



Institut Matériaux Microélectronique Nanosciences Provence

La préparation des substrats de silicium pour la croissance de nanostructures SiGe

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Institut Matériaux Microélectronique
Nanosciences de Provence



**Linking fundamental
research to applications
within our fields of
expertise**

www.im2np.fr

Introduction

Development of new technologies: 2D, 1D, 0D and strain

IMEC LOGIC DEVICE ROADMAP DEVICE TECHNOLOGY FEATURES

Early production	2013 - 2014	2015 - 2016	2017 - 2018	2019 - ...			
	16 - 14nm	10nm	7nm	5nm			
Vdd (V)	0.8	0.8-0.7	0.7-0.5	0.7-0.5			
	Planar SOI	Bulk FinFET	SOI FinFET	SiGe/Ge channel	III-V channel	Lateral Nanowire	Vertical Nanowire
Device	FinFET (Bulk, SOI), FDSOI	FinFET (Bulk, SOI)	FinFET (GAA, QW, SOI)	GAA lateral NW; (Vert. NW)			

Improve Electrostatics

Channel n/p	Si / Si	Si / SiGe	Si / SiGe (Ge)	Si / SiGe (III-V / Ge)
S/D Strain	N S/D Si:P P S/D eSiGe (55%) Low-k spacer	N S/D Si:P:C P S/D eSiGe (>60%) Low-k spacer	N S/D Si:P:C P S/D eSiGe (>60%) Low-k spacer	TBD

Improve Performance

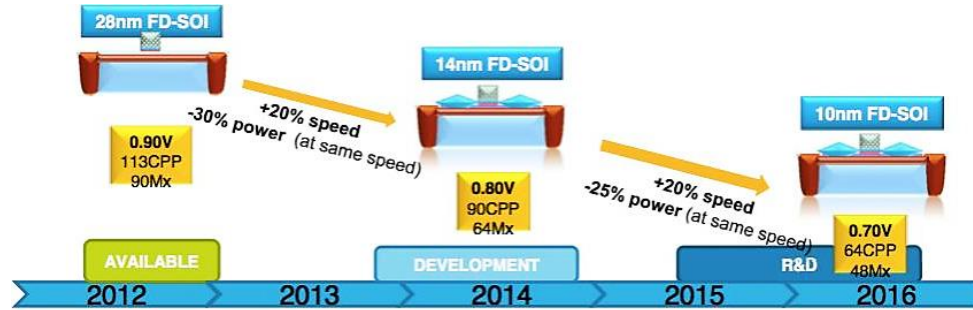
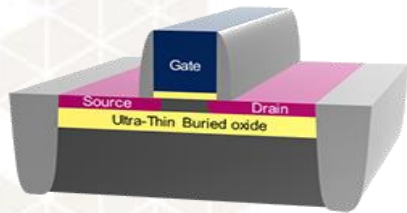
In new strategies: 3D structures and Strain engineering



Introduction

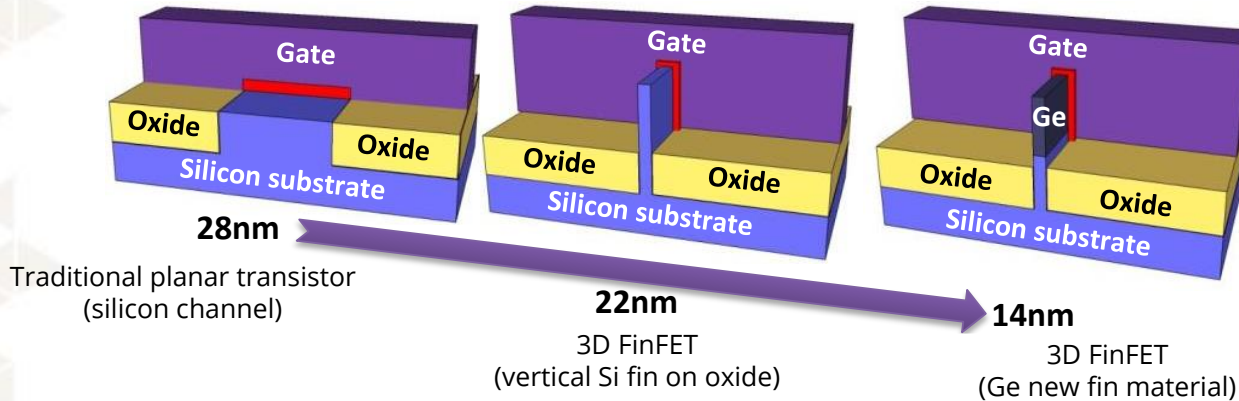
Development of new technologies: 2D, 1D, 0D and strain

STMICROELECTRONICS LOGIC DEVICE ROADMAP



FD-SOI

INTEL LOGIC DEVICE ROADMAP

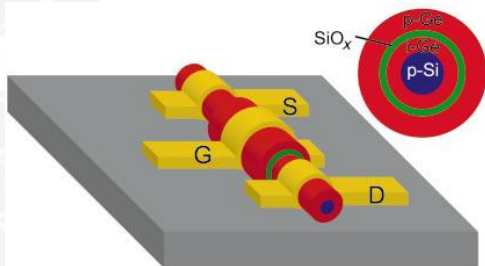
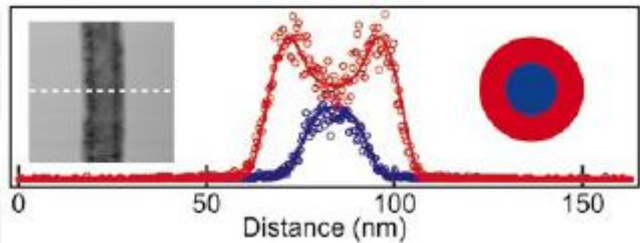


FIN-FET

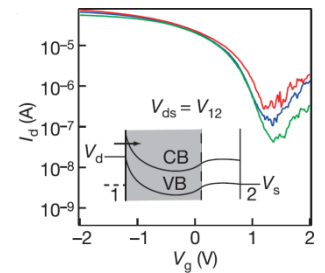
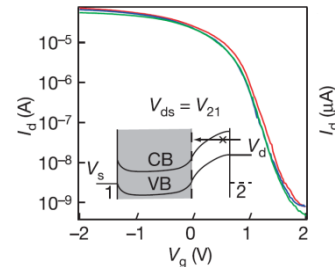
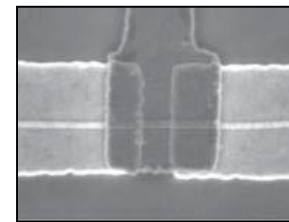
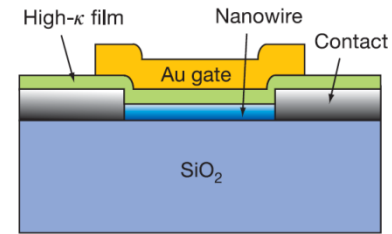
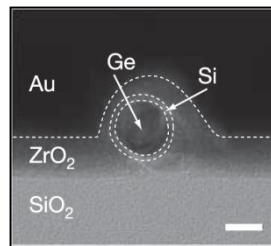
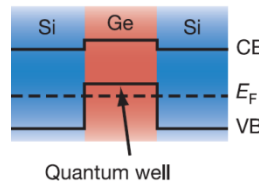
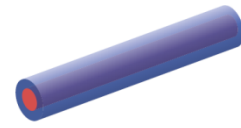
Introduction

Example: nanowires application

Synthetizing a single device



Lincoln J. Lauhon *et al.*, Nature (2002)



Jie Xiang *et al.*, Nature (2006)

- Ge/Si nanowires \Rightarrow one-dimensional hole-gas
- One-dimensional quantum confinement effects \Rightarrow reduced carrier scattering
- Enhanced gate coupling with high-k dielectrics give high-performance FETs
- Intrinsic switching delay comparable to similar length carbon nanotube FETs

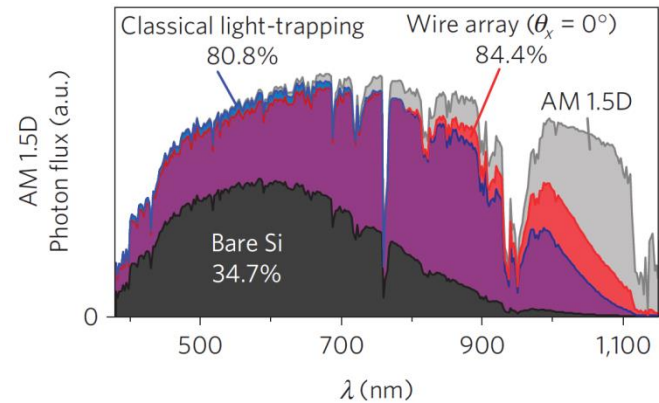
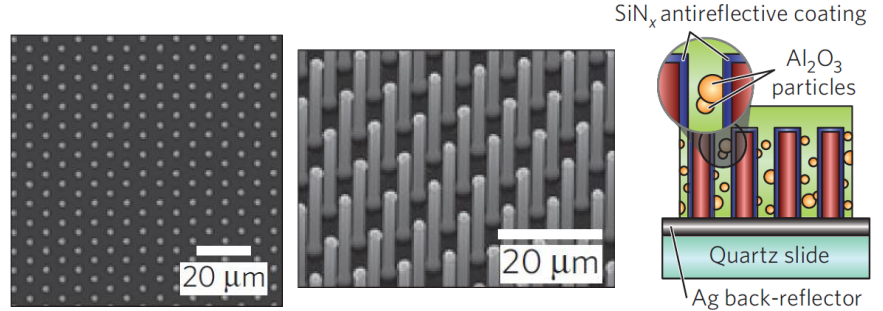
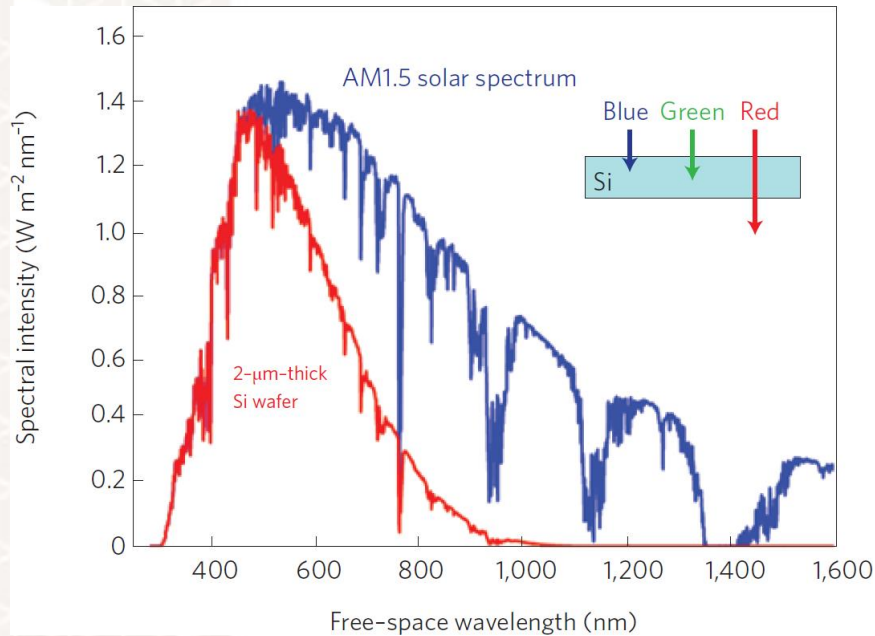
J.-N. Aqua, *et al.*, Phys. Rep., 522, 59 (2013)

Introduction

Example: nanowires application

Organizing multiple objects

Si cells absorption spectrum



Large unused spectrum range

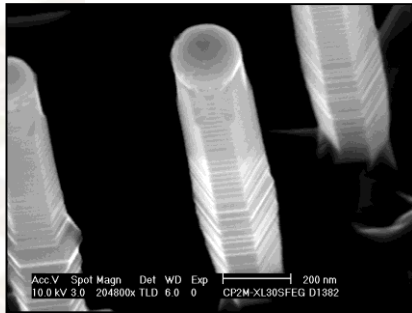
H. Atwater, A. Polman, Nat. Mater., 9, 10, 865 (2010)

Nanowires arrays enhance absorption

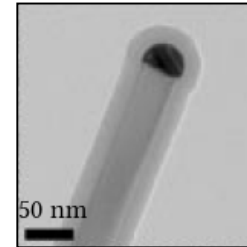
Kelzenberg *et al.*, Nat. Mater., 9, 3, 239 (2010)

