# Nanometer Scale Patterning and Processing

Spring 2016

## Lecture 27

## Non-Lithographic Applications of FIB (continued)

# Non-Lithographic Applications of FIB

1. Overview
2. FIB milling, sputtering yield.
3. Redeposition.
4. Single line milling.
5. Other types of FIB lithographies (implantation, intermixing…).
6. Gas-assisted FIB patterning.

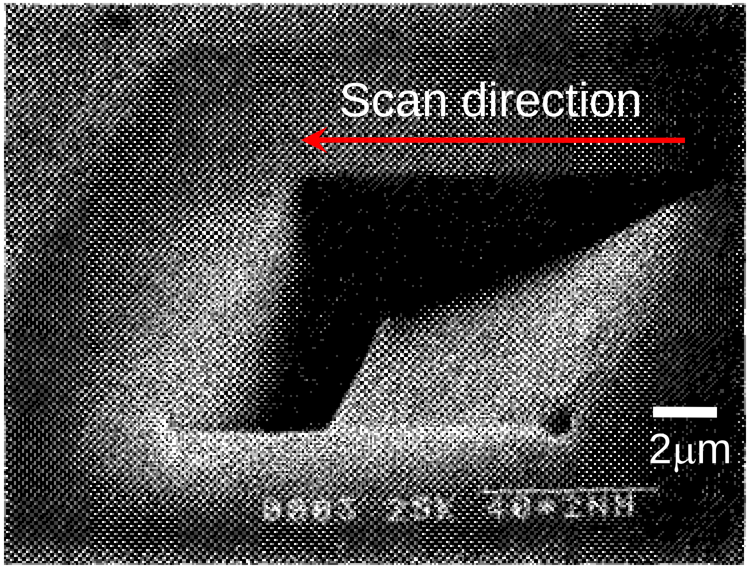
# Re-deposition

Redeposition depends on:

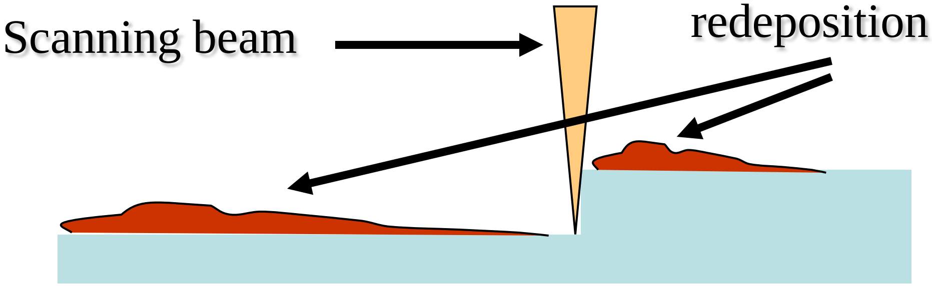
- Kinetic energy of atoms leaving surface
- Sticking coefficient of target
- Sputtering yield of target
- Geometry of feature being milled

Factors that increase sputtering rate tend to increase redeposition:

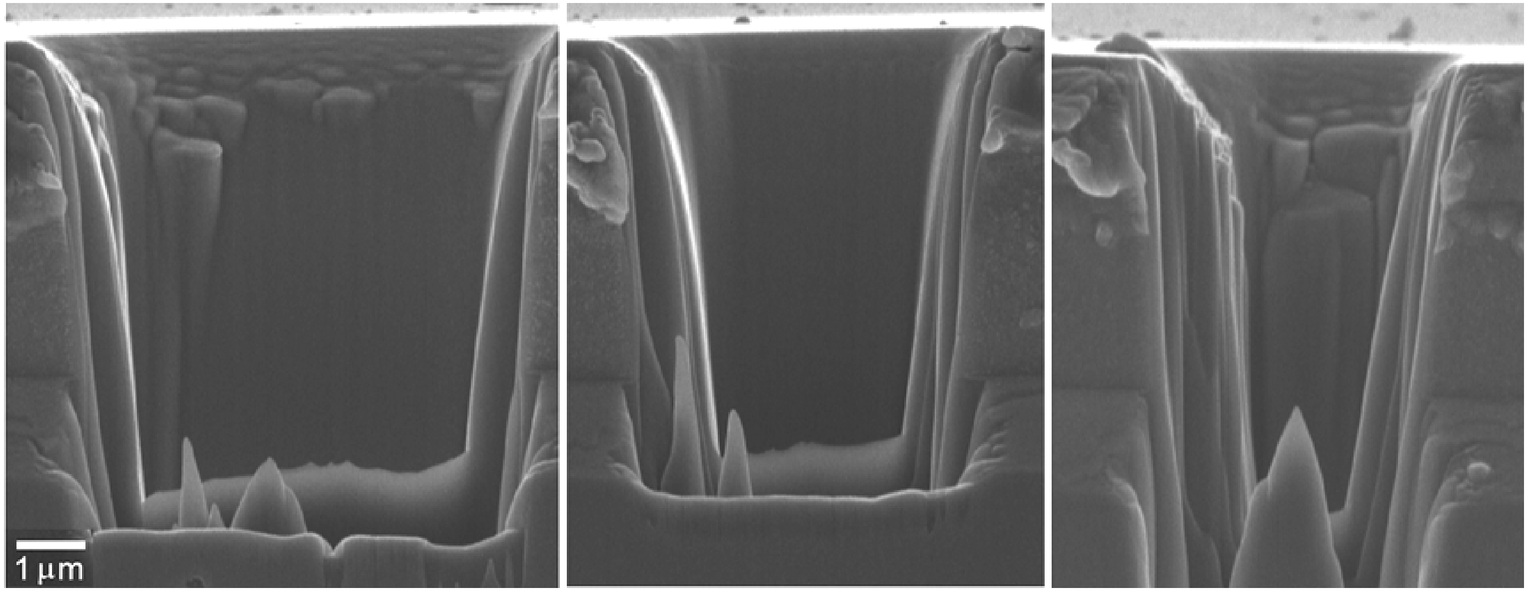
- Beam current
- Incident ion kinetic energy
- Incident ion attack angle
- Target material properties

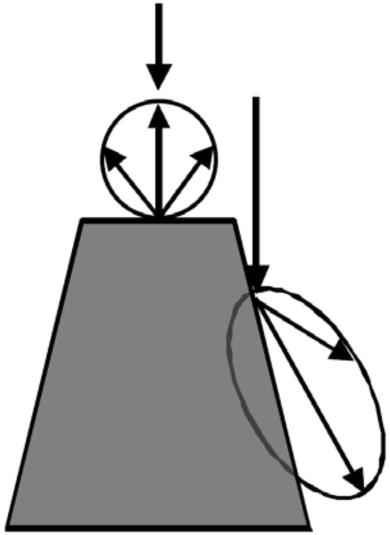


Si milled by 30keV $Ga^+$, slow *single* pass at 2s per line with 300 scan lines.



