
Nanometer Scale Patterning and Processing

Spring 2016

Lecture 28

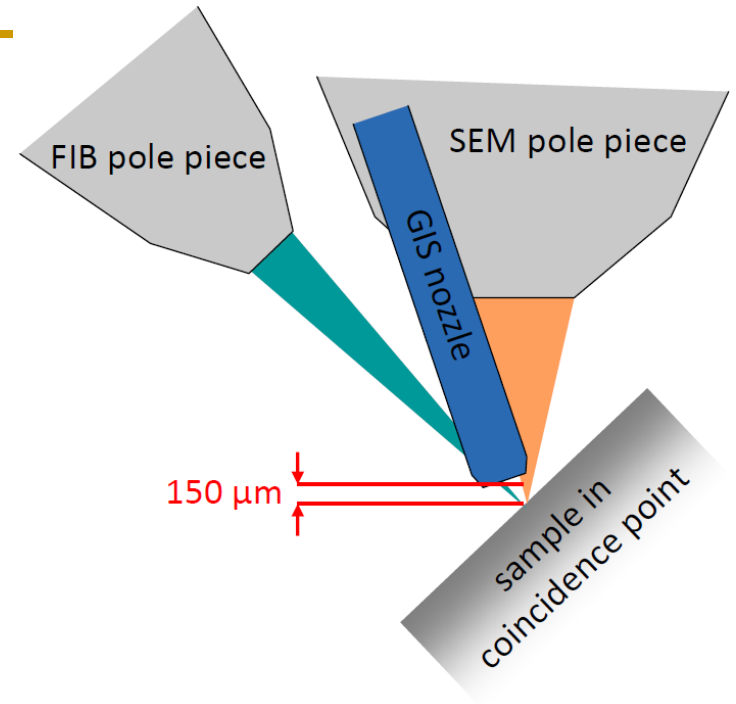
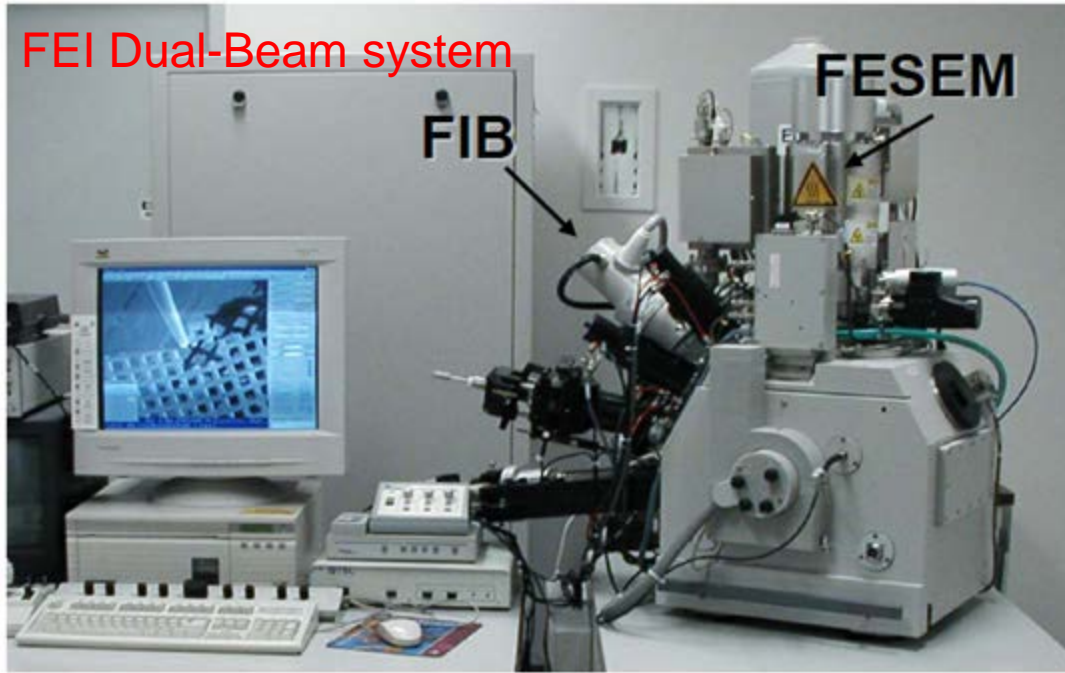
Focused Ion Beam Induced Deposition

Focused ion beam (FIB)

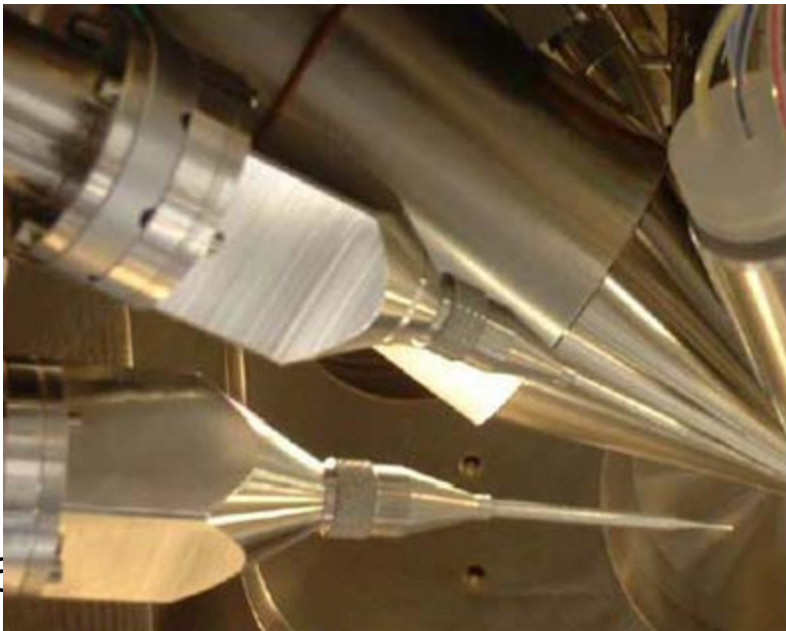
1. Focused ion beam induced deposition.
2. Focused electron beam induced deposition.
3. Deposition rate (electron and gas flux-limited regimes)
4. Deposit composition (carbon/metal)