Introduction

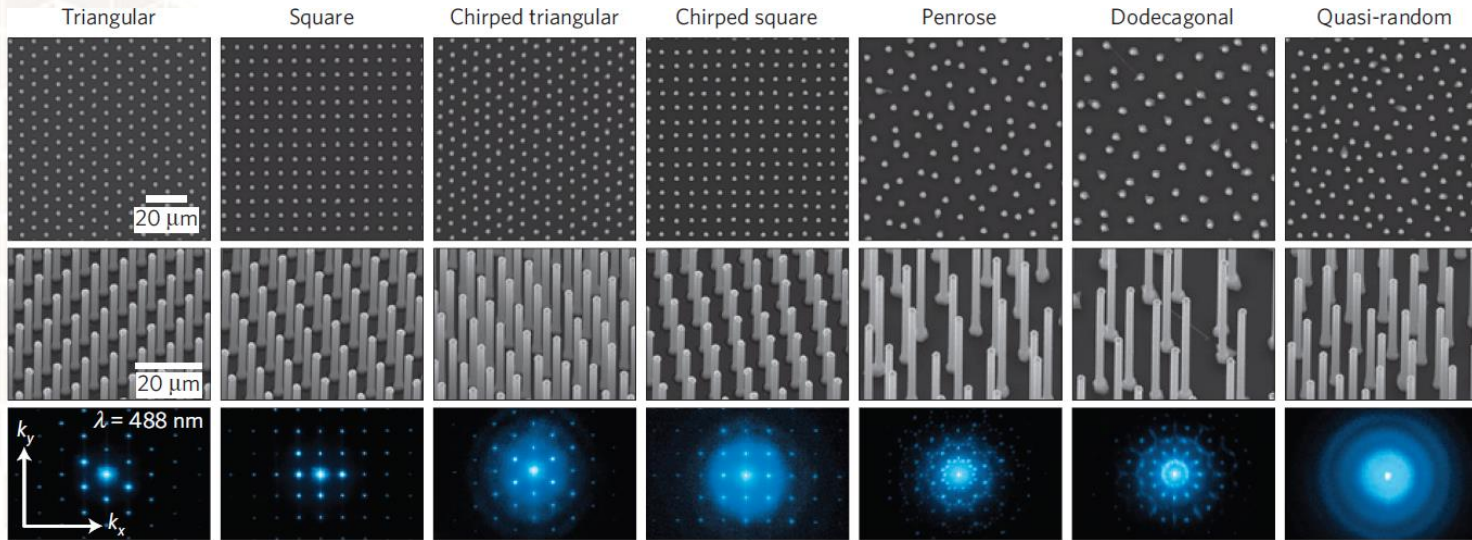
Example: nanowires organization



Metallic droplets
act as a catalyst
in NW growth



L. J. Lauhon *et al.*, *Nature*, **420**, 6911, 57 (2002)



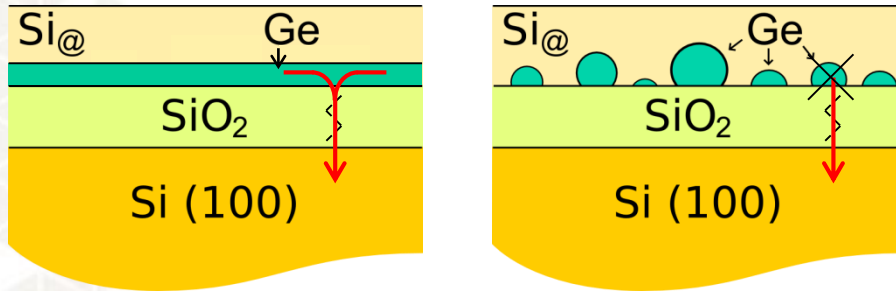
↓
Lithography:
for organisation

Kelzenberg *et al.*, *Nat. Mater.*, **9**, 3, 239 (2010)

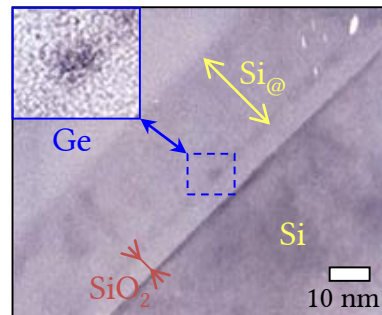
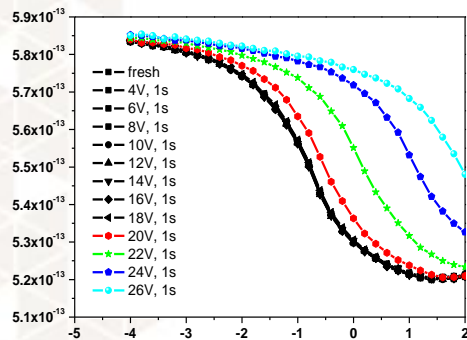
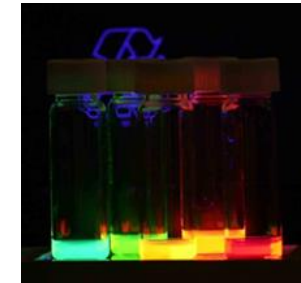
Introduction

Other examples: Quantum dots applications

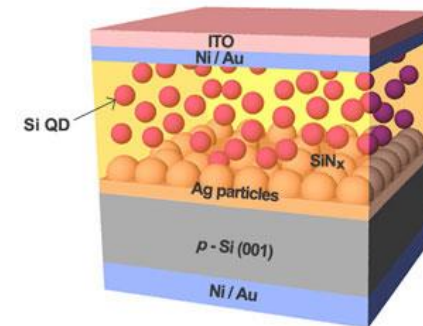
Floating gate memories



Quantum dots:
light-emitting diodes



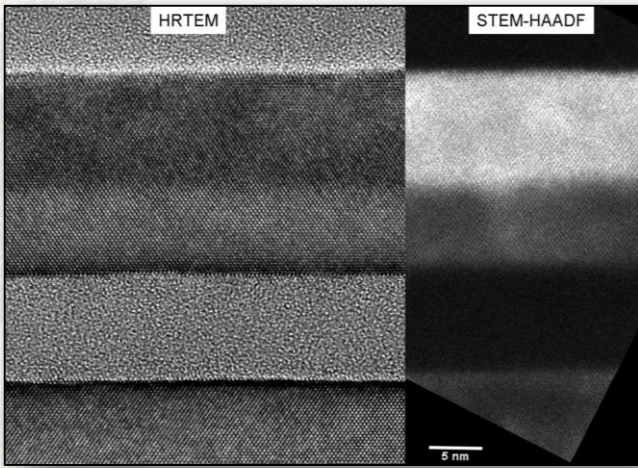
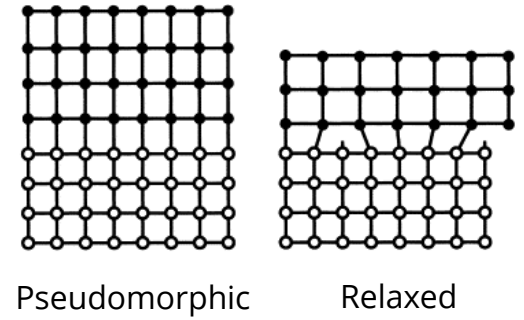
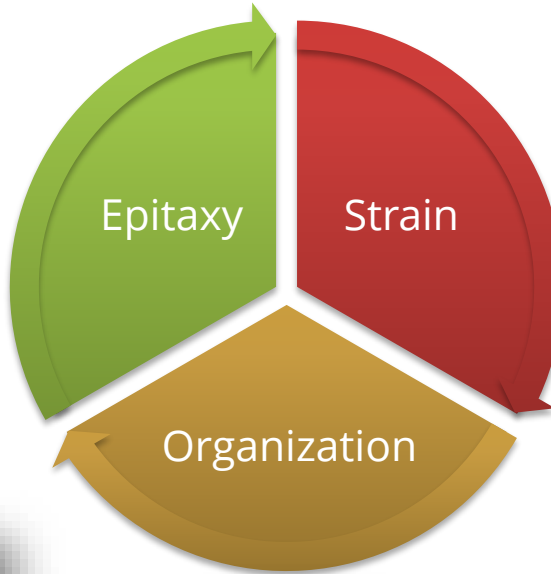
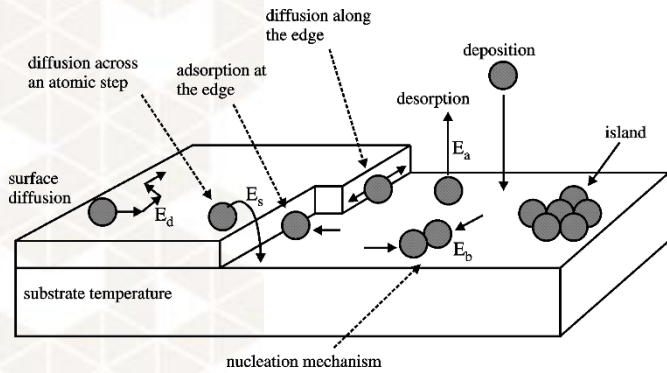
TEM cross section



Kim *et al.*, *Adv. Mater.*, **20**, 24, 1521 (2008)

Introduction

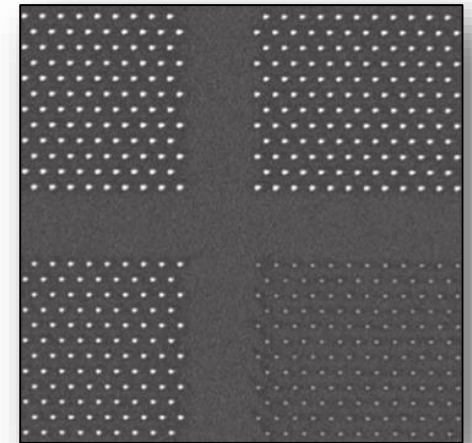
Silicon substrate preparation for SiGe nanostructures growth



2D



1D



0D

Synopsis

Introduction

- Usual Si cleaning process
- Bottom-up is not, yet, efficient

Ion beam lithography

- Fast review of lithography technics
- Focused Ion Beam principles
- High resolution
- Limits: surface and volume defects

Using Focus Ion Beam for surface preparation

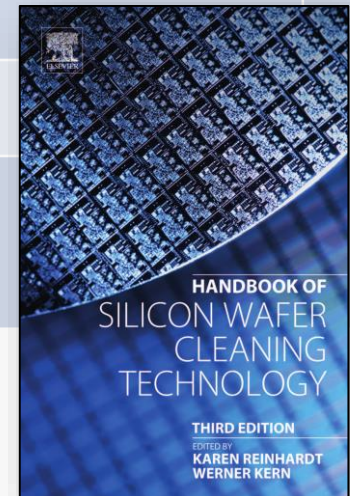
- Gold dewetting
- Au dots
- Silicon dewetting

Conclusion

Usual Si substrate cleaning processes

Wet cleaning : industrial solutions

Name	Diluted HF	Caro	RCA 1	RCA 2	Aqua Regia	...
Solutions	HF 1% T=20°C t=1min	H ₂ SO ₄ (d=1,83) 3 vol. H ₂ O ₂ 30% 1 vol. T=140°C t=10min	NH ₄ OH 0.25 to 1 vol. H ₂ O ₂ 30% 1 vol. H ₂ O ₅ vol. T=70°C to 80°C t=10min Rinsing DI 5 to 10 min	HCL 37% 1 vol. H ₂ O ₂ 30% 1 vol. H ₂ O 5 to 6 vol. T 70°C to 80°C t=10min	HCl (d= 1,19) 2 to 4 vol. HNO ₃ (d= 1,40) 1 vol. T= room to boiling	
Target	Native SiO ₂ Ti, TiN	Organic materials (Metallic particles)	Particles	Metallic materials		
Principe	Acid dissolution	Oxidization followed by acid dissolution		Acid dissolution		
Advantage	High dilution	Low roughness	Eliminate particles			
Limits	No action on plentiful metallic particles and organic particles	Difficult rinsing possible particle redeposition	Weak efficiency on organic and metallic materials	Difficult rinsing possible particle redeposition		



CARO cleaning: DHF + CARO
 RCA cleaning: SC1 + SC2
 Clean B: CARO + (D)HF + RCA

Usual Si substrate cleaning processes

Dry cleaning

Categories

- Physical interactions
- Physically-enhanced chemical reactions
- Chemical thermal reaction
- Mechanical process

Contaminants removal by dry cleaning

- **Organic contaminants:** volatilization, UV/O₃ reaction, remote or downstream oxygen plasma treatment
- **Native, chemical, thermal oxide, silicate glasses:** chemical etching, physical sputter etching, low-energy ECR plasma etching
- **Metal and absorbed ions:** remote plasma, photo-induced reaction
- **Particle:** vapor etching

Thermal Desorption and Oxidation

- 700°C: reduce carbon contamination
- 800°C: eliminate carbon contamination and oxide
- 900°C: oxide and a carbon contamination below detection limit

Dewetting process organization

Chemical potential

$$\mu^\beta = \underbrace{\mu_m^\beta(T)}_{\text{Original } \beta \text{ phase chemical potential}} + \underbrace{\frac{1}{2} C^\beta \varepsilon^2}_{\text{Elastic strain energy density}} - \underbrace{\sigma_n \Omega^\beta}_{\text{Work (pV)}} + \underbrace{\left[\left(\gamma^{\alpha\beta} + \frac{d^2 \gamma^{\alpha\beta}}{d\theta_1^2} \right) \frac{1}{R_1} + \left(\gamma^{\alpha\beta} + \frac{d^2 \gamma^{\alpha\beta}}{d\theta_2^2} \right) \frac{1}{R_2} \right]}_{\text{Surface energy (including anisotropy)}} \Omega^\beta$$