# Geometry effect of re-deposition



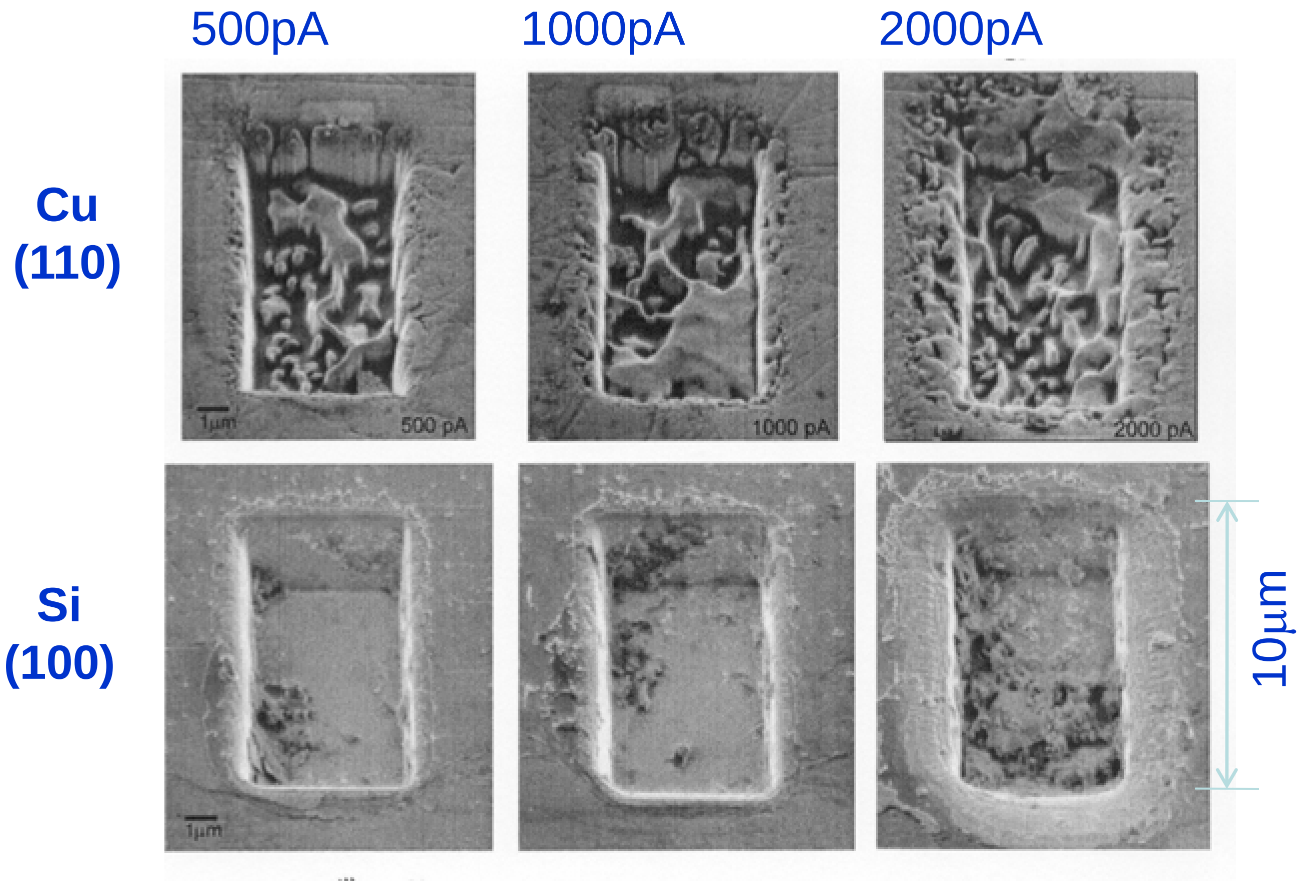


Constant dose variable area, milling of (100) Si at normal incidence with 1.5x10$^{11}$ Ga$^{+}$/µm$^{3}$.

Slope of sidewall increases (less steep) as trench width decreased, as sputtered atoms have less chance to get out of the trench.

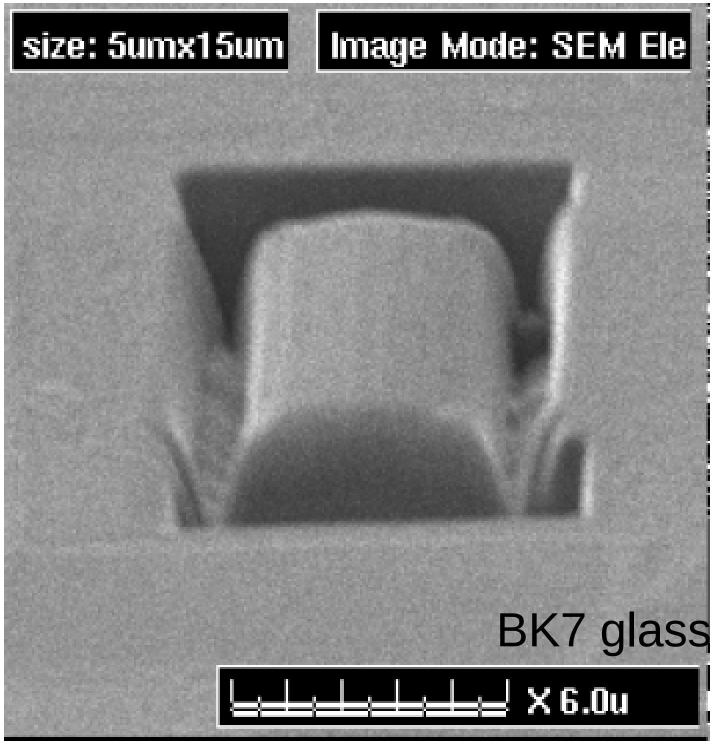
B. Prenitzer, Univ. Cent. Florida dissertation (1999)

# Effect of beam current on re-deposition

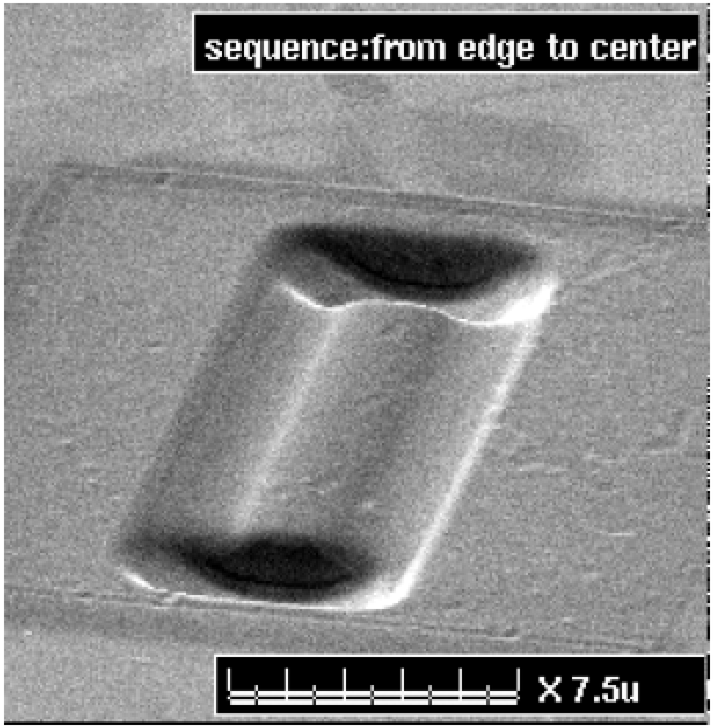


For higher beam current, re-deposition increase, sidewall definition worse. For Cu, rough surface at all beam currents.

# Effect of scan sequence



Milled from center to edge



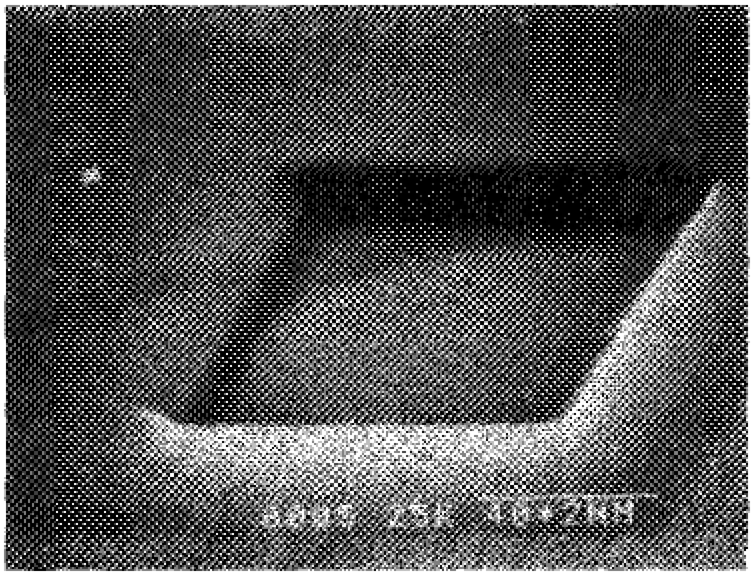
Milled from edge to center

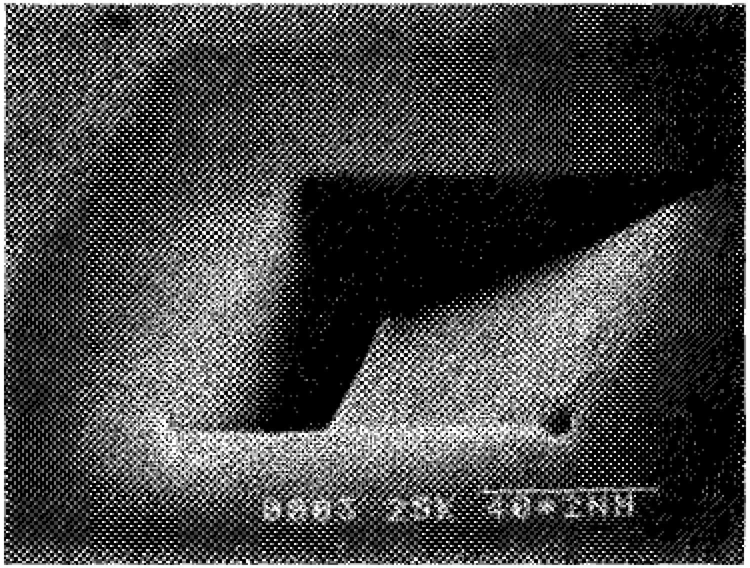
The earlier the region is milled, the more re-deposited material is accumulated there.

