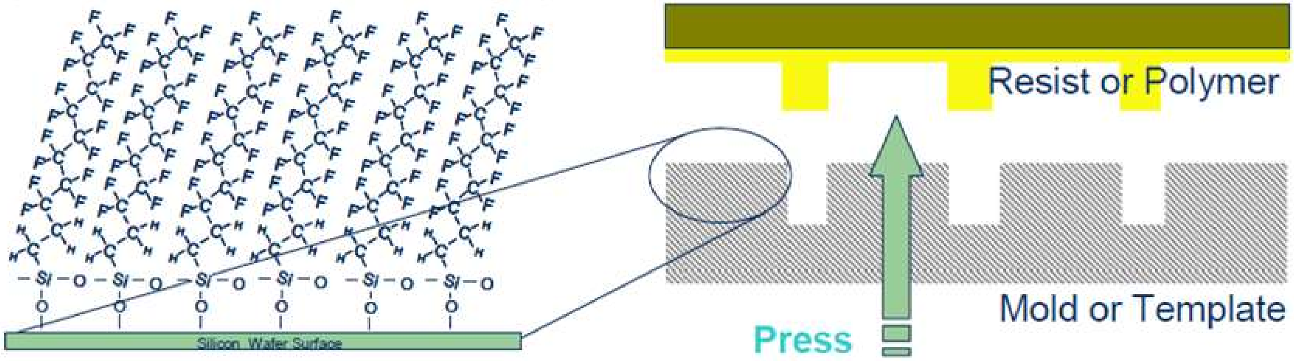
Fig. 1. Formation mechanism of SAM on the hydroxylized silicon substrate.

Put the wafer and a tiny drop of FOTS into the desiccator.

- FOTS or FDTS is popularly used as mold release agent for nanoimprint lithography.
- SAM is ~1nm thick, vs. 10nm for polymer brush, thus more challenging for pattern transfer.

FDTS SAM

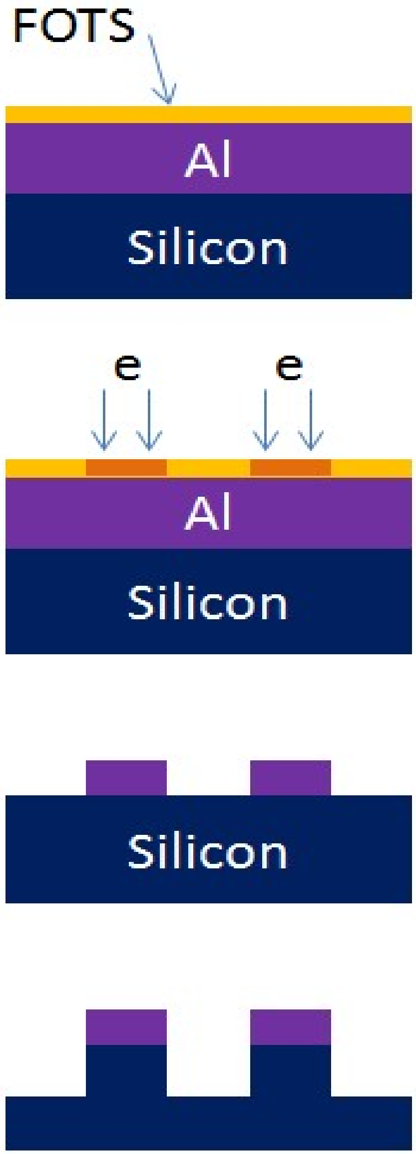
(SAM: self assembled monolayer)



Zhang, Journal of Microelectromechanical Systems, 16, 1451 (2007)