DualBeam FIB/SEM plus GIS

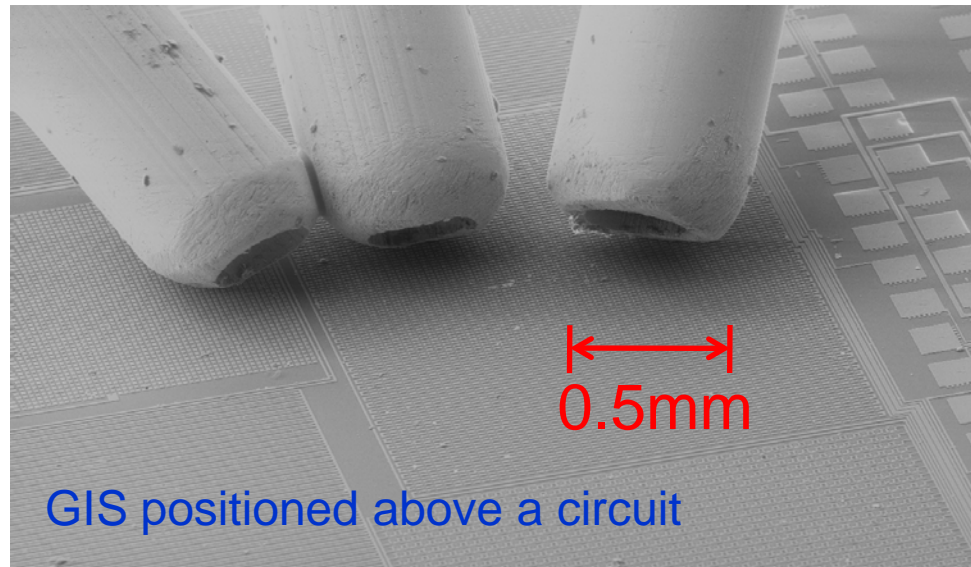
FEI Dual-Beam system



GIS: Gas Injection Systems

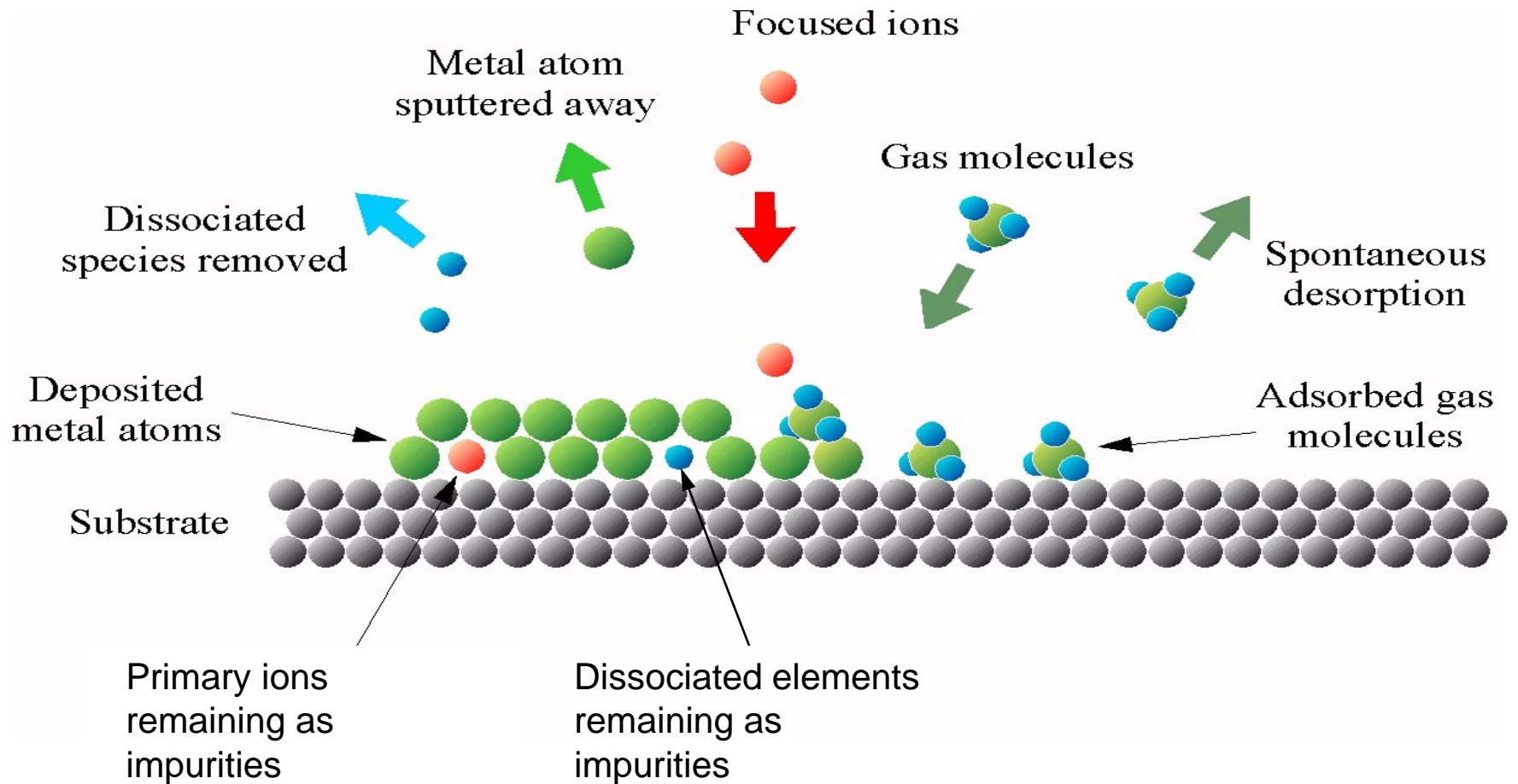


GIS: Gas Injection Systems



- Gases are delivered to the sample surface via a needle in close proximity to the surface ($\sim 50\text{-}150\mu\text{m}$).
- Gas pressure is of order 10^{-5} mbar.
- Interaction of ion beam with gas causes either enhanced removal of sample material, or deposition of one of the elements within the gases.
- A metal-organic gas is used to deposit metals via ion/electron beam assisted chemical vapor deposition.
- An insulator can also be deposited, e.g. SiO_2 from tetraethyl orthosilicate (TEOS).
- Reactive gases are used to etch samples via reactive ion etching.

FIB assisted (chemical vapor) deposition process



1. Adsorption of gas molecules on substrate

2. Dissociation/decomposition of gas molecules by the ion beam

3. Deposition of the material atoms and removal of the organic ligands

"Studies of structures elaborated by focused ion beam induced deposition", Prestigiacomo, MEE 2004.

Gases used for metal, oxide and C deposition

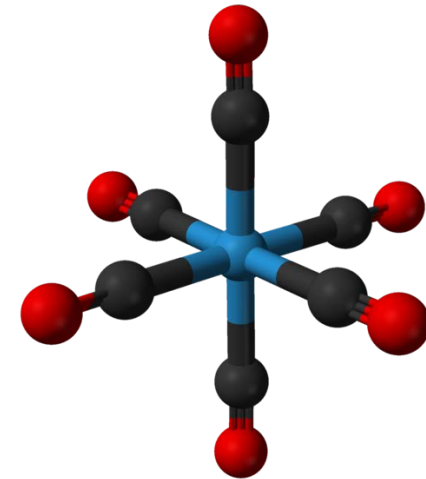
Gas	Yield (atoms/ion)	Composition (M : C : Ga)	Resistivity ($\mu\Omega\text{cm}$)
$\text{W}(\text{CO})_6$	2	75:10:10	150-225
$\text{Mo}(\text{CO})_6$	2-3	67:19:12	200
$(\text{CH}_3)_3\text{NAlH}_3$	≈ 5		900
$\text{C}_9\text{H}_{17}\text{Pt}$	2.5 -35	45:24:28	70-700