“Recent developments in micromilling using focused ion beam technology”, Tseng, 2004

# How to deal with re-deposition

Si milled by 30keV Ga$^+$, total dose 1.9×10$^{18}$ ions/cm$^2$.



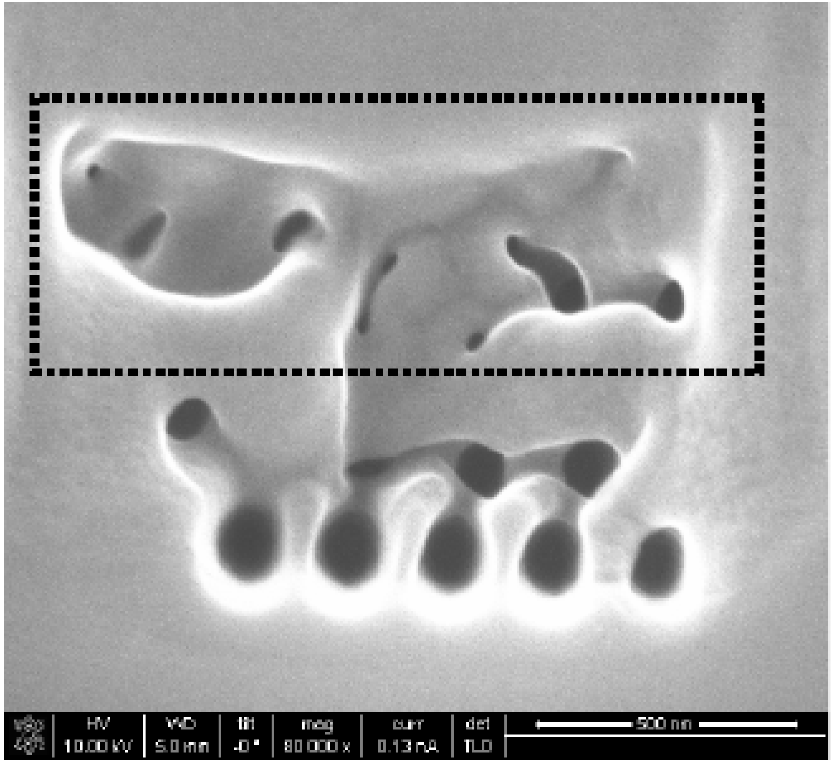


Fast 200 repetitive passes at 10ms per line with 300 scan lines.

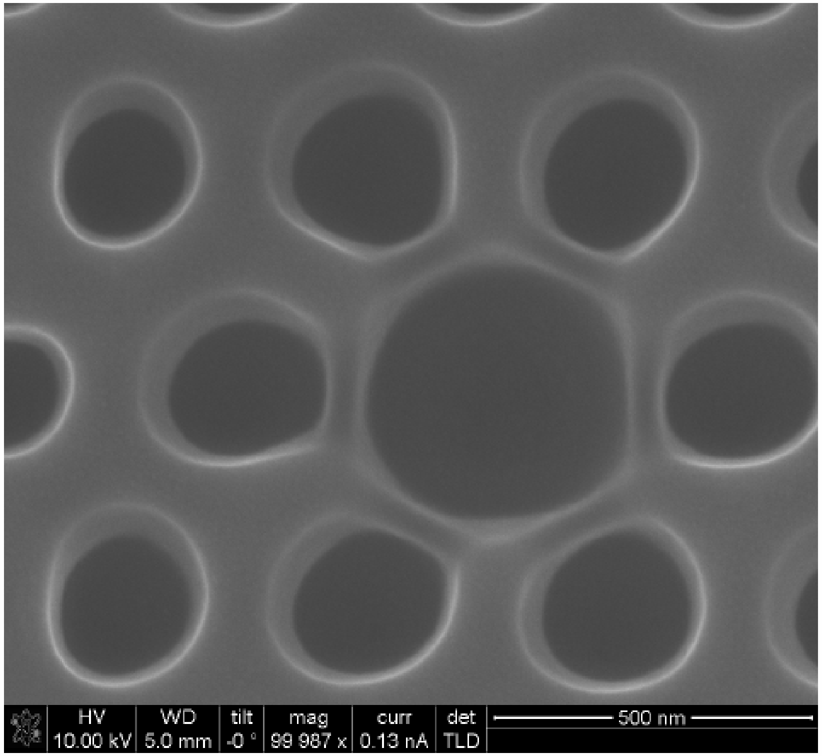
Slow *single* pass at 2s per line with 300 scan lines.

“Characteristics of Si removal by fine focused gallium ion beam”, Yamaguchi, JVST, 1985.

# How to deal with re-deposition, another example



Serial: mill each pixel all the way through before milling the next, re-deposited material covers holes milled previously.



Parallel: mill all pixels to a small depth, then return to mill deeper … until targeted depth achieved, re-deposited material milled away by following mills.

Summary for minimizing re-deposition effect:

- Mill large and less critical pattern first, using high beam current.
- Use multiple passes, not single pass.
- Mill fine and critical pattern last, using low beam current and multiple pass.

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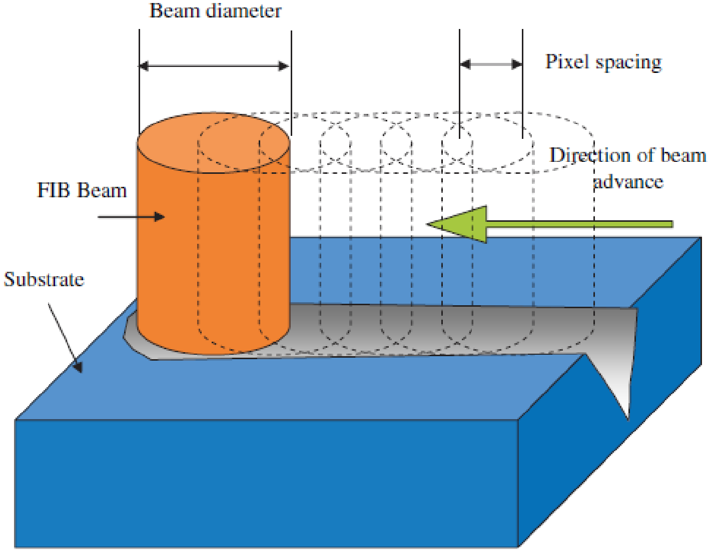
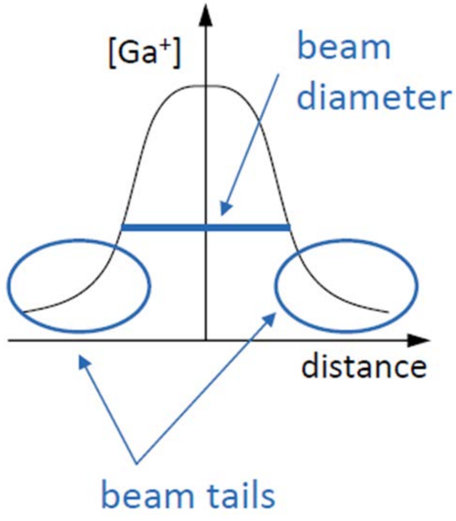
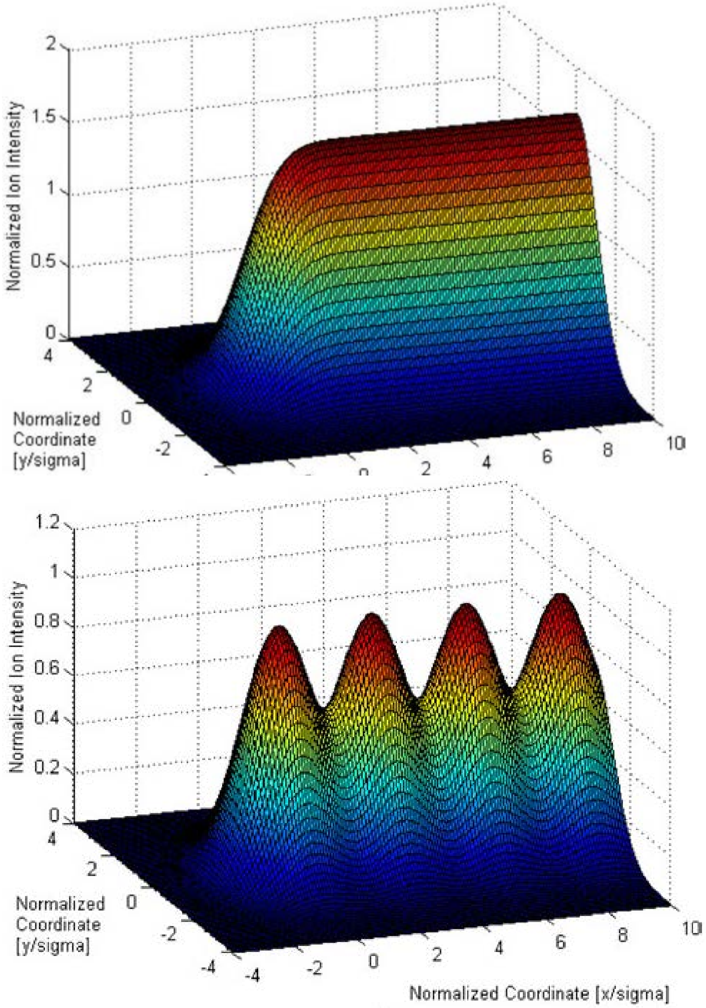
# Pixel size vs. beam size