Patterning Si using FOTS SAM resist

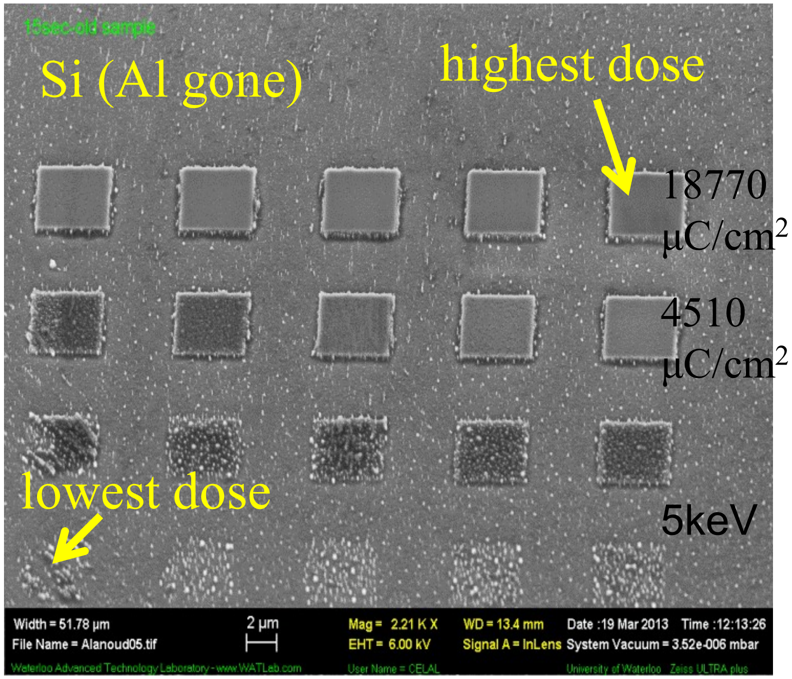
- Coat 10 nm Al on bare silicon wafer.
- FOTS silane treatment in a vacuum container for >10 hours.
- Bake SAM on hotplate at 150°C for 20 min.
- Expose FOTS at 5 keV, no development was conducted here.
- Dip sample into PAN solution (16:1:1:2 mixture of H_3PO_4:HAc:HNO_3:H_2O, HAc is acetic acid) to etch the Al underneath at room temperature.
- Transfer pattern into silicon substrate using SF_6/C_4F_8 gas, which etches silicon ~100× faster than Al.

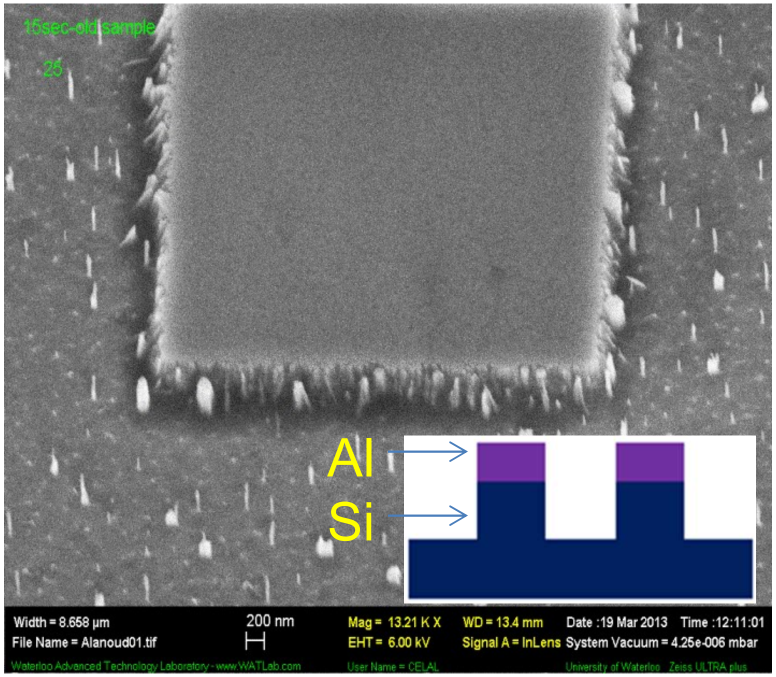


Shown here is for negative tone, can also be positive tone, see next slides.