$\text{Au}(\text{hfac})(\text{CH}_3)_2$	3-8	50:35:15	500
$\text{Cu}(\text{hfac}) \text{TMVS}$	10-30		100 (*5)

hfac – hexafluoro acetyl
acetanoate

TMVS – trimethyl vinyl silane

More deposition materials:

- Al: Trimethylamine alane (TMAA), 1Torr vapor pressure at 25°C
- SiO_2 : O_2 and tetramethoxysilane (TMOS)
 $\text{Si}(\text{OCH}_3)_4$
- C: phenanthrene



Ion beam assisted deposition

Properties:

- Resistivity much higher than bulk value (10-5000×)
- Carbon contamination
- Gallium contamination
- Chemistry not well understood

Collision cascade model – Au deposition

- In principle, decomposition of precursor gas may take place either in the gas phase or on the surface; but actually deposition rate insensitive to gas pressure, so gas phase decomposition is unimportant.
- Deposition goes beyond beam spot size with yield higher than would be by only direct ion-molecule collision.
- Yield better correlated with energy loss to the nuclei than with energy loss to the electrons.
- Therefore, collision cascade has to play an important role in the decomposition.
- In the case of gold, sputtering (of excited surface atoms) will occur for $E > 3.8\text{eV}$.
- For energy $E > 0.95\text{eV}$ (dissociation energy of the adsorbate), deposition will occur.

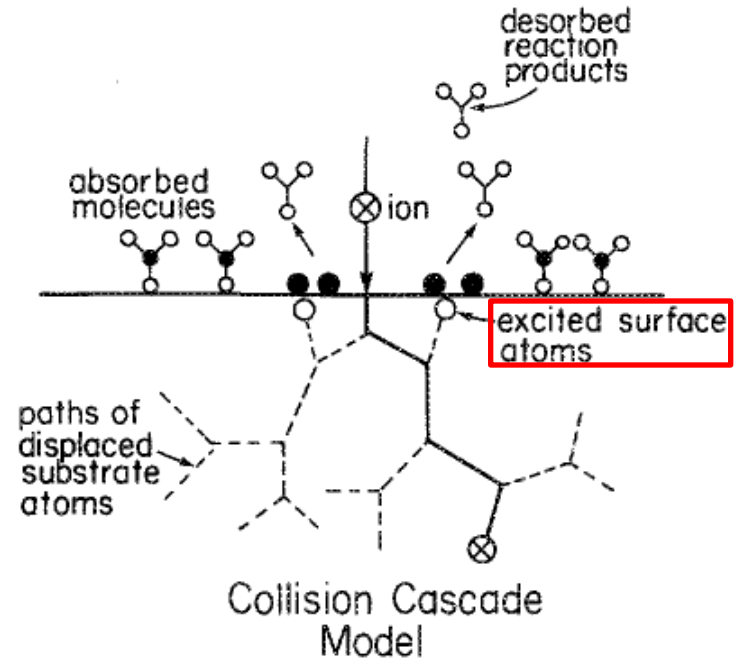


FIG. 4. Schematic of the collision cascade process of surface adsorbate decomposition.