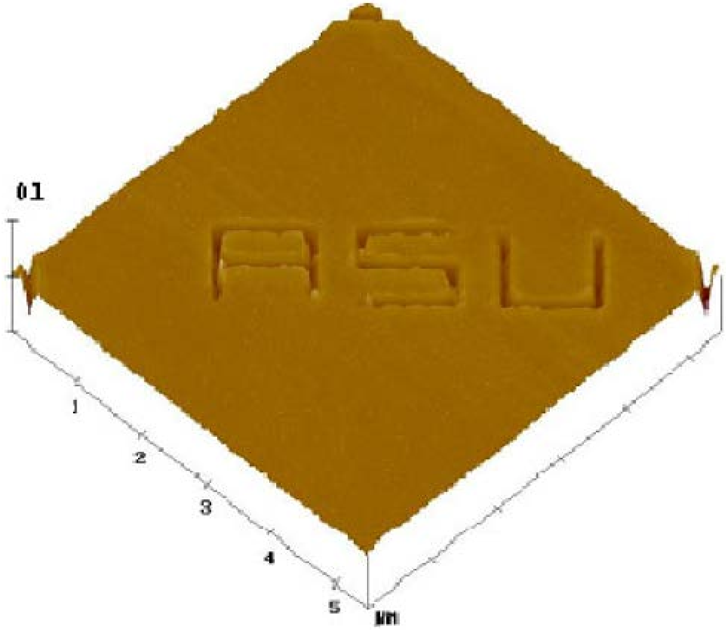


Ion flux distribution along a scan line with pixel/diameter=1.5 (top), 3.0(bottom)

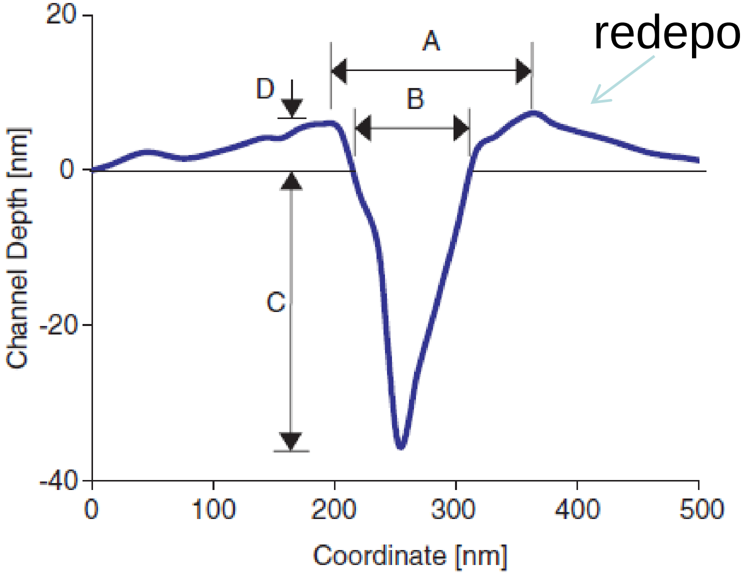
As a rule, for continuous non-wavy milling, pixel spacing/beam diameter ≥ 1.5

“Recent developments in micromilling using focused ion beam technology”, Tseng, 2004

# Channel milling using single pass



Channel profile by AFM

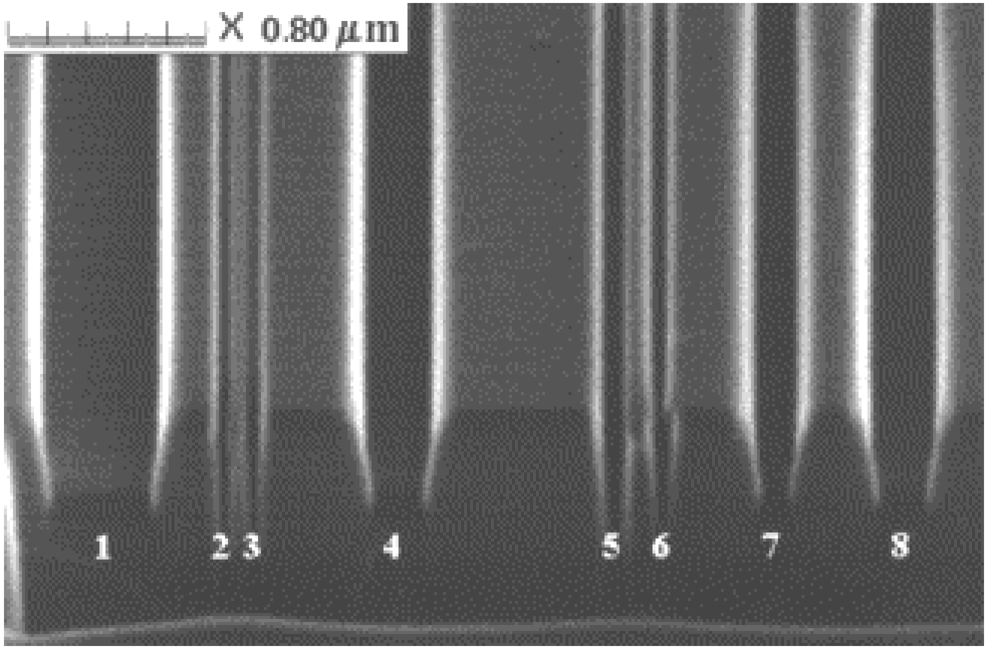


redeposition

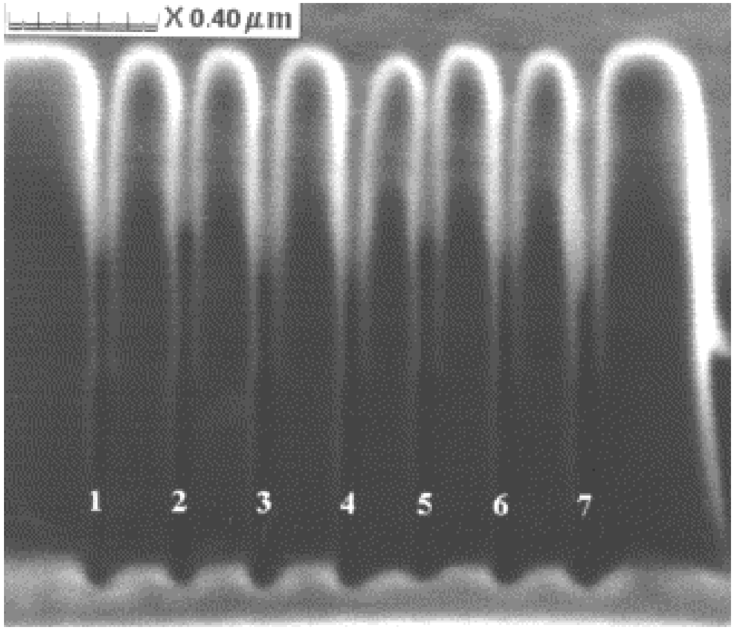
Milled into Au by 90keV As$^{2+}$ FIB with 5ms dwell time. I=5pA, pixel spacing 14.5nm, beam diameter 50nm. Single pass, aspect ratio ~1/3 and is insensitive to dwell time.