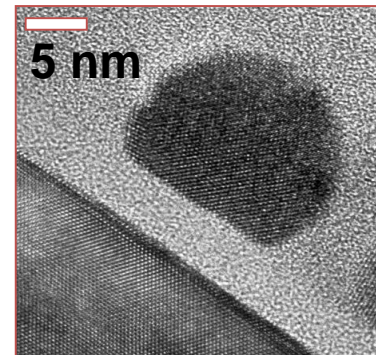
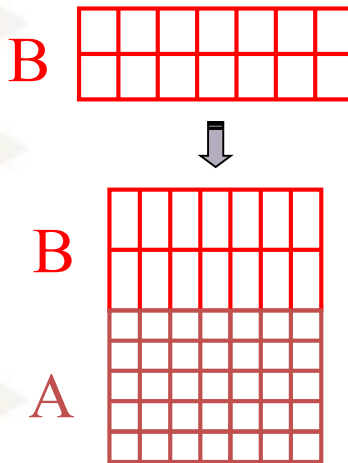
Original β phase
chemical potential

Work (pV)

Surface energy (including anisotropy)

- Ω : atomic volume (or molecular)
- R_i : main radius of curvature
- θ_i : surface orientation

Elastic strain
energy density



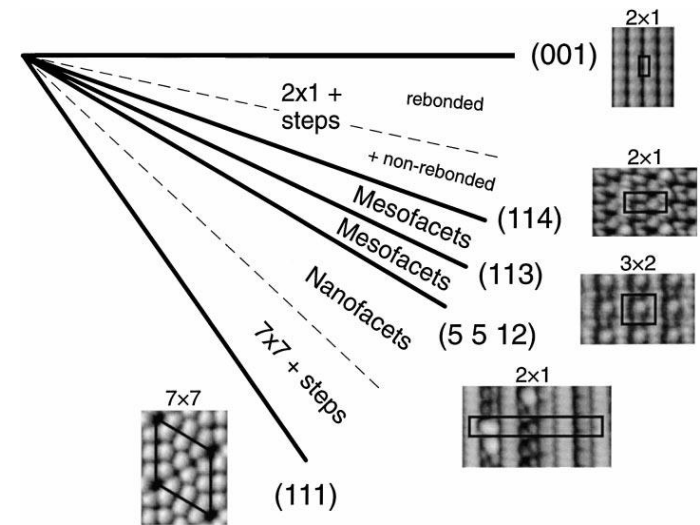
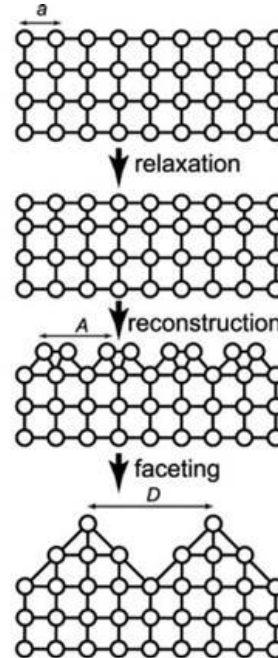
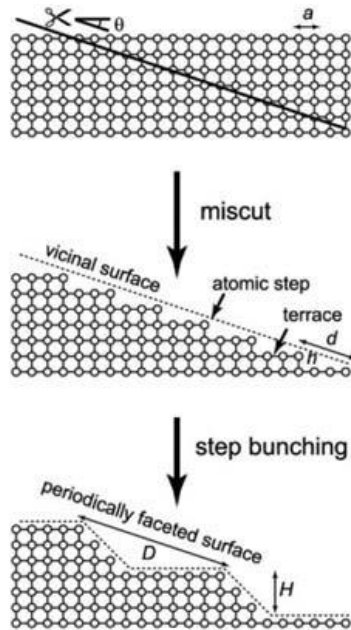
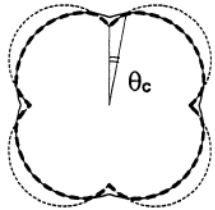
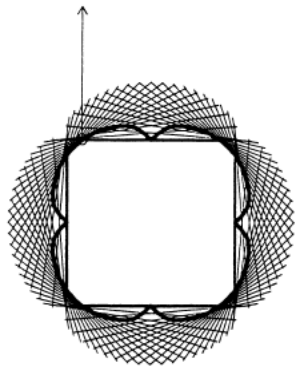
+ Scattering Barriers
Surface defects, steps, ...

Growth and self-organisation of SiGe nanostructures
J.N. Aqua, I. Berbezier, L. Favre, T. Frisch, A. Ronda, Phys. Rep. (2013)

SiGe Nano-structures organization

Self organization strategies

- Vicinal surfaces
- Vicinal reconstructed surfaces
- Surfactant effects

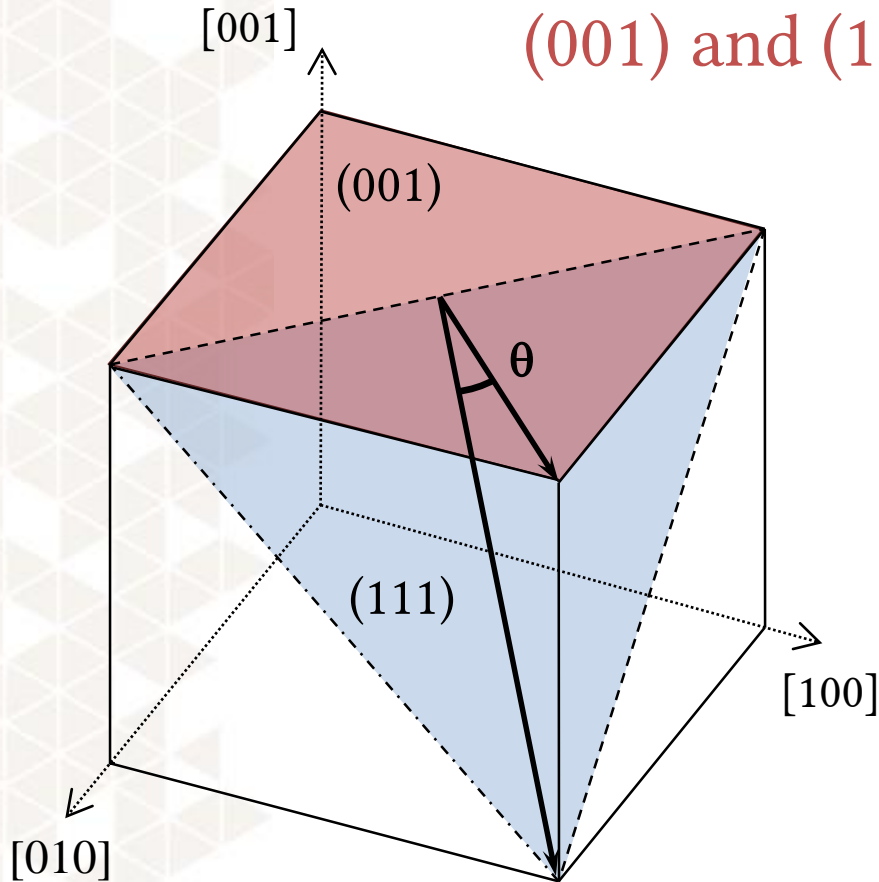


H.-C. Jeong and E.D. Williams, Surface Science Reports 34, 171–294 (1999)

SiGe Nano-structures organization

Vicinal surfaces

(001) and (111) misoriented surfaces



θ : from 0° to 10°

(001) plane misorientation:

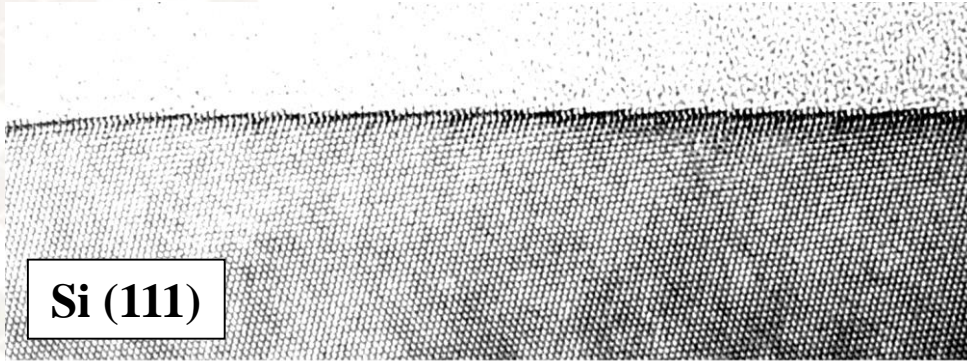
- around $[1\bar{1}0]$ axis
- to (118) plane

(111) plane misorientation:

- around $[\bar{1}10]$ axis
- to $[11\bar{2}]$ and $[\bar{1}\bar{1}2]$ axis

SiGe Nano-structures organization

Homoepitaxy on vicinal surfaces: Si/Si(111)-(11 $\bar{2}$) vicinal

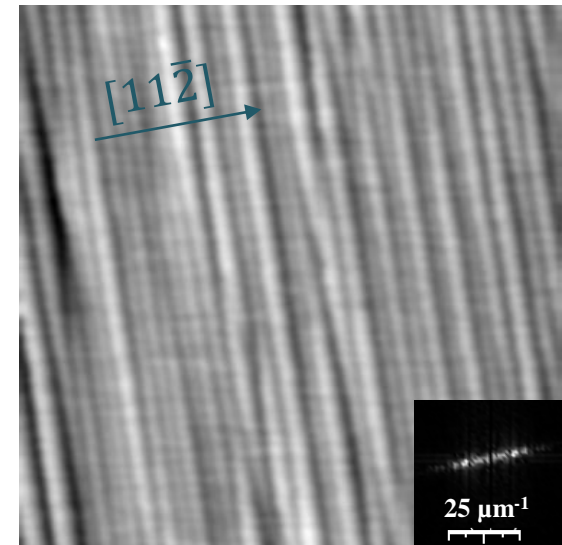
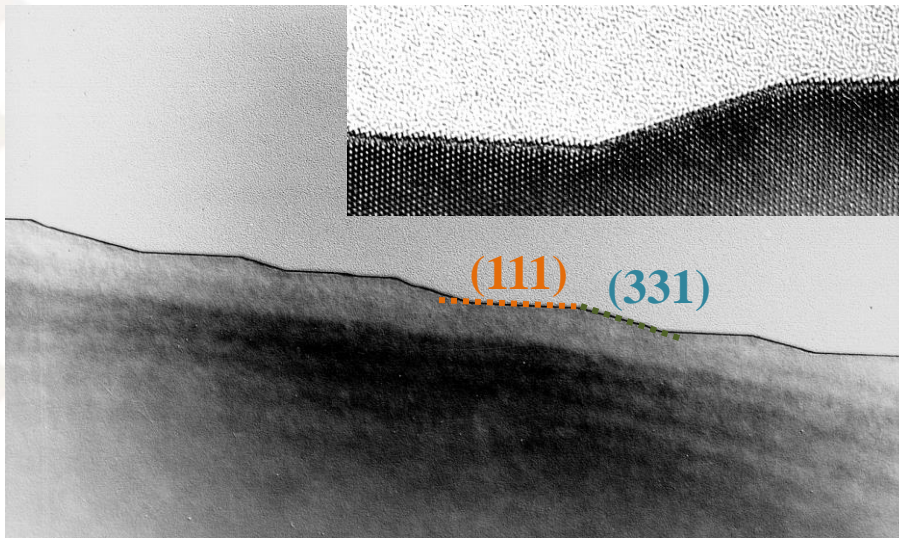