“Mechanism of ion beam induced deposition of gold”, Melngailis, JVST B, 1994.

FIB deposition: role of SE and primary ion

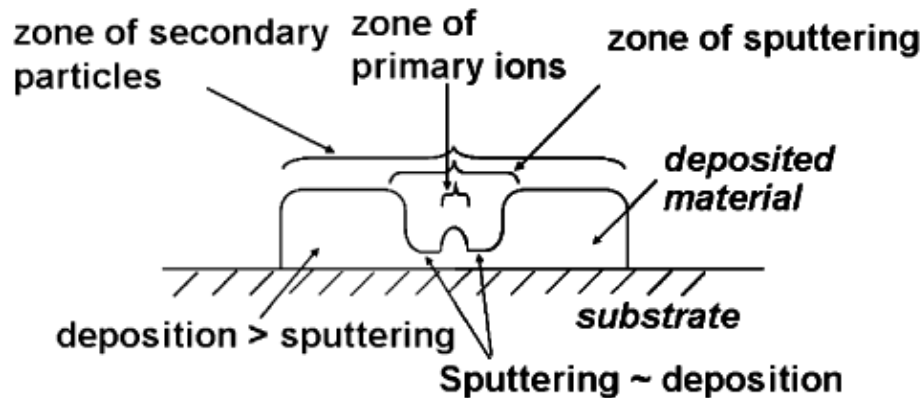


Fig. 5. Sketch of reaction zones of different mechanisms involved in IBID.

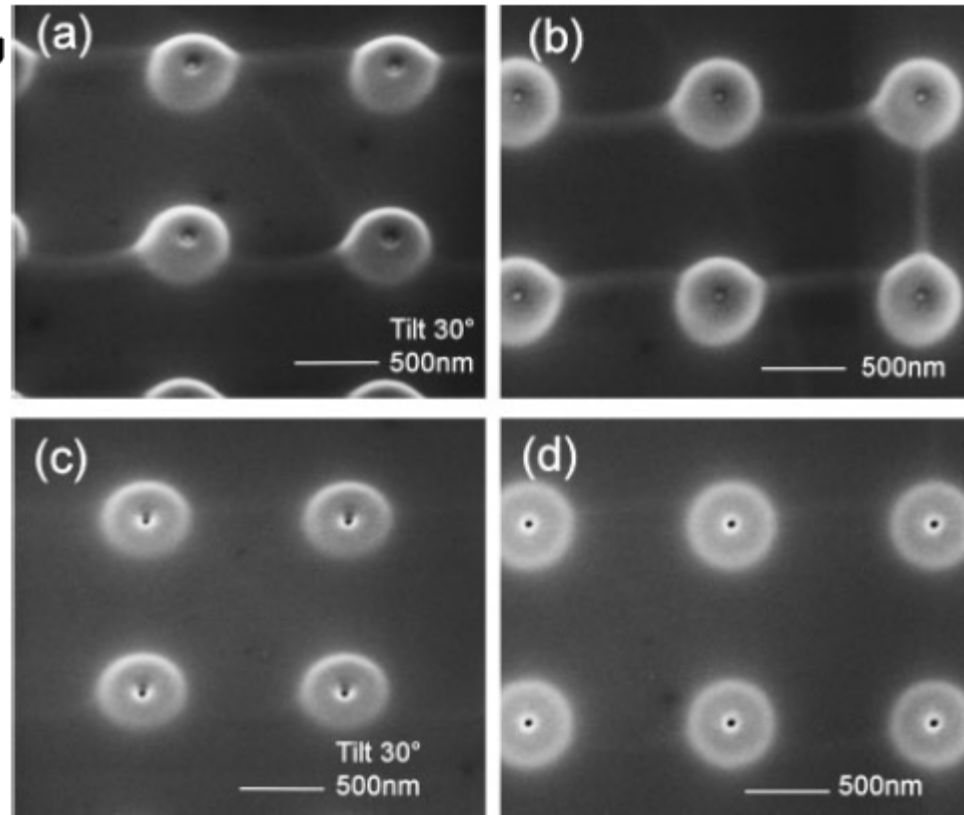


Fig. 4. SEM images of doughnut-like structures grown at spot mode with 15pA ion beam current (a,b) with a central tip (dwell time 0.1ms, exposure time 20s per spot); (c,d) with a central nanohole (dwell time 1ms, exposure time 20s).

30keV Ga⁺ FIB using (CH₃)₃Pt(CpCH₃),
beam size 13nm

- Though the yield for primary ion induced deposition is low, its contribution is not negligible due to high ion flux (central tip).
- SE is most important, causing deposition well beyond beam size.
- Next to the beam, both sputtering and deposition (induced by secondary/ sputtered atoms) are important.
- At longer dwell time (1ms), gas is depleted at the beam center and net sputtering occurs, leading to a central nano-hole.