- Due to re-deposition, channel depth cannot be calculated simply by sputter yield.
- V-shaped channel profile is the inherent shape obtained by single-pass FIB milling.
- At long dwell time, the mouth width of the channel (B) can be one order larger than beam size, because the “tail” of the beam can mill sizable amount material.

# How to mill high aspect ratio narrow trench



FIB milled trenches in ta-C film using beam current (from left to right) of 32, 3, 3, 32, 7, 7, 32 and 32 pA, respectively.



Trenches with width 30–40 nm, aspect ratio up to 25, beam current 1.8-3pA

| | Trench No. | | | | | | |
|---|---|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 |
| Preset trench size (nm) | 10 | 10 | 20 | 30 | 6 | 10 | 20 |
| Milled trench size (nm) | 30 | 30 | 35 | 40 | 30 | 30 | 40 |
| Ion dose (nC $\mu m^{-2}$) | 30 | 20 | 20 | 20 | 50 | 40 | 30 |
| Approx. trench depth (nm) | 660 | 540 | 680 | 760 | 570 | 800 | 870 |

For high aspect ratio, use small beam current and large number of repetitive passes.

“Focused ion beam patterning of diamond-like carbon films “, Stanishevsky, 1999.

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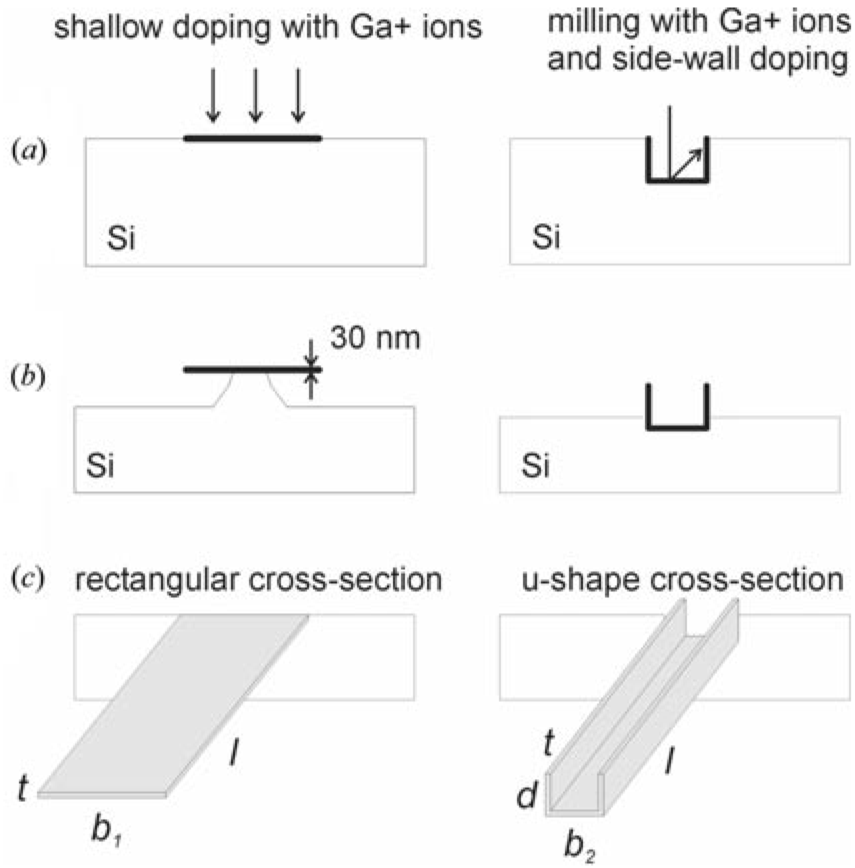
# Patterning etching mask by ion implantation

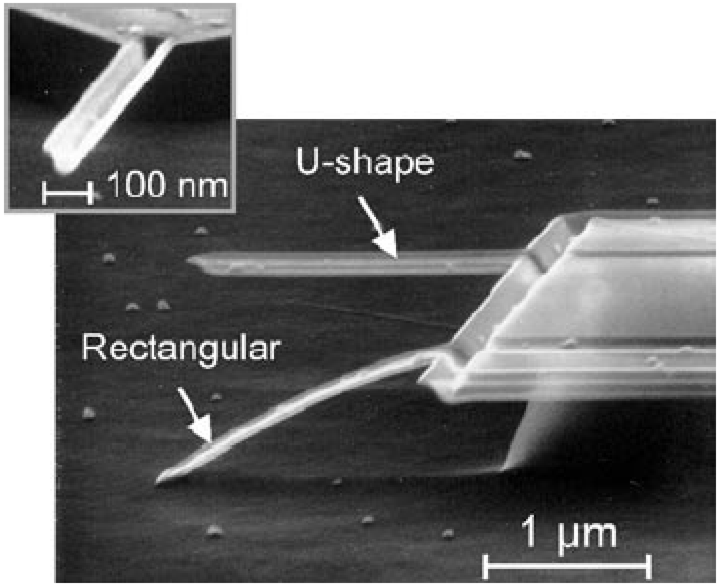


Fabrication of cantilevers by shallow doping (left-hand side) and milling and sidewall doping (right-hand side): (a) FIB exposure (milling and/or implantation), (b) during KOH etching and (c) after etching is completed.

- High concentration p$^+$ doping in Si drastically reduces the etch rate by KOH.
- The implantation depth is roughly 1nm/keV (e.g. 30nm for 30keV Ga$^+$ ion).
- The critical dose for the etch mask to be effective is $\sim 1\times10^{15}$ ions/cm$^2$ (=160$\mu$C/cm$^2$=10 ions/nm$^2$).
- The implanted region is completely amorphous (amorphization dose is $1\times10^{14}$ ions/cm$^2$ ).
- At higher doses such as $1\times10^{16}$ ions/cm$^2$ , sputtering (milling) of sample surface becomes significant.

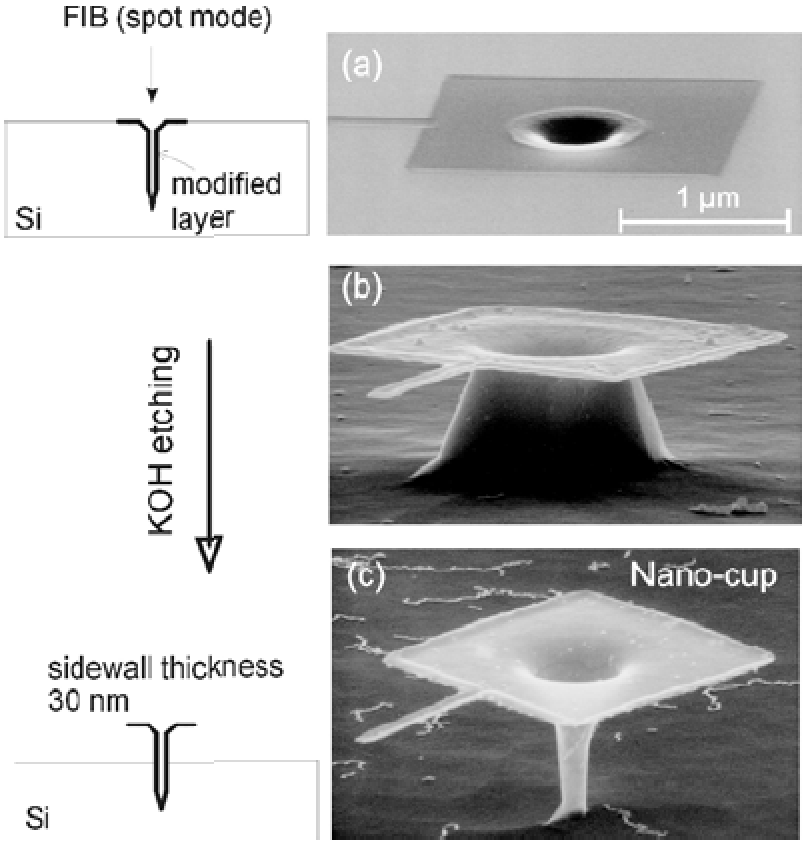
Reyntjens and Puers, “A review of focused ion beam applications in microsystem technology”, J. Micromech. Microeng. 11, 287-300 (2001).