cut surface

(111) misoriented around $[\bar{1}10]$
in direction of $[11\bar{2}]$

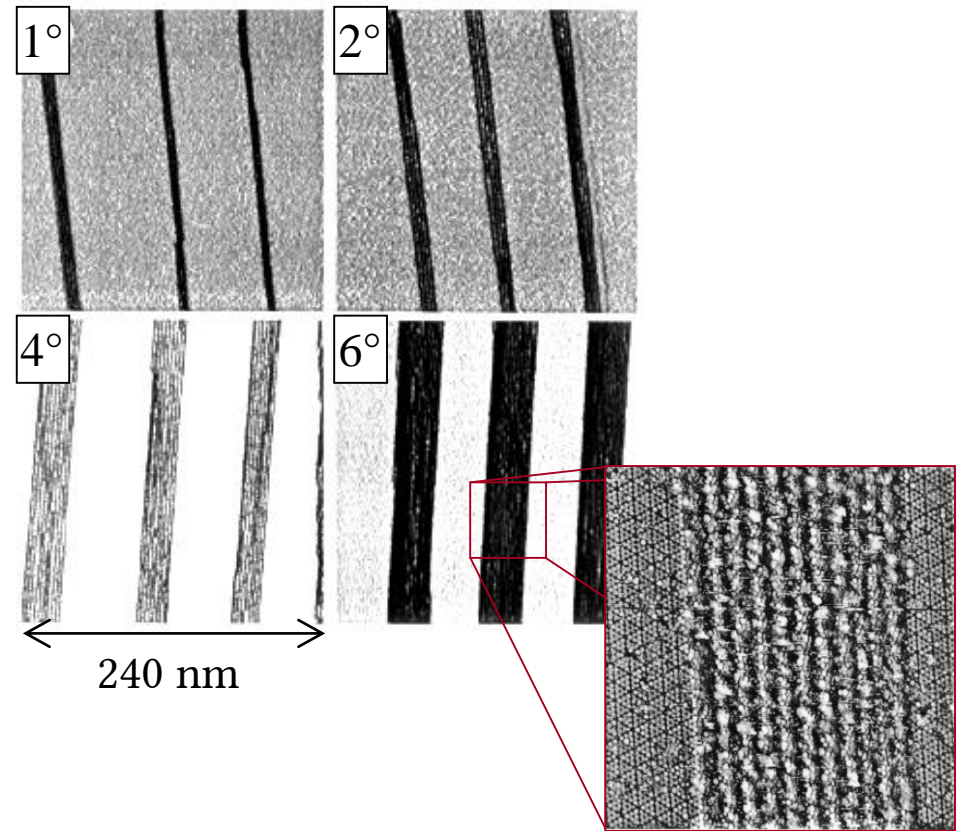
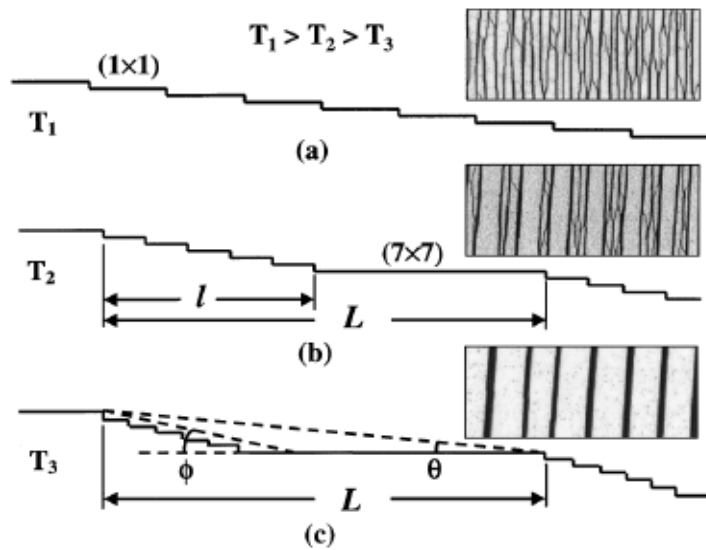
faceting process

After
annealing
or growth



SiGe Nano-structures organization

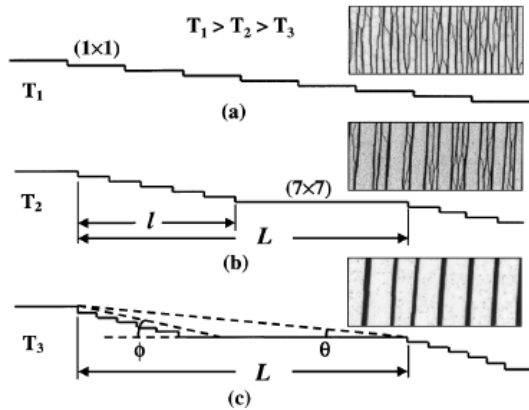
Homoepitaxy on vicinal surfaces: Si/Si(111)-(11 $\bar{2}$) vicinal



F. K. Men, *et al.*, Phys. Rev. Lett., **88** (2002)

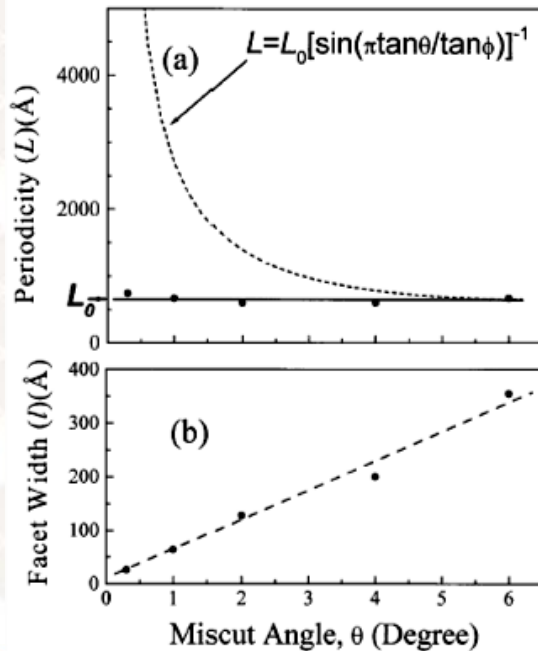
SiGe Nano-structures organization

Homoepitaxy on vicinal surfaces: Si/Si(111)-(11 $\bar{2}$) vicinal



Two steps process:

- (elastic) free energy minimization
 \Rightarrow **L tuning** (L independent from miscut if $l = L/2$)
- facet free energy minimization
 \Rightarrow **l tuning**

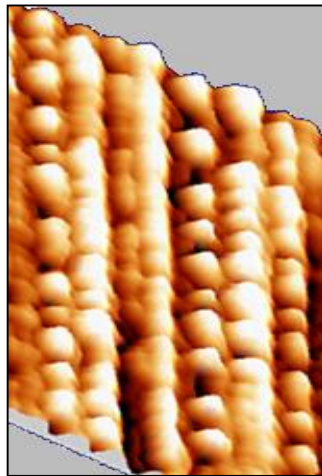
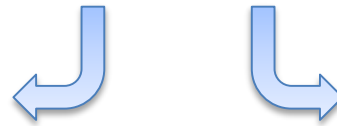
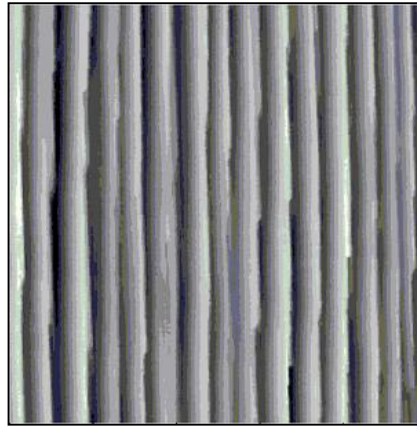


$$\Delta f(T) = -\frac{\pi c}{L} \cot\left(\frac{l}{L}\pi\right)$$

$$L = \pi a_0 \left[\sin\left(\frac{l}{L}\pi\right) \right]^{-1}$$

SiGe Nano-structures organization

Homoepitaxy on vicinal surfaces: Si/Si(111)-(11 $\bar{2}$) vicinal



On steps bunching



On (311) facets

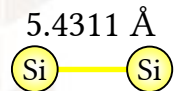
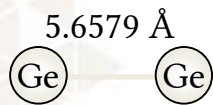
Si dots organisation

SiGe Nano-structures organization

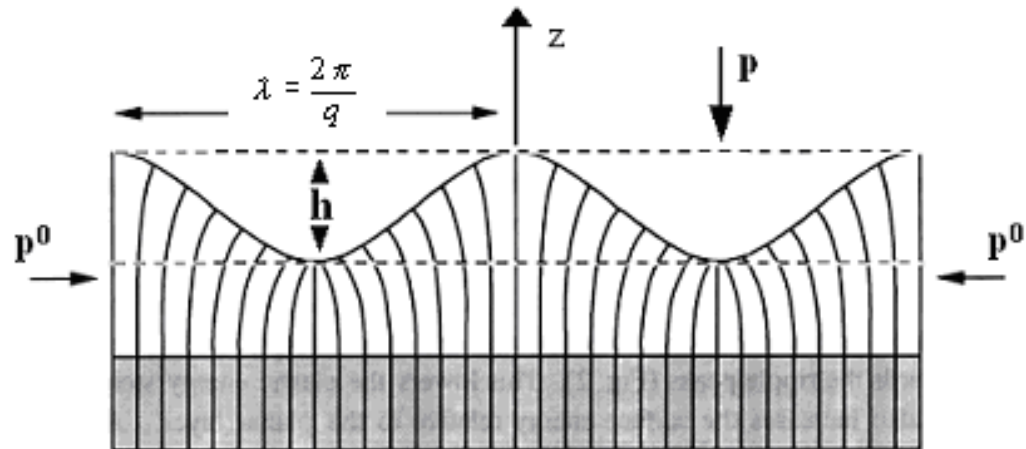
Heteroepitaxy on vicinal surfaces: SiGe/Si



TEM cross-section



$\varepsilon = 4.2 \%$



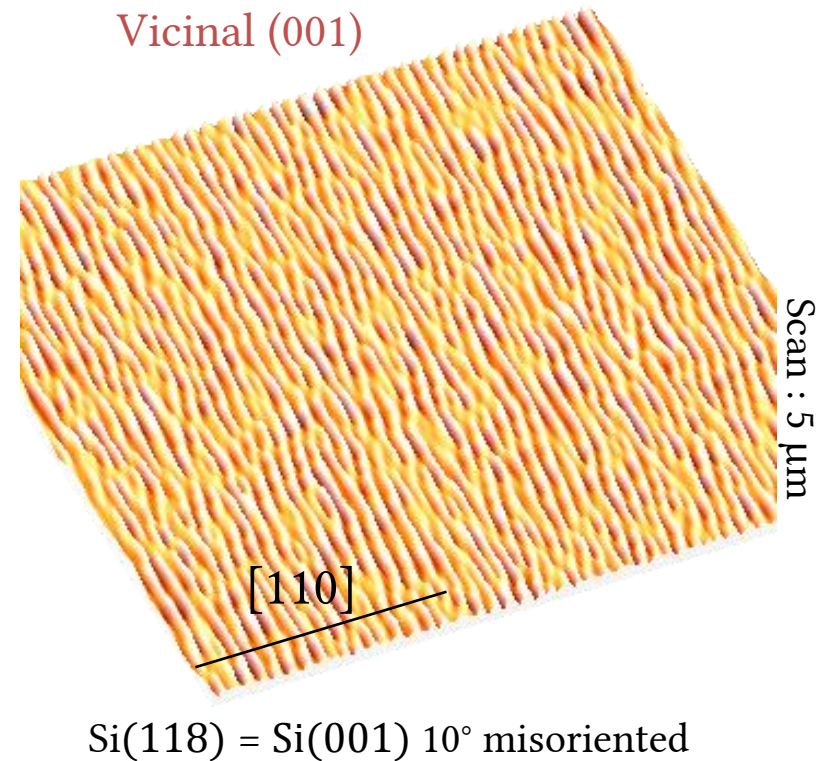
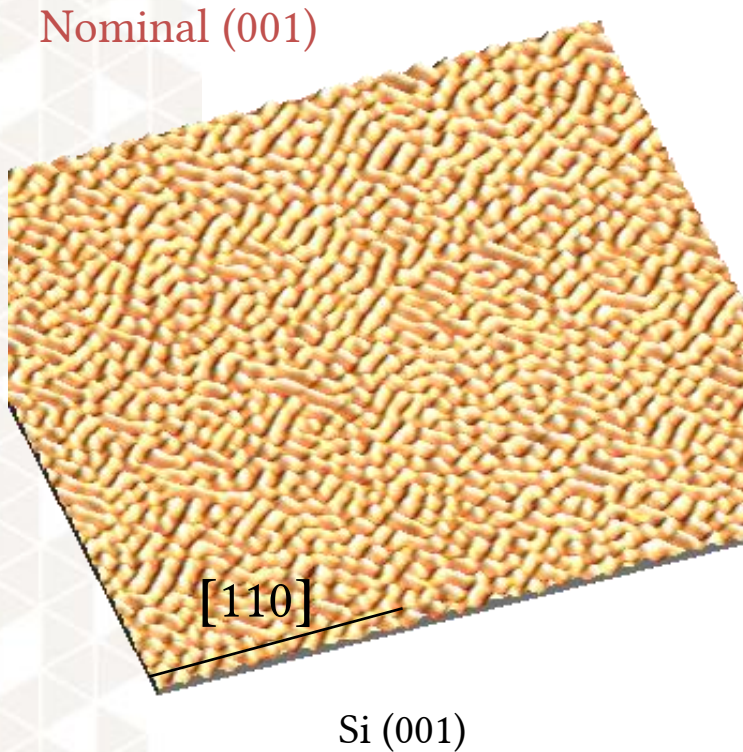
Stranski krastanov growth mode

SiGe Nano-structures organization

Heteroepitaxy on vicinal surfaces: SiGe/Si

Stransky Krastanov
first step: pseudomorphic epitaxy

$x = 0.4$
 $m = 1.6 \%$
 $h = 50 \text{ \AA}$



P.D. Szkutnik, A. Sgarlata, A. Balzarotti, N. Motta, A. Ronda, I. Berbezier, PRB, 2006