Alshammari and Cui, MNE 2013.
(manuscript not prepared yet)

FOTS as negative resist when exposed with high dose





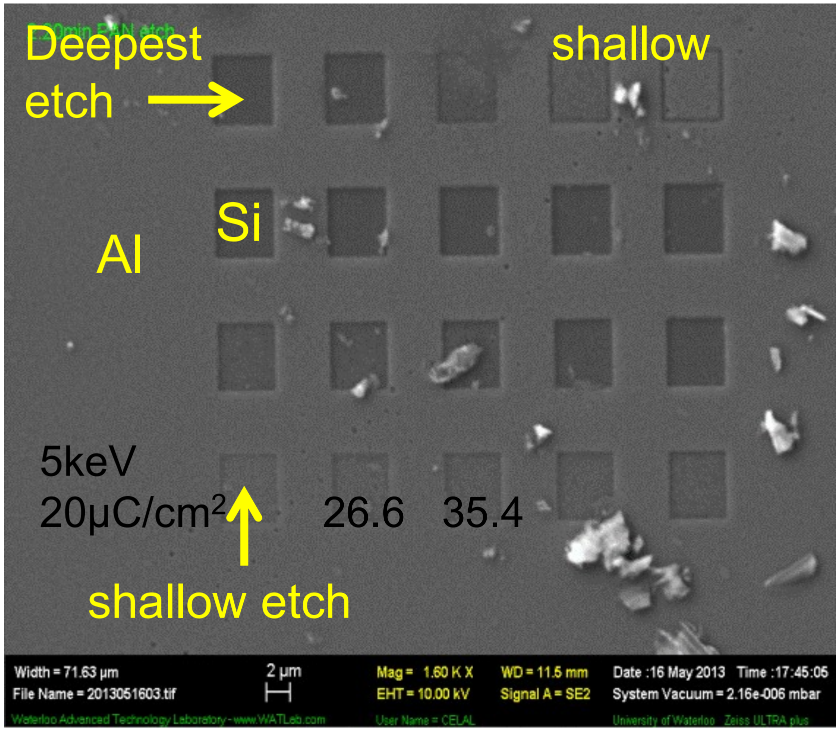
Dose increased exponentially from lower left to upper right.

- With over-etch, the Al on the <u>un</u>exposed area was completely etched away.
- FOTS behaved as a **negative** resist when exposed with high enough dose – the heavily exposed SAM increasingly protected the Al underneath with increasing exposure doses.

Alshammari and Cui, MNE 2013.

FOTS as positive resist when exposed with low dose

- With Al **under**-etch, the Al on the unexposed area was not completely etched away.
- FOTS behaved as a **positive** resist when exposed with low dose – the lightly exposed SAM failed gradually to protect the Al with increasing exposure doses.
- For high dose (the square at upper right corner), the resist showed a "**neutral**" tone (turning point between positive and negative tone).



We know that for thick PMMA it is a positive resist with sensitivity of ~250 µC/cm^2 (at this dose, chain cut short to <~10 kg/mol), but turns into negative tone at high exposure dose (~3000 µC/cm^2 for 30 keV exposure). That is, further exposure will cause cross-linking of the chain-cut PMMA. Here the results implies that SAM of FOTS has similar exposure property.

Alshammari and Cui, MNE 2013.

Summary

- E-beam lithography using evaporated resist
  - Metal halide (AlF_3, NaCl, …)
  - Non-polymeric sterol
  - Polymer (polystyrene)
- E-beam lithography using ice resist
  - Water (H_2O) ice
  - Organic ice (anisole…)
- E-beam lithography using mono-layer polymer brush resist
  - PMMA brush, positive and negative tone
  - Polystyrene brush, negative and positive tone
- E-beam lithography using self-assembled mono-layer (SAM) resist.

Thanks for your attention!

Waterloo Nanofabrication Group