# FIB implantation and pattern transfer: results



SEM photomicrographs of cantilevers (2 µm long, 100 nm wide and 30 nm thick).

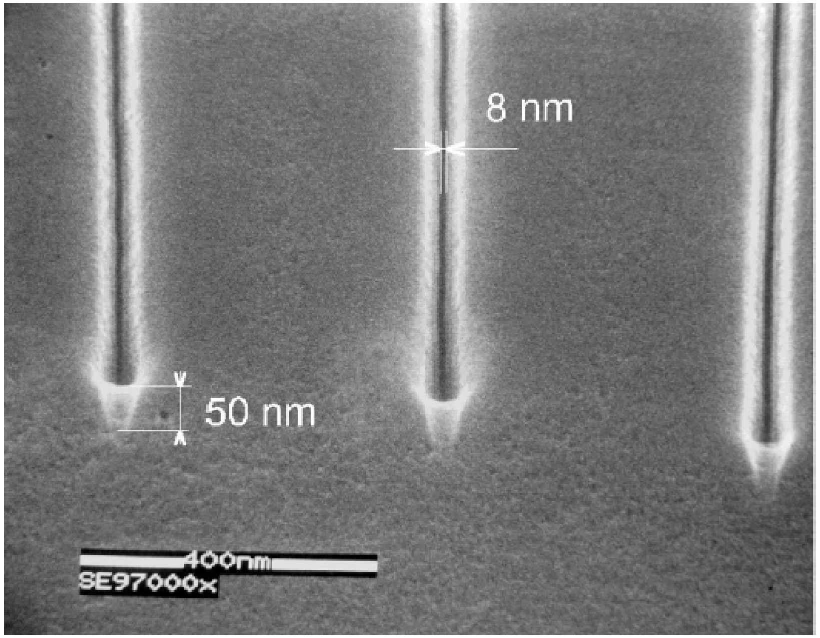
Nano-cup by extending vertical FIB milling to several $\mu$m.

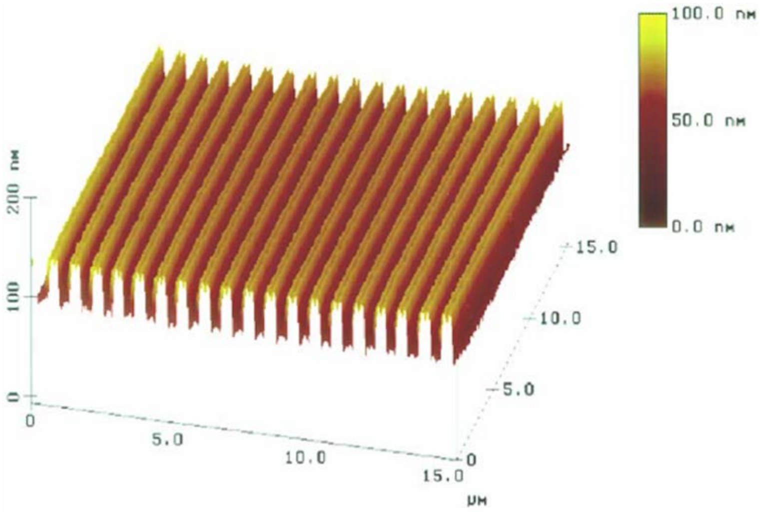


Tseng, “Recent developments in nanofabrication using focused ion beams”, Small, 1(10), 924-939 (2005).

# Ion beam lithography on $AlF_3$ resist

$AlF_3$: *inorganic* e-beam lithography resist.
Form $F_2$ gas upon exposure, leaving behind Al.





$AlF_3$ inorganic resist using 30kV Ga ion, large features, pitch ~1µm.

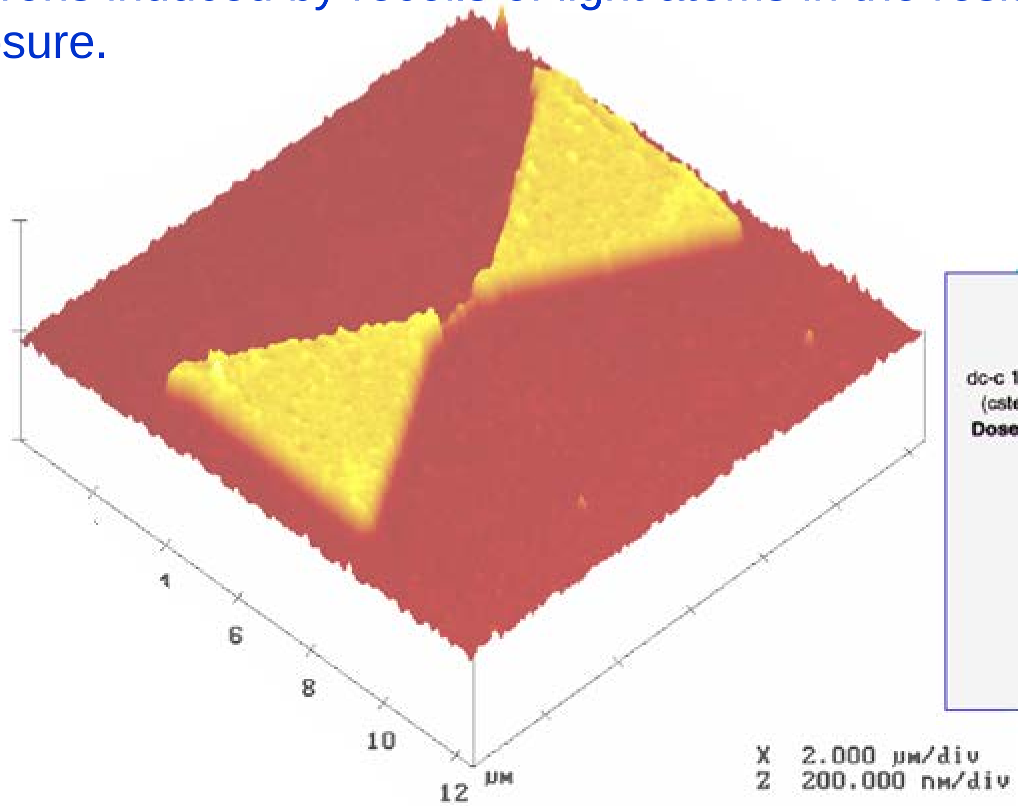
High-resolution direct machining: FIB etched lines on $AlF_3$(50nm)/GaAs, ion dose ~92nC/cm. The edges of the resist etched layer appear transparent and the lines in GaAs display a very reproducible width of ~8nm.

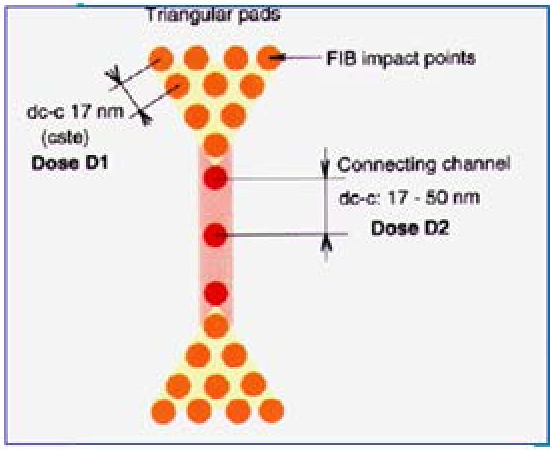
Explanation of this high resolution: flux-dependent vaporization of $AlF_3$, which is acting as a filter versus the tails of the ion-probe current distribution.

“Nano-fabrication with focused ion beams”, Microelectronic Engineering 57–58, 865–875 (2001)

# Ion beam lithography on $Au_{55}(PPh_3)_{12}Cl_6$ resist

$Au_{55}(PPh_3)_{12}Cl_6$: ion-sensitive negative resist, though recoil atoms and secondary electrons induced by recoils of light atoms in the resist also plays a role for resist exposure.