“The Complex Mechanisms of Ion-Beam-Induced Deposition”, Chen, JJAP, 2008

Effect of ion species and energy on yield and purity

TABLE I. Effect of ion species and energies on the decomposition yield and film purity. Decomposition yield was calculated by summing a measured net deposition yield and a measured sputtering yield. Films were deposited at 1.0 mTorr and room temperature. For comparison the measured sputter yield of pure gold is also included in parentheses.

Ion	Energy (keV)	J current density ($\mu\text{A}/\text{cm}^2$)	Purity (%Au)	Y_N (Au atoms per ion)	Y_S (Au atoms per ion)	Y_S (Pure Au)	Y_D (Au atoms per ion)
Ne	50	2.6		2.3	1.7	(4)	4.0
	100	4.2	40%	2.2	1.2	(3.5)	3.4
Ar	50	3.4		5.5	3.1	(8.8)	8.6
	100	5.0	45%	4.7	4.2	(9.2)	8.9
Kr	50	3.0		6.2	18.0	(29)	24.2
	100	5.5	80%	7.0	18.7	(35)	25.7
Xe	50	3.6		7.7	19.6	(45)	27.3
	100	5.4	80%	10.6	21.3	(52)	31.9

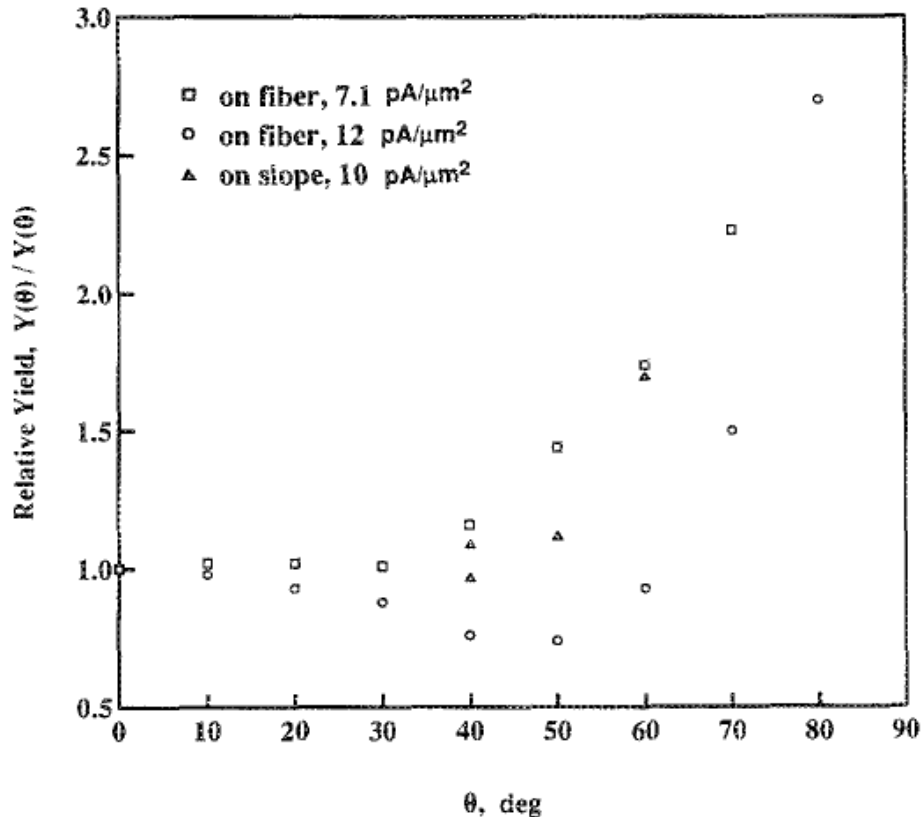
$$Y_N = Y_D - Y_S$$

Y_N =net deposition, Y_D =deposition, Y_S =sputtering

Heavier ion, higher yield.
Lighter ion, lower purity, would be worse for e-beam induced deposition.

Melngailis, "Mechanism of ion beam induced deposition of gold", JVST B, 1994.

Effect of ion incident angle



According to collision cascade model, deposition yield should be closely related to sputter yield, as atoms come out of the surface (sputtered away) or excited at the surface (not sputtered away) would induce decomposition.

It was found that deposition yield does increase with angle of incidence, but not as fast as milling/sputtering yield.

FIG. 6. The relative deposition yield vs angle of incidence for deposition from $\text{W}(\text{CO})_6$. The current density values quoted are the average ion current density that would be incident on a sample normal to the ion beam. Total beam current was 280 pA. The data for $\theta > 45^\circ$ can only be taken as the upper limit because of the rippling effect.

“Focused ion beam induced deposition and ion milling as a function of angle of ion incidence”, JVST B, 1992.

Deposition parameters

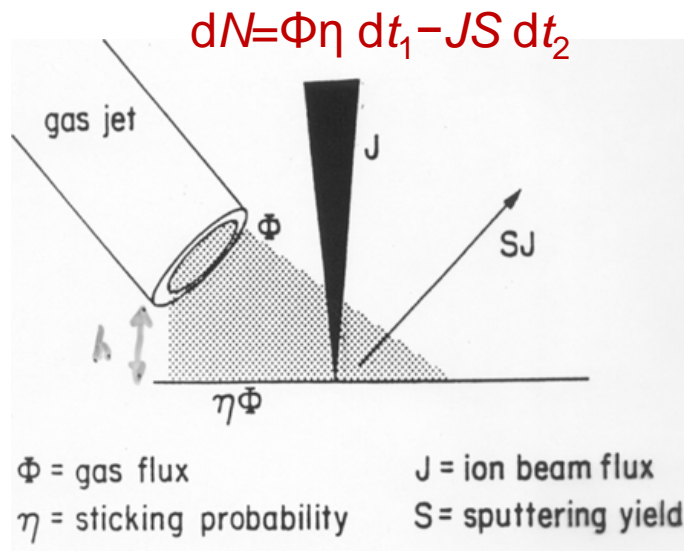
Dwell time: residence time at one point, desire one monolayer deposition per dwell time. About $0.4\mu\text{s}$ for C; $0.2\mu\text{s}$ for Pt.