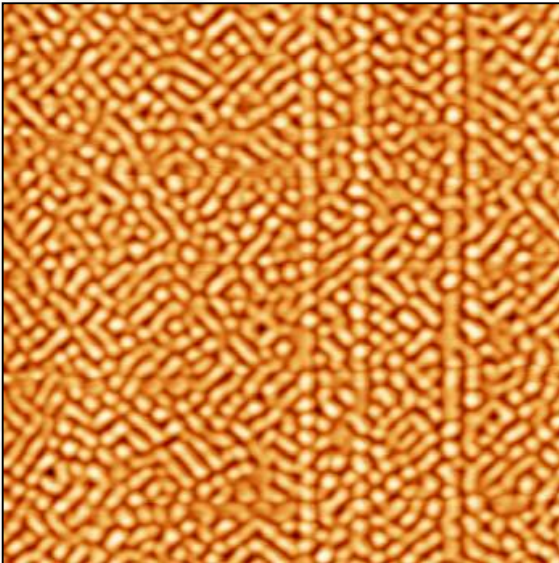
SiGe Nano-structures organization

Heteroepitaxy on vicinal surfaces: Ge/Si

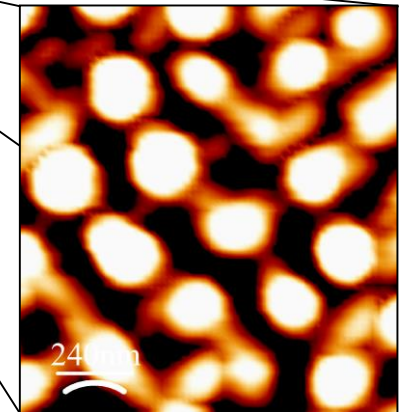
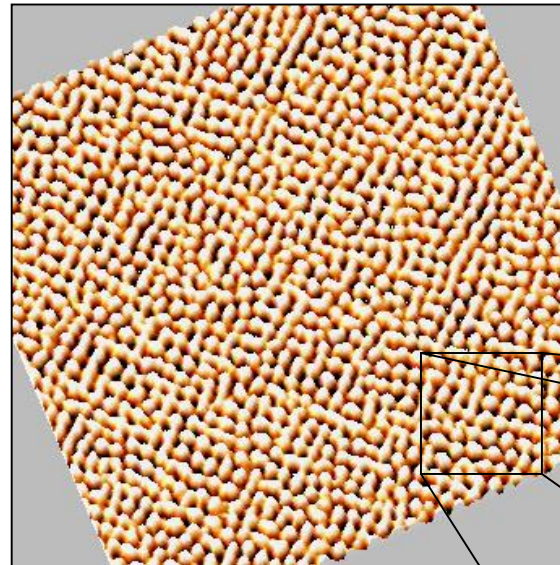
Stransky Krastanov
Second step: dots nucleation

$x = 0.4$
 $m = 1.6 \%$
 $h = 50 \text{ \AA}$

Nominal (001)



On top of undulations



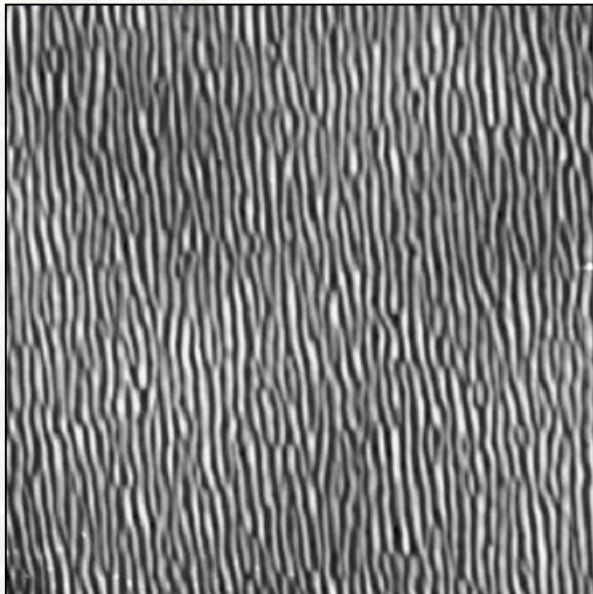
SiGe Nano-structures organization

Heteroepitaxy on vicinal surfaces: Ge/Si

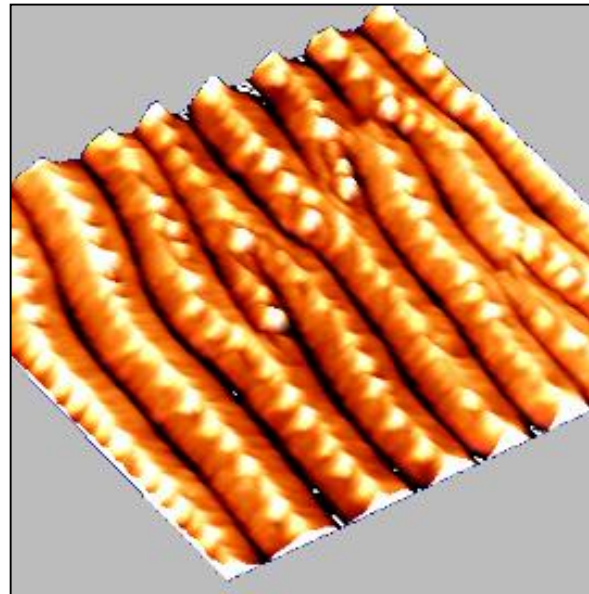
$x = 0.4$
 $m = 1.6 \%$
 $h = 50 \text{ \AA}$

Stransky Krastanov
Second step: dots nucleation

Vicinal (001)



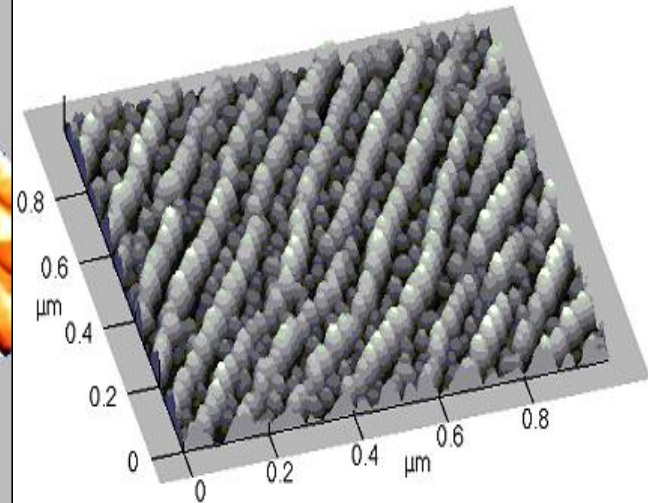
On top of undulations



$2 \cdot 10^{11}$ dots / cm^2

with surfactant:

On top and bottom
of undulations



Chemical influence
on surface energy

Misoriented substrates and strain instability leads to self-organization

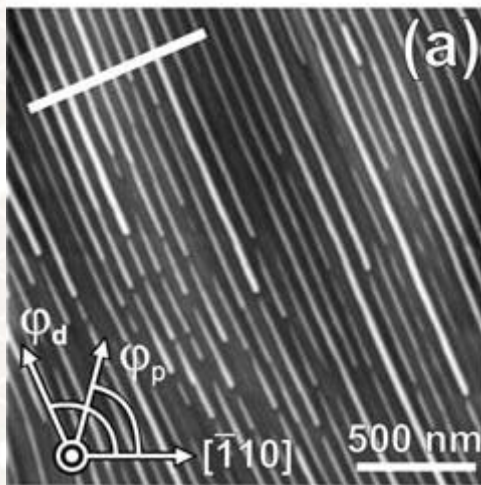
Bad control of size distribution and fixed periodicity

Berbezier *et al.*, Appl. Phys. Lett., **83** (2004)

SiGe Nano-structures organization

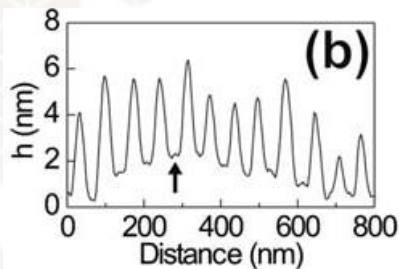
Heteroepitaxy on Very High Index vicinal surfaces: Ge/Si

Self-organized Ge nanowires formed on Si(173 100 373)

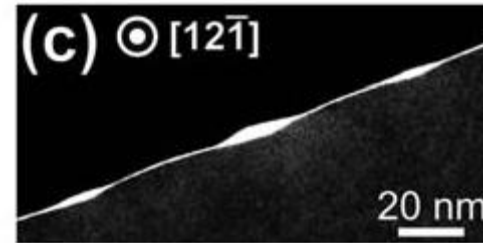


(a) AFM image
2.0 x 2.0 μm^2

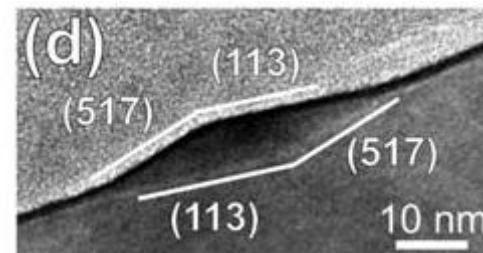
altitude angle $\theta = 28.21$
azimuthal direction $\varphi_p = 75^\circ$



(b) Height h as a function of the position along the white line in (a)



(c) HADF STEM



(d) HRTEM

[12 $\bar{1}$] zone-axis

K. Ohmori, *et al.*, Nano Lett., 5, 369–372 (2005)

Synopsis

Introduction

- Usual Si cleaning process
- Bottom-up is not, yet, efficient

Ion beam lithography

- Fast review of lithography technics
- Focused Ion Beam principles
- High resolution
- Limits: surface and volume defects

Using Focus Ion Beam for surface preparation

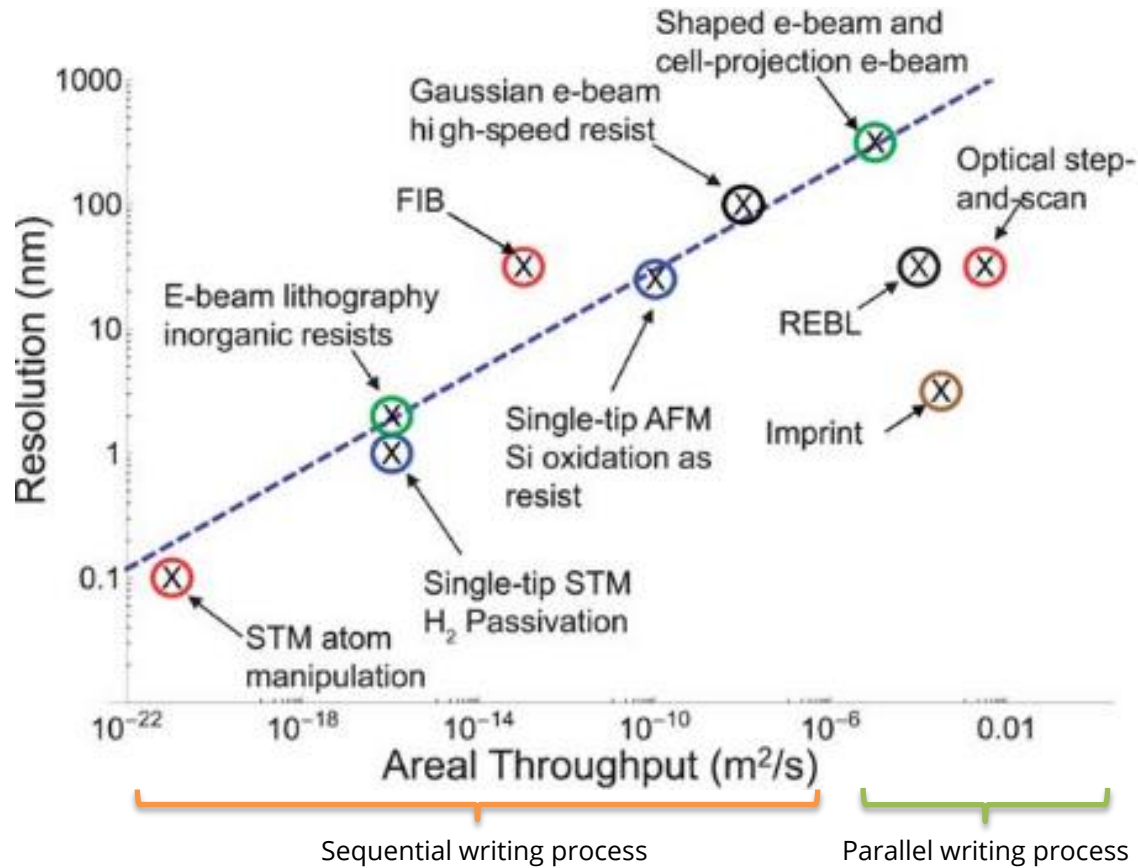
- Gold dewetting
- Au dots
- Silicon dewetting