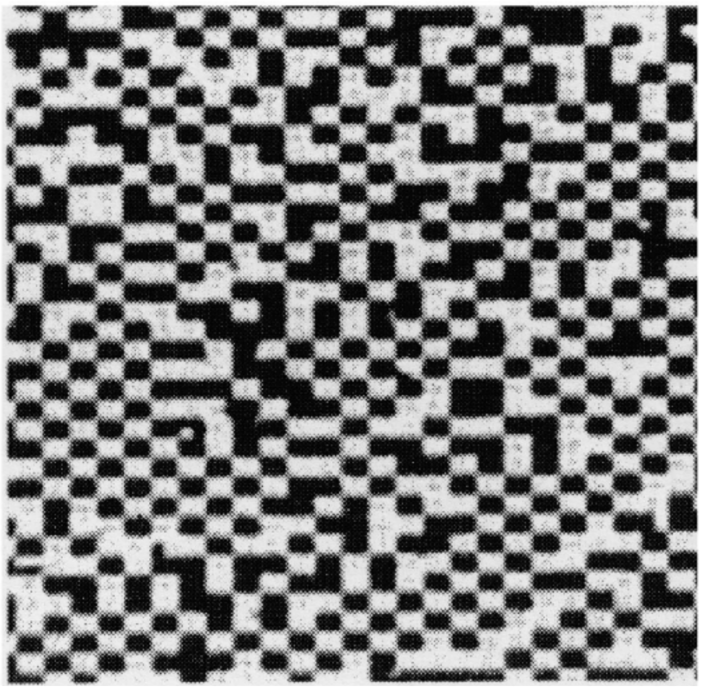




AFM image of a typical gold-containing butterfly-type pattern, realized by FIB irradiation of a single solid $Au_{55}(PPh_3)_{12}Cl_6$ resist layer. Dose on big pad is $10^{14}$ ions/cm$^2$.
The 3D connecting channel with granular morphology is clearly shown: width 30nm; height 20nm; length 1.5μm.
(Ga ions, energy 30keV, initial thickness for gold cluster compound 50nm)

"Nano-fabrication with focused ion beams", Microelectronic Engineering 57–58, 865–875 (2001)

# Ion beam induced intermixing

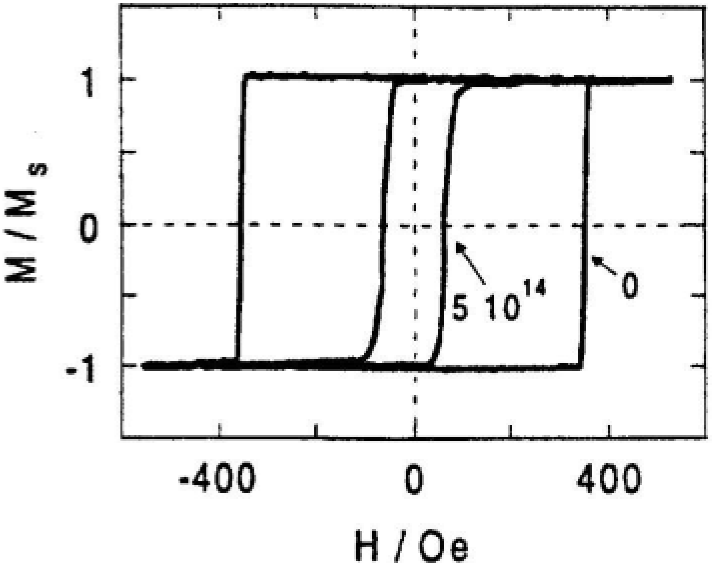


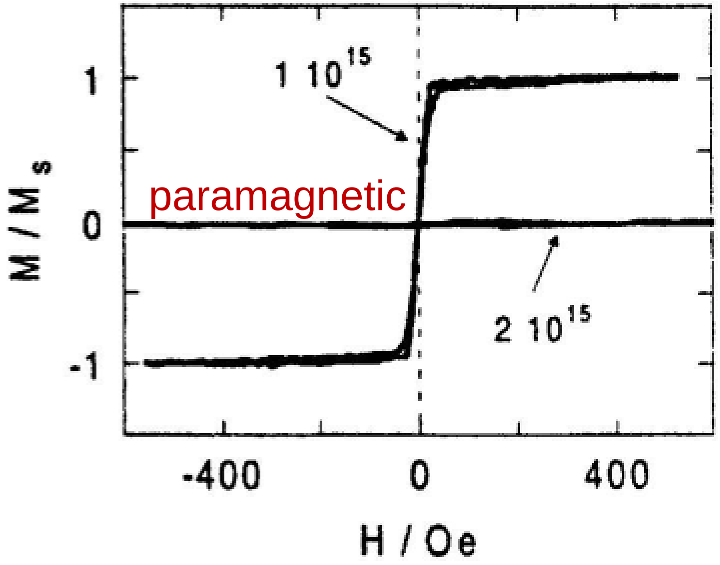
Faraday microscopy image of magnetic patterns of a FIB dot array (Ga ions, 28 keV).

(either bright or dark region is irradiated)

Initial film: Pt(3.4nm)/Co(1.4nm, ferromagnetic)/Pt(4.5nm).

Mechanism: ion-induced atom displacement at the Co/Pt interfaces (ion beam mixing effect).





Faraday ellipticity loops obtained at room temperature after uniform Ga irradiation. Irradiation reduces coercivity; at even higher dose $2\times10^{15}$, the film becomes paramagnetic.

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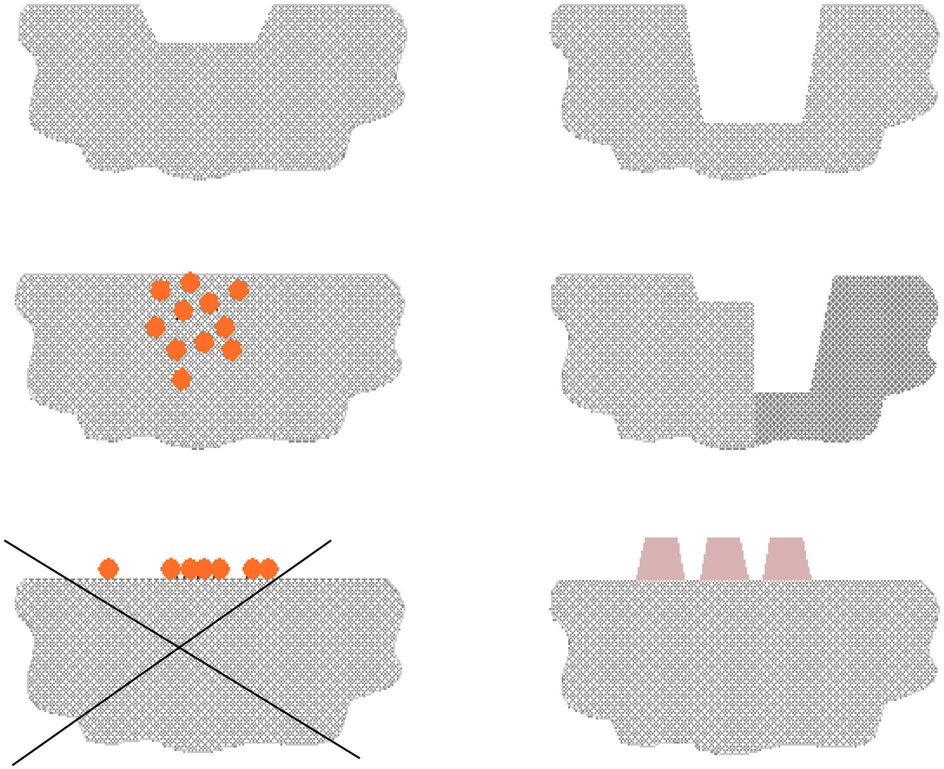
# Gas-assisted FIB lithography