Refresh time: time between successive dwells at same point

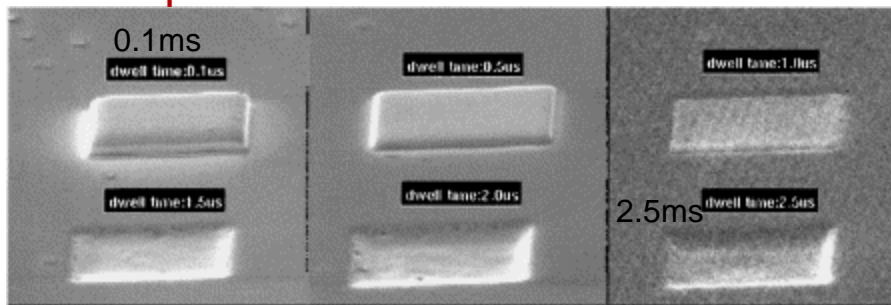
Overlap: amount two consecutive points overlap to obtain uniform deposition. typical 25% (positive overlap) to -100% (negative overlap)

Ion current: $\text{area} \times 5\text{pA}/\mu\text{m}^2$

Competition between deposition and sputtering



Net deposition



Net milling

Fig. 3. Deposited tungsten by FIB with different dwell times from 0.1 to 2.5 ms, and the same ion dose $6nC/\mu m^2$, refresh time $1000\mu s$, X and Y step size 71 nm and 68 nm, beam spot size 25 nm
 ECE 695 Nanometer Scale Patterning and Processing

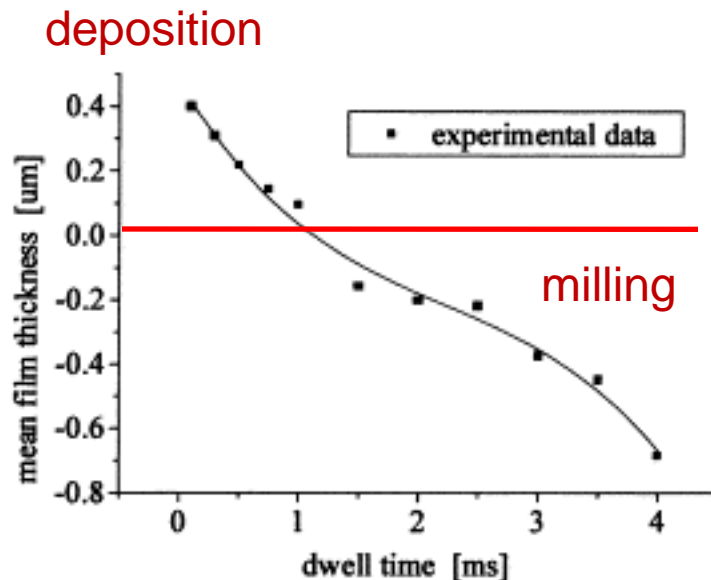
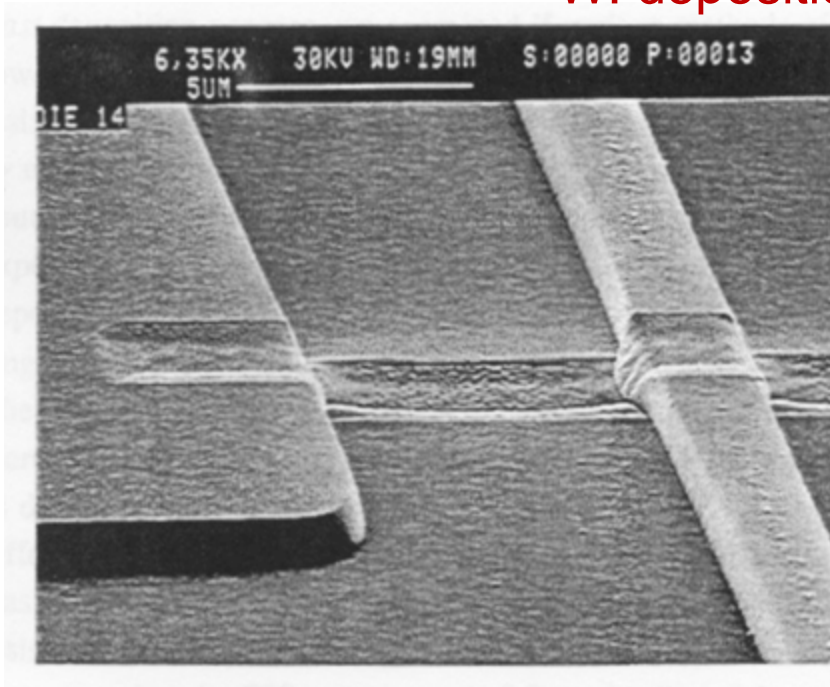


Fig. 4. Dwell time vs mean film thickness under ion dose $3nC/\mu m^2$, spot size 25 nm, and current 207 pA.