Conclusion

Lithography technics

Development of new technologies: working at nanoscale

Top-Down lithography techniques

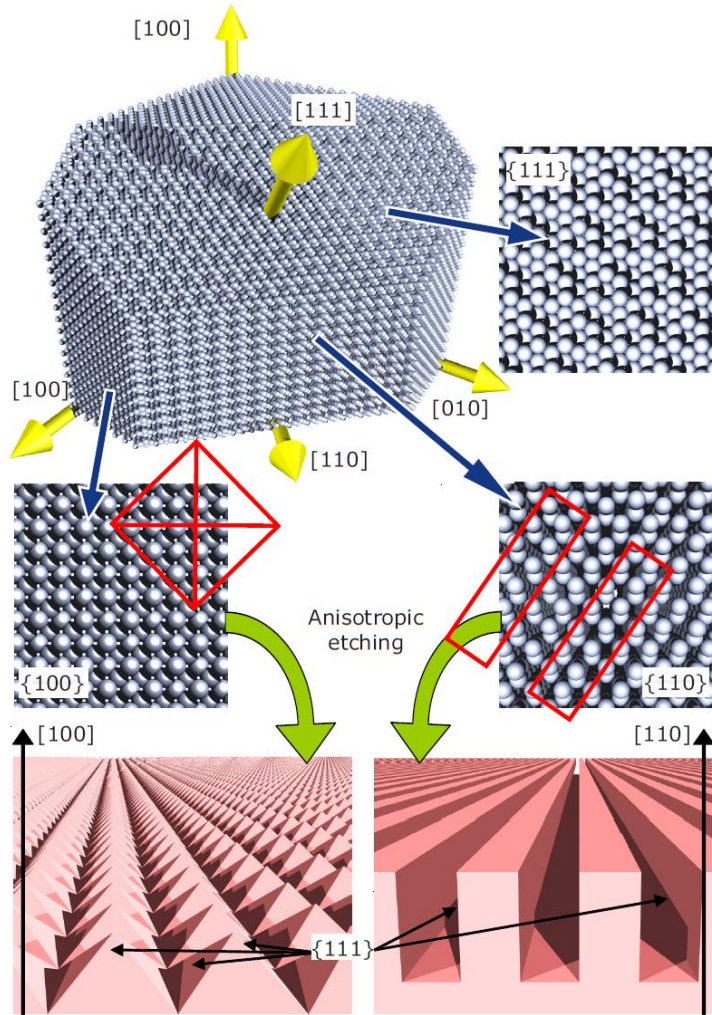


Industrial productivity: 10⁻⁴ m²/s

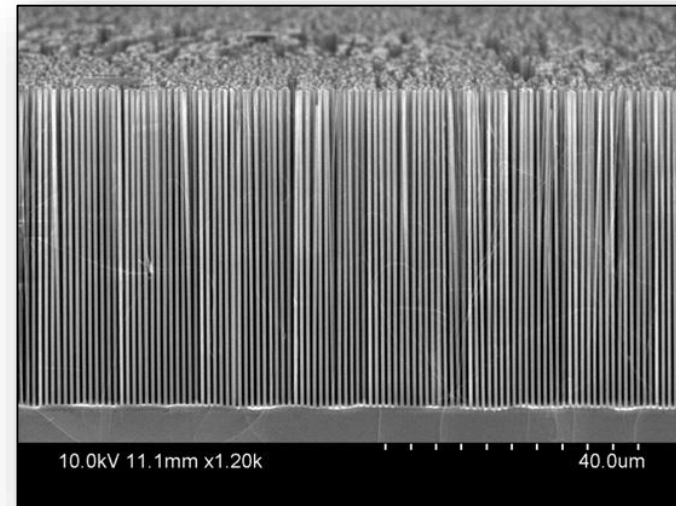
J. A. Little, G. M. Gallatin, *Nanoscale* 3, 2679–2688 [2011]

Wet etching

Development of new technologies



Etchant	Etch rate ratio		Etch rate (absolute)			Advantages (+) Disadvantages (-)
	(100)/(111)	(110)/(111)	(100)	Si ₃ N ₄	SiO ₂	
KOH (44%, 85°C)	300	600	1.4 mm/min	<1 Å/min	14 Å/min	(-) Metal ion containing (+) Strongly anisotropic
TMAH (25%, 80°C)	37	68	0.3-1 mm/min	<1 Å/min	2 Å/min	(-) Weak anisotropy (+) Metal ion free
EDP (115°C)	20	10	1.25 mm/min	1 Å/min	2 Å/min	(-) Weak anisotropy, toxic (+) Metal ion free, metallic hard masks possible

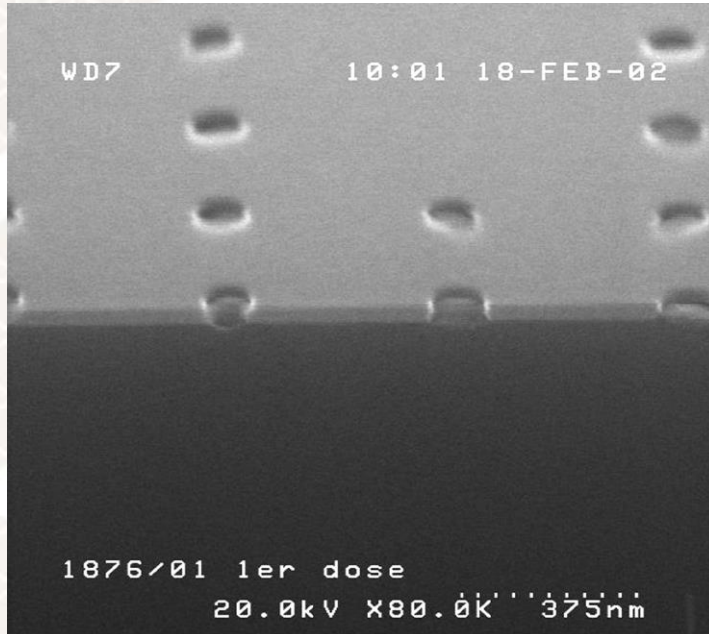


Si nanowires with diameter of 550 nm, height of 51 μm, thus an aspect ratio of ≈ 93 , produced through Au-MacEtch with Au mesh film patterned using soft lithography on p+ Si

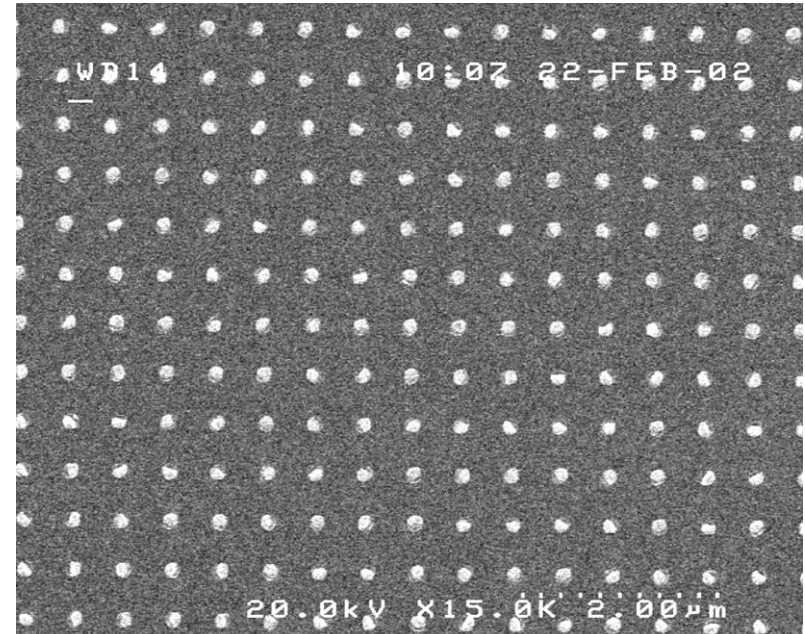
e-beam lithography

CVD deposition on e-beam patterns

Ge nanodots growth on patterned oxide layer



e- beam patterning



Selective CVD Ge growth

Size > 80 nm

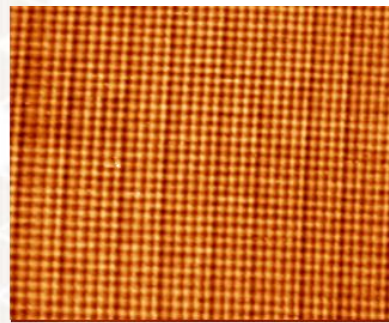
Density $\approx 10^{10}/\text{cm}^2$

O. Kermarrec, Y. Campidelli, D. Bensahel, STMicroelectronics

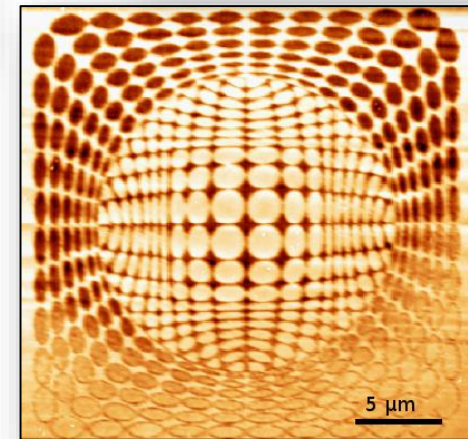
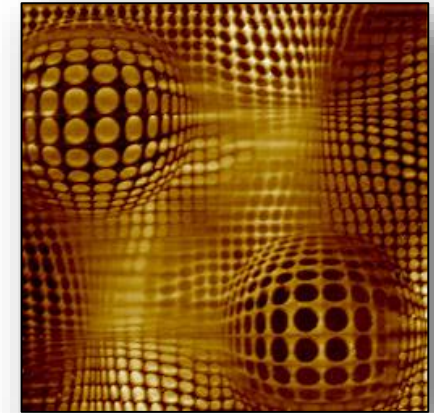
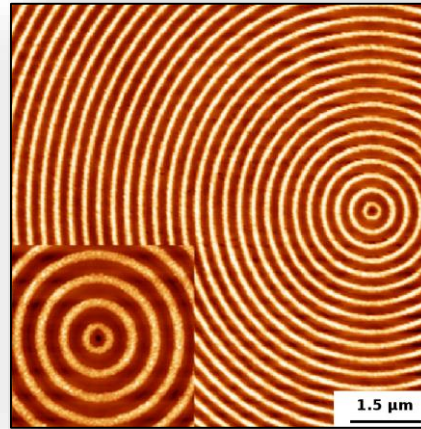
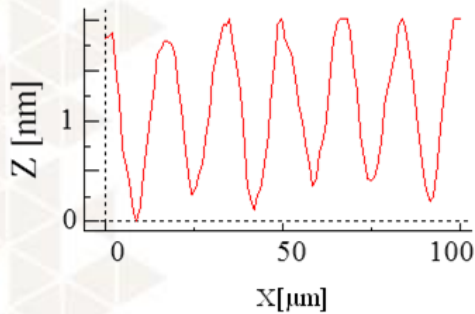
Ion-beam lithography

Silicon substrate FIB patterning

Pitch	24 nm
Pattern diameter	15 nm



X [μm]



High resolution

Pattern flexibility

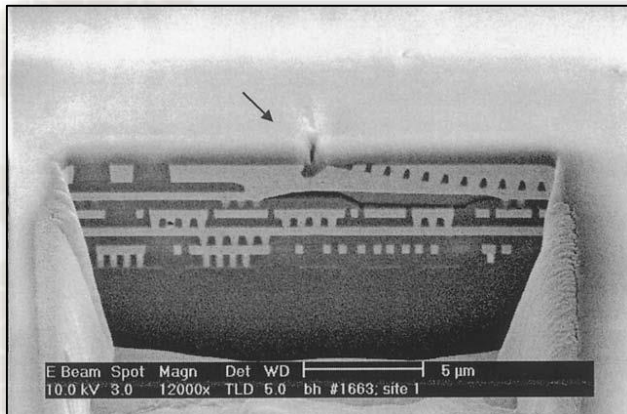
Ion-beam lithography

FIB use

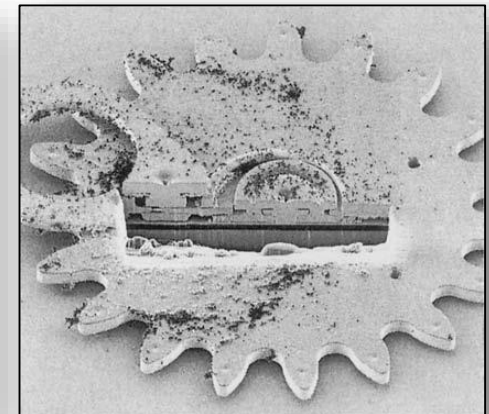
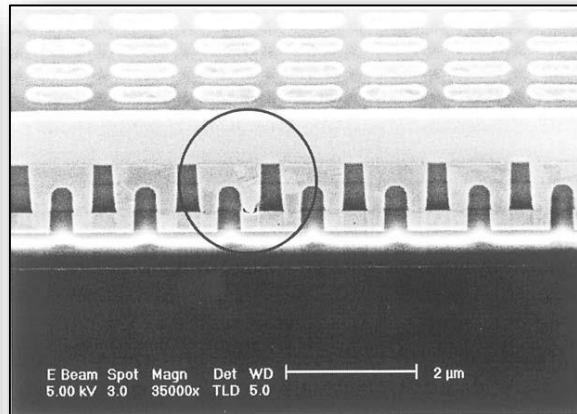
Failure analysis