FIB alone capabilities

FIB with gas

Ion Etching

Enhanced etching

Poor selective Etching

Selective etching

Ion deposition

Material deposition

# Gas-enhanced FIB milling (etching)

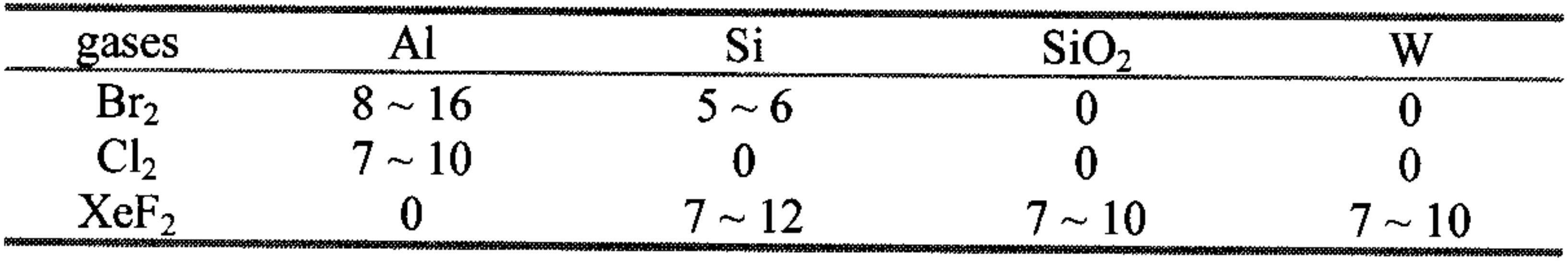
- Impinging $Ga^+$ knocks out atoms and ions from sample
- Redeposition is prevented by chemical reaction with the adsorbed gas - formation of volatile species.
- Etching gases that react only with certain species - selective milling
- Examples of etching gases

  $XeF_2$ enhances Si and insulator milling.

  $Cl_2$, $Br_2$, $I_2$ enhances metal milling.

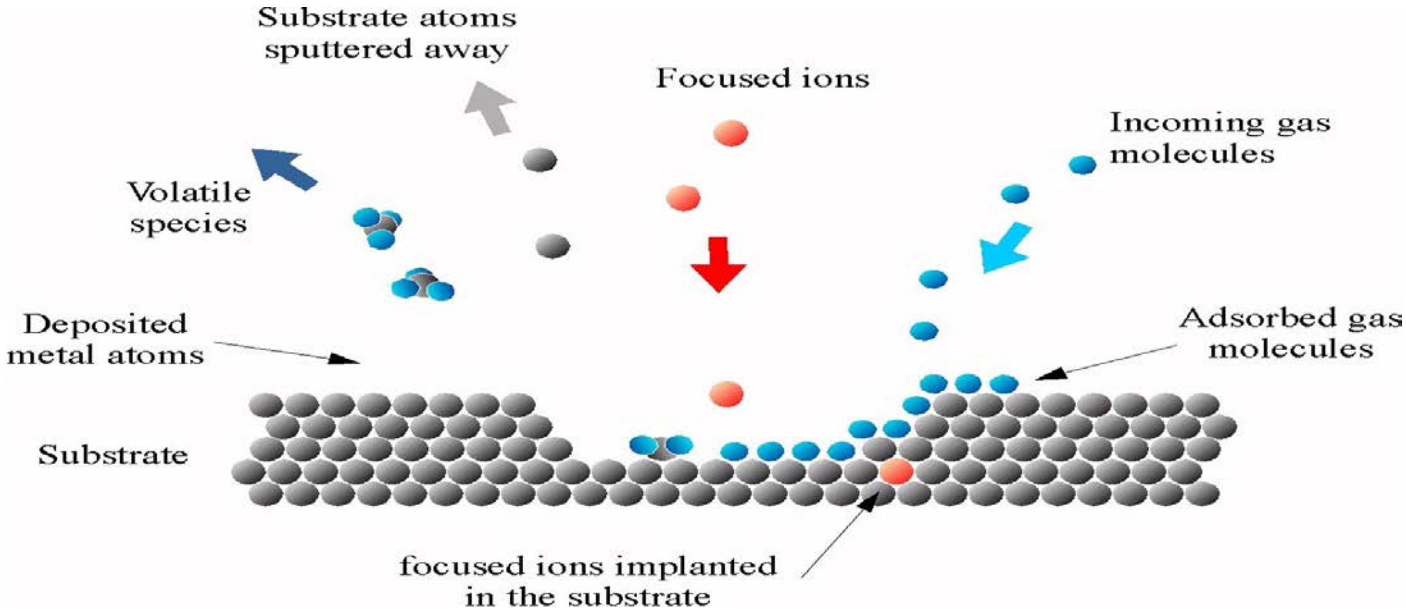
  $H_2O$ enhances carbon (polymers, ...) milling.

Sputtering yield enhancement factors



| gases | Al | Si | $SiO_2$ | W |
|---|---|---|---|---|
| $Br_2$ | 8 ~ 16 | 5 ~ 6 | 0 | 0 |
| $Cl_2$ | 7 ~ 10 | 0 | 0 | 0 |
| $XeF_2$ | 0 | 7 ~ 12 | 7 ~ 10 | 7 ~ 10 |

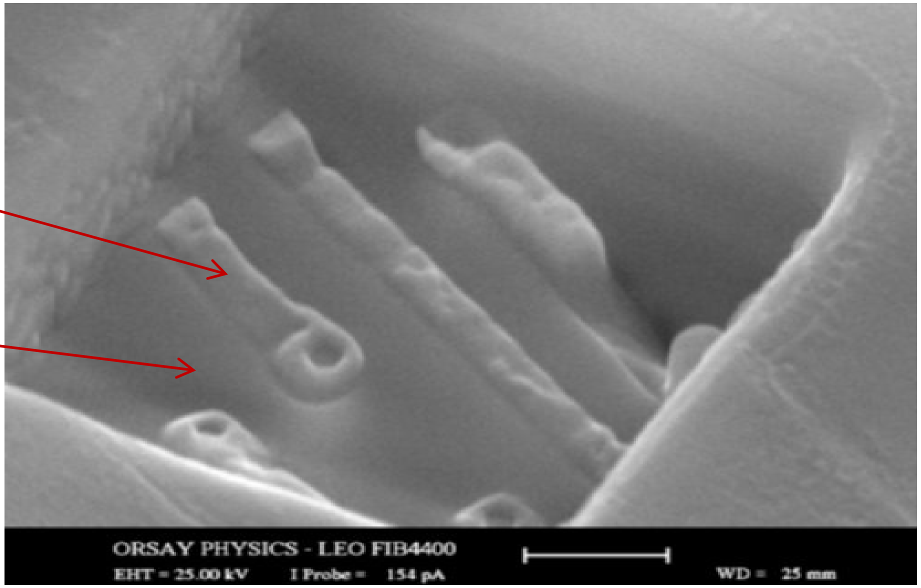
# Gas-enhanced FIB milling (etching)



- Adsorption of the gas molecules on the substrate
- Ion bombardment ionize the absorbed gas atoms, making them reactive.
- Interaction of the gas molecules with the substrate
    - o Formation of volatile species: $GaCl_3$, $SiCl_4$, $SiF_4$
    - o No chemical reaction
    - o Formation of non-volatile species: $AlF_3$, oxide layer
- Evaporation of volatile species and sputtering of non volatile species

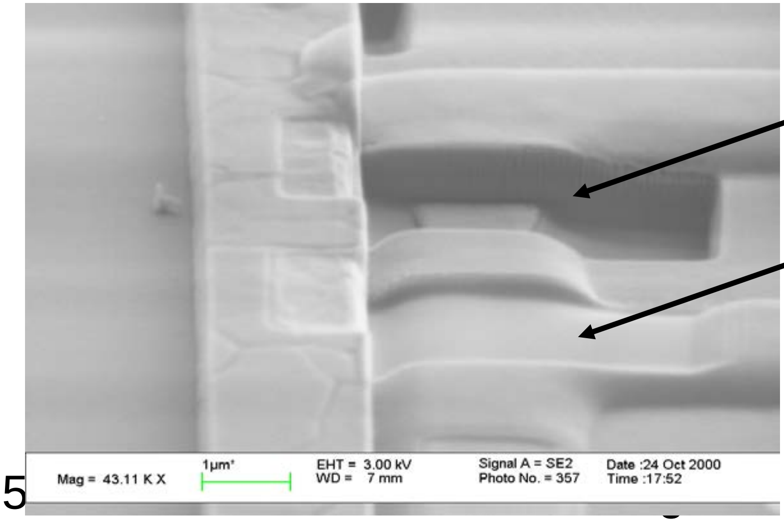
# Gas-assisted selective/enhanced FIB milling

Selective etching using



Al

SiO$_2$



With XeF$_2$

Without XeF$_2$

Enhanced etching using XeF$_2$

Orsay Physics