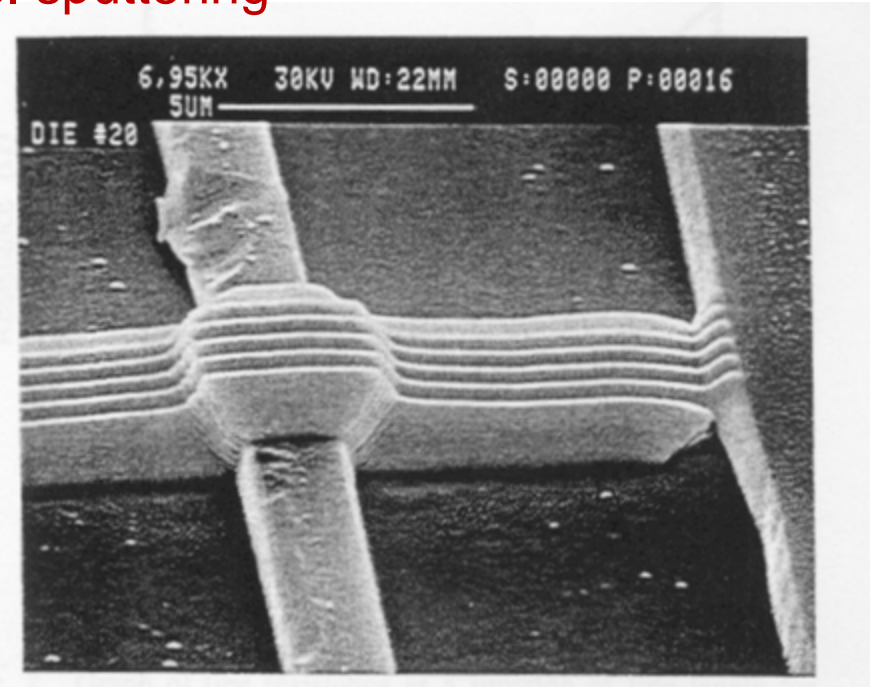
Fu, "Characterization of focused ion beam induced deposition process and parameters calibration", Sensors and Actuators A, 2001.

Competition between deposition and sputtering

W: deposition vs. sputtering



W deposited with 15 μ s dwell and 0.16 μ m step size, sputtering exceeds deposition.