Cross-section tool



Microelectronic



Mechanic

Ion-beam lithography

FIB use

Failure analysis

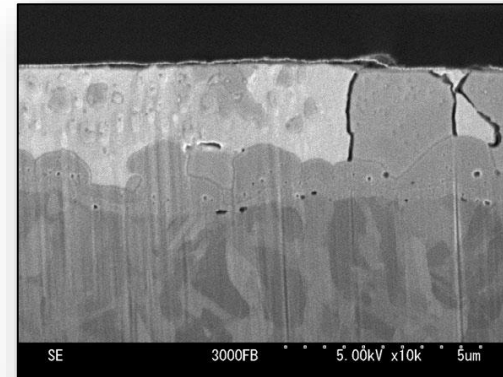
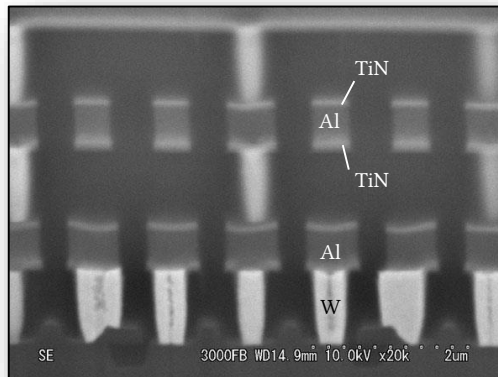


Z contrast

Channeling effect

Secondary Electron (SE)

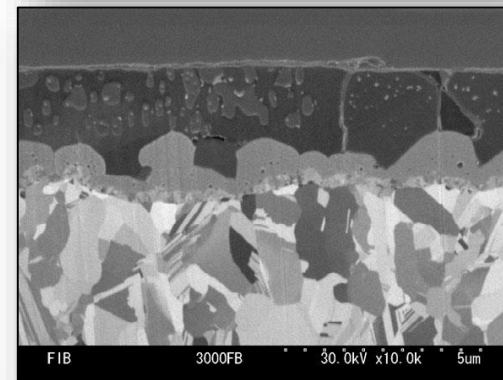
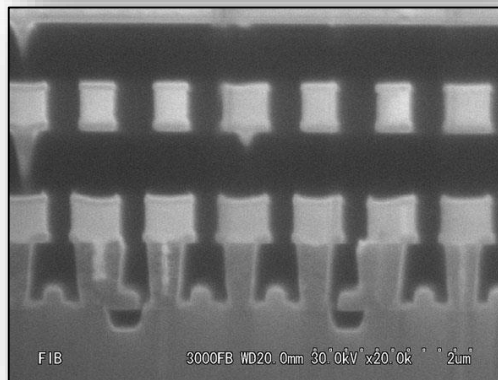
Si device
(static random
access memory)



Solder (Pb-Sn)

Cu

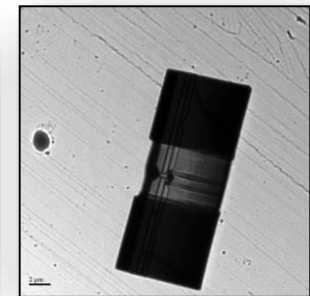
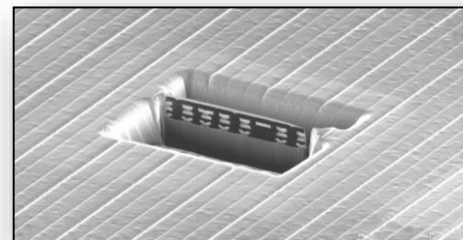
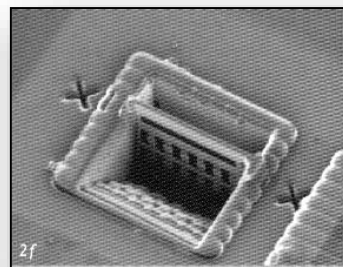
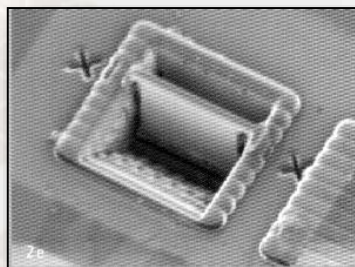
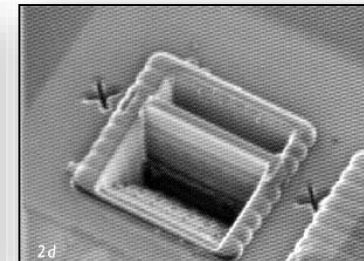
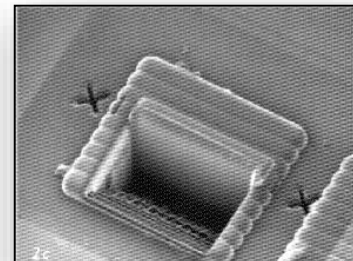
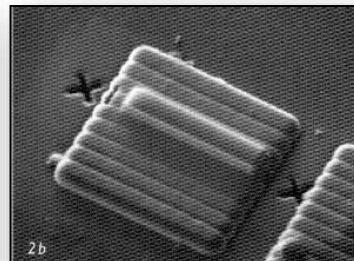
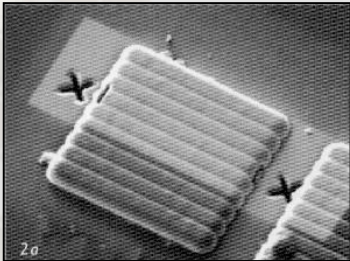
Ion-induced
Secondary Electron (ISE)



Ion-beam lithography

FIB use

TEM lamella preparation



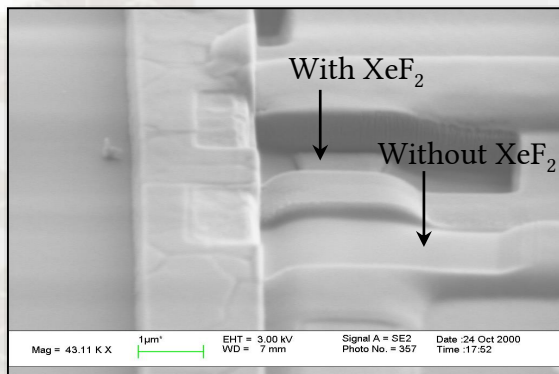
Ion-beam lithography

FIB use

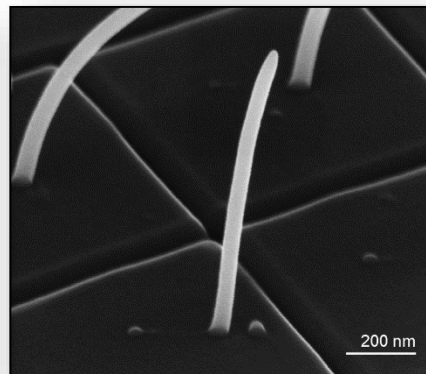
Gas Injection System



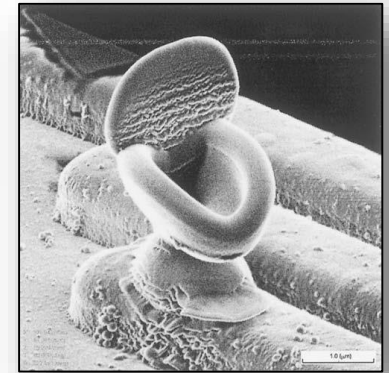
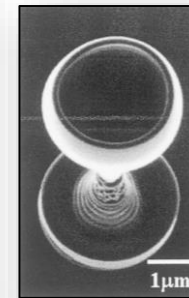
Selective/enhanced etching



Deposition (W, Pt, SiO₂, ...)



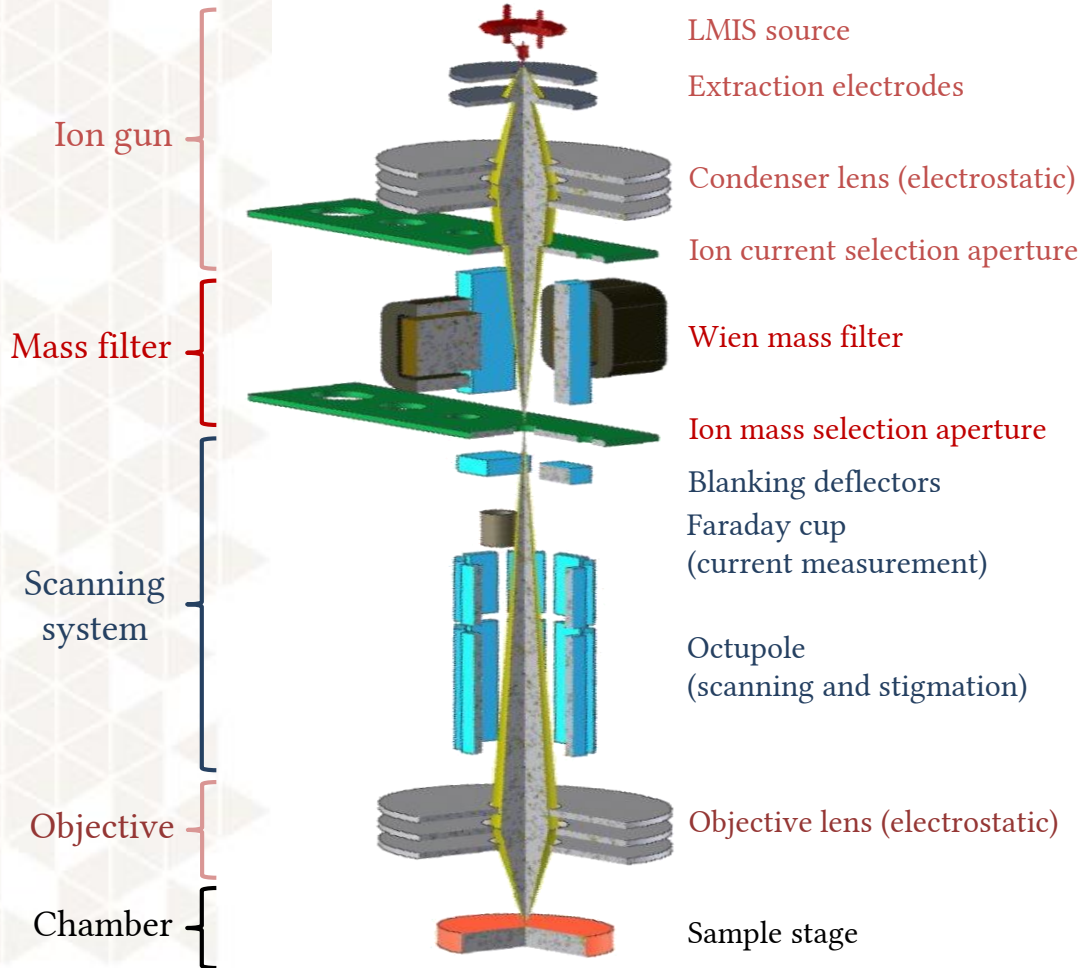
GIS deposition of 50 nm thin,
1 µm tall, Pt nanowires



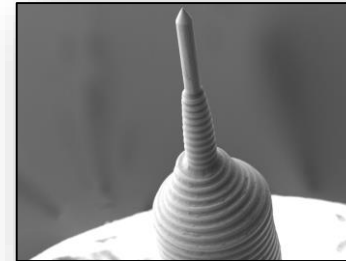
Courtesy of Orsay Physics

Ion-beam lithography

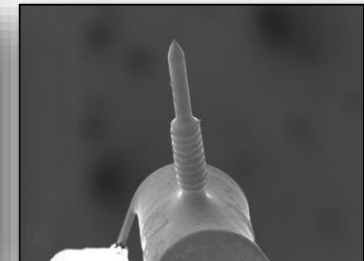
Ion column with mass filter



Orsay Physics LMIS:



Ga



Au-Si

Working voltage: 30 kV

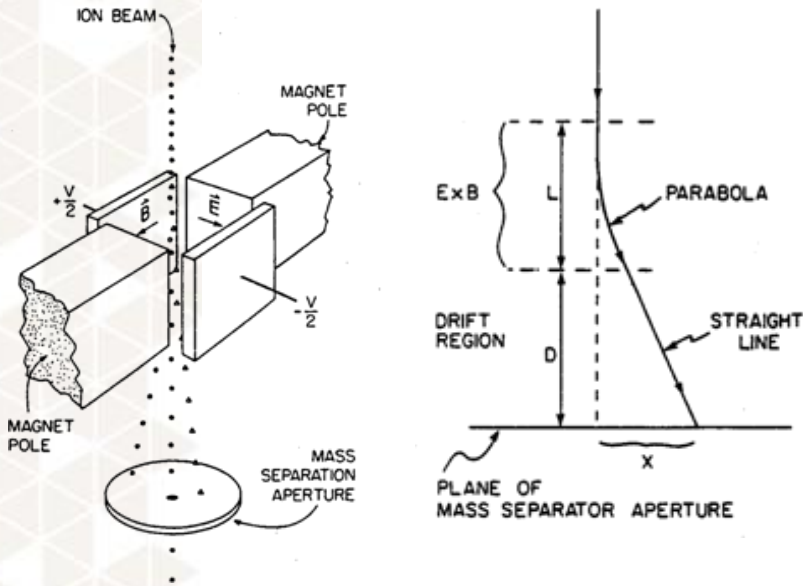
Intensity (Source)	10^6 A/cm ²
Source size	50 nm
Energetic dispersion	5-10 eV
Species	Ga, Au, Si, Ge, ...