W deposited with 0.3 μ s dwell and 0.6 μ m step size

J. Mohr and R. Oviedo, 19th ISTFA Proceedings, Nov. 1993, p.391

Deposition limited by gas diffusion and absorption

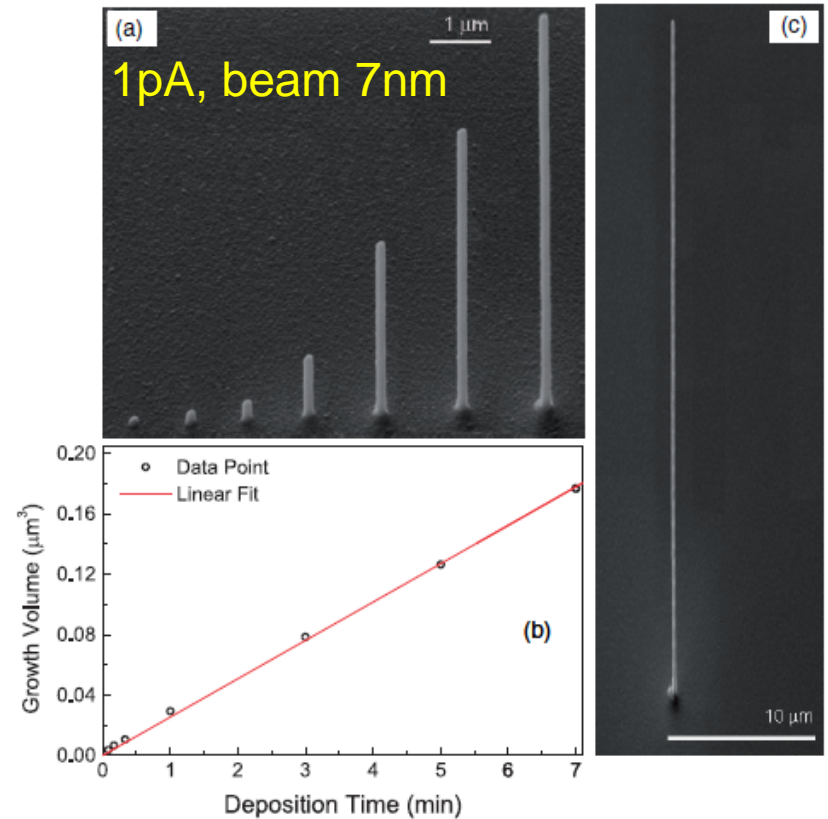
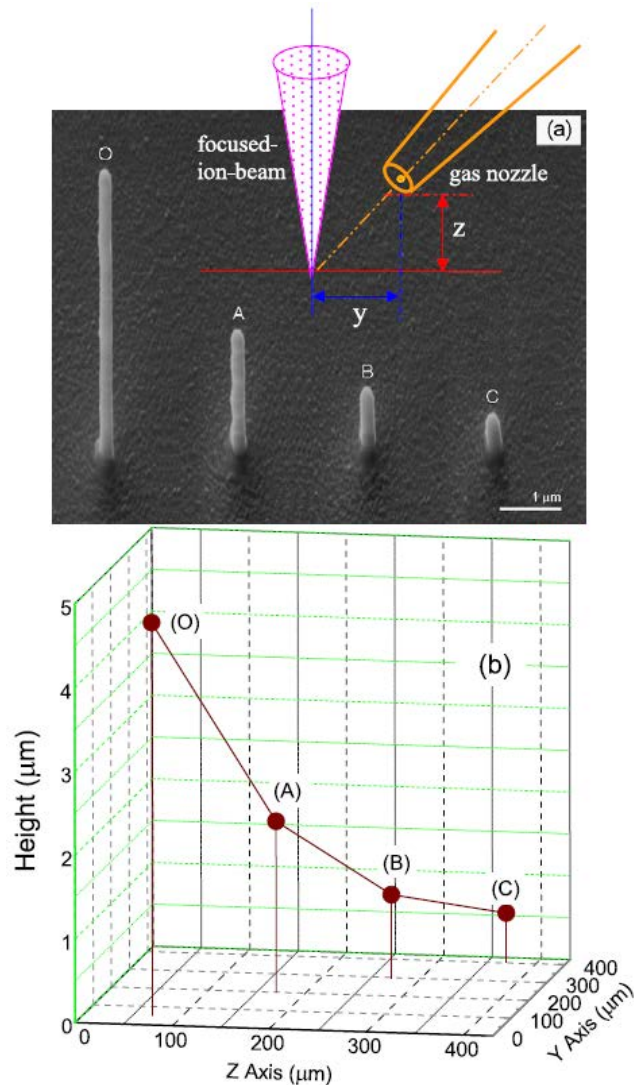
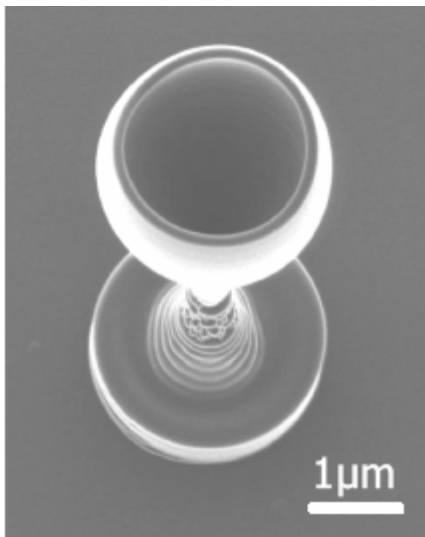
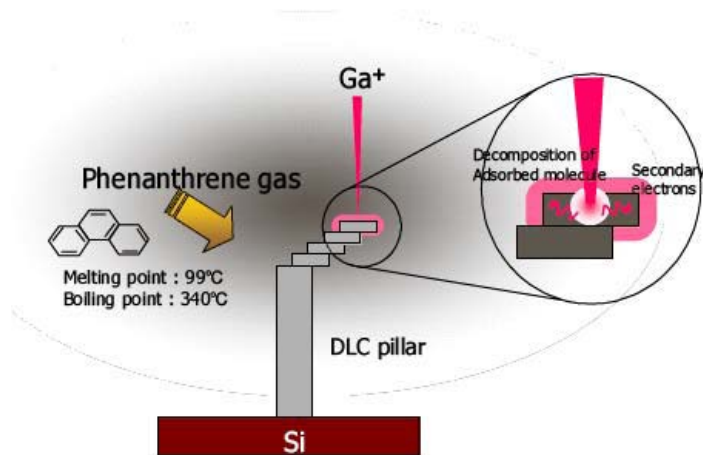


Figure 3. Nanorods deposited under optimized conditions with target cross-sectional size of $40 \times 40 \text{ nm}^2$: (a) SEM images of rods deposited for 5, 10, 20, 60, 180, 300 and 420 s (from left to right); (b) growth volume as a function of deposition time; (c) a very tall nanorod with a height up to $45 \mu\text{m}$. SEM images were taken with a viewing angle of 54° .

Effect of the gas precursor nozzle position on nano-rod growth. Limited by diffusion.

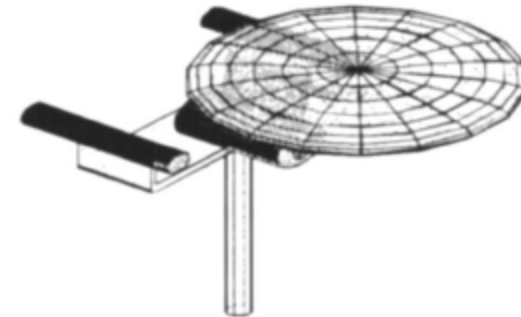
NOT limited by gas diffusion under optimal conditions.

3D structure by FIB deposition

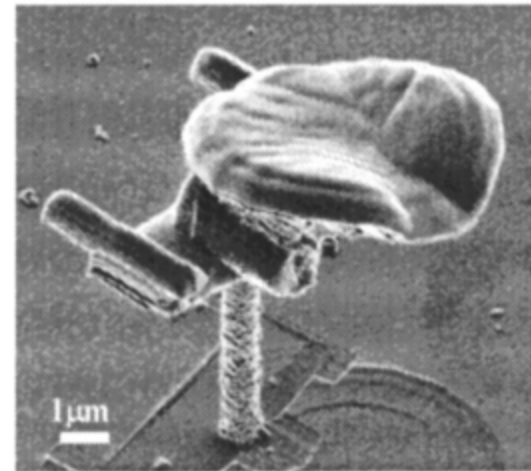


Diamond-Like Carbon Wineglass

Fujii et al, MRS Symp. Proc. 0983-LL08-08 (2007)



(a) 3-D CAD model



(b) SIM image (tilt 45deg)

Hoshino et al, *J. Vac. Sci. Technol. B*, 21 2732 (2003)

(order of pixels matters)