Sputtering rate: Ga⁺ 30 kV: 2.2 atoms/ion
(0.26 mm³/nA.s)

Courtesy of Orsay Physics

Ion-beam lithography

Mass filter



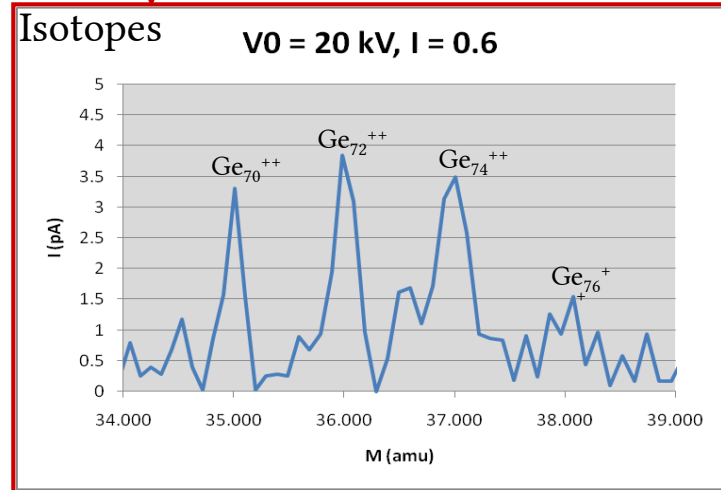
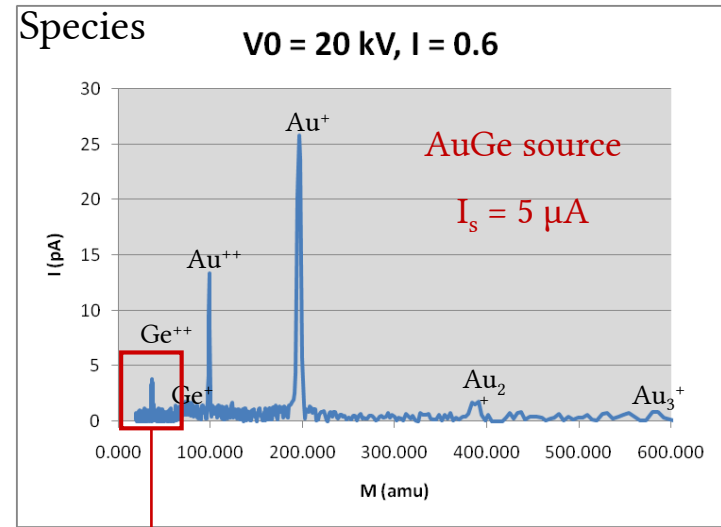
Straight trajectory condition: $m = m_0$

Forces equilibrium condition: $E = v \times B$

$$v = \frac{E}{B} = \frac{V}{dB} \Leftrightarrow \sqrt{\frac{2qV_0}{m_0}} = \frac{V}{dB}$$

Deflection: $m = m_0 + \Delta m$

$$x = \frac{1}{4} \frac{V}{V_0} \frac{L}{d} \left(\frac{L}{2} + D \right) \frac{\Delta m}{m}$$

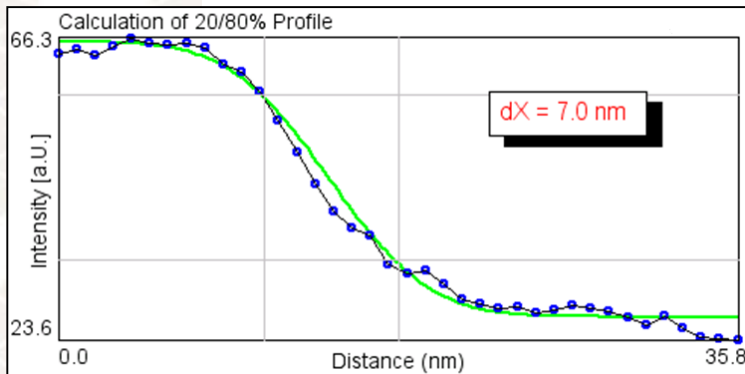


Mass resolution: Au: $r_m < 4\%$ Ga: $r_m < 2\%$

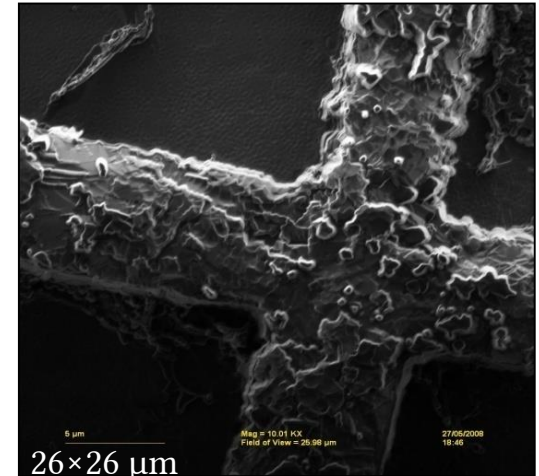
Ion-beam lithography

Performances: lateral resolution

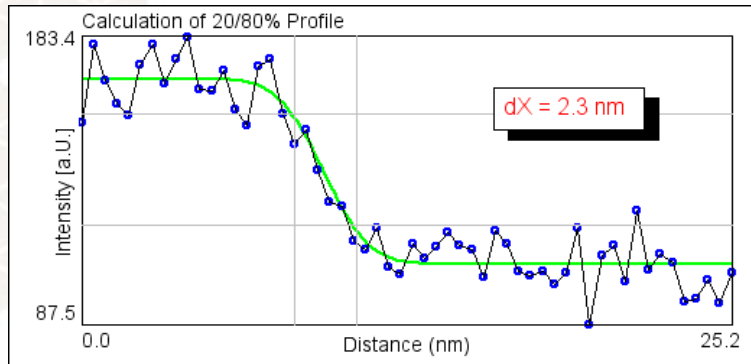
Resolution with mass filtered COBRA-FIB for AuSi source:



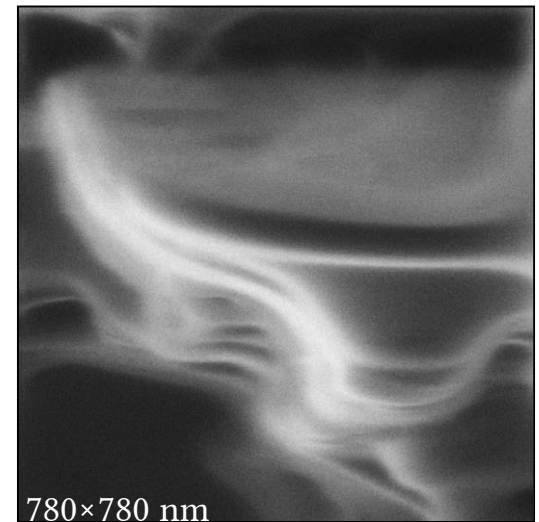
for $I \approx 10 \text{ pA}$ (20-80%)
 \Rightarrow Si beam $< 10 \text{ nm}$
 \Rightarrow Au beam $< 10 \text{ nm}$



Resolution with new COBRA-FIB for Ga source:



for $I \approx 1 \text{ pA}$ (20-80%)
 \Rightarrow Ga beam $< 2.5 \text{ nm}$

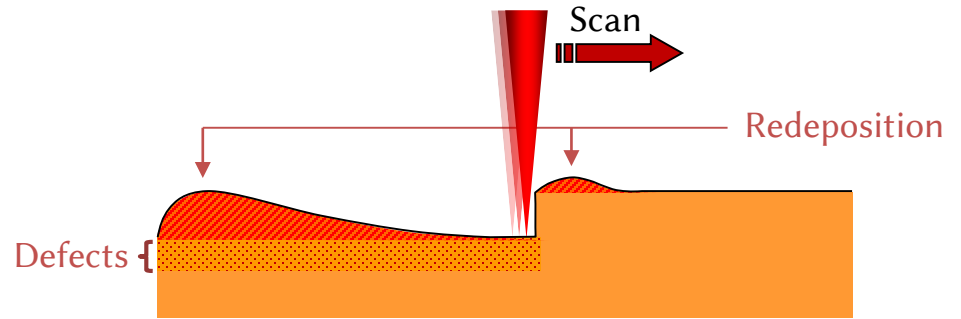
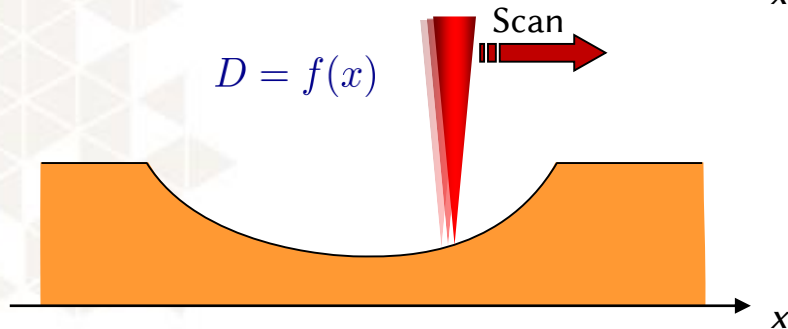
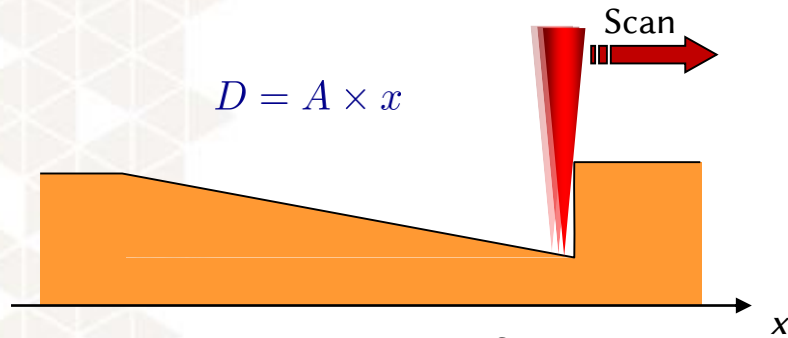
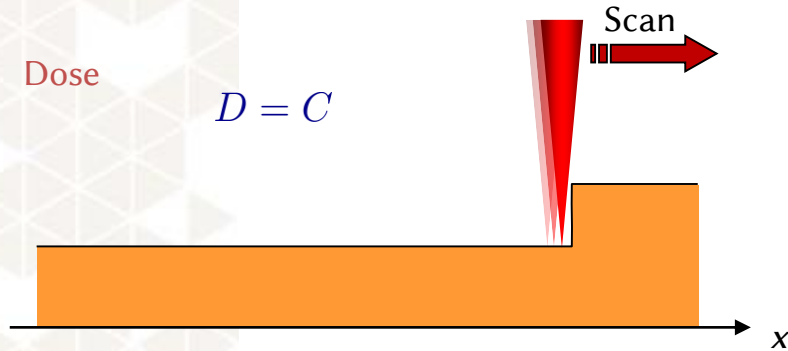


1.7 nm resolution has been performed at 20-80 with Cobra-FIB

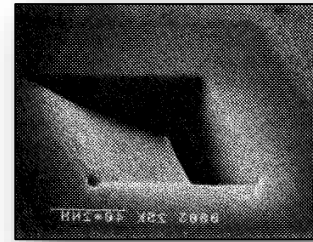


Ion-beam lithography

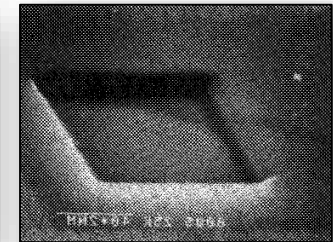
Lithographic process



Si Substrate
30keV Ga⁺ D = 1.9 × 10¹⁸ ions/cm²



300 scan lines
single pass
(2 s/line)



300 scan lines
200 repetitive passes
(10 ms/line)

2 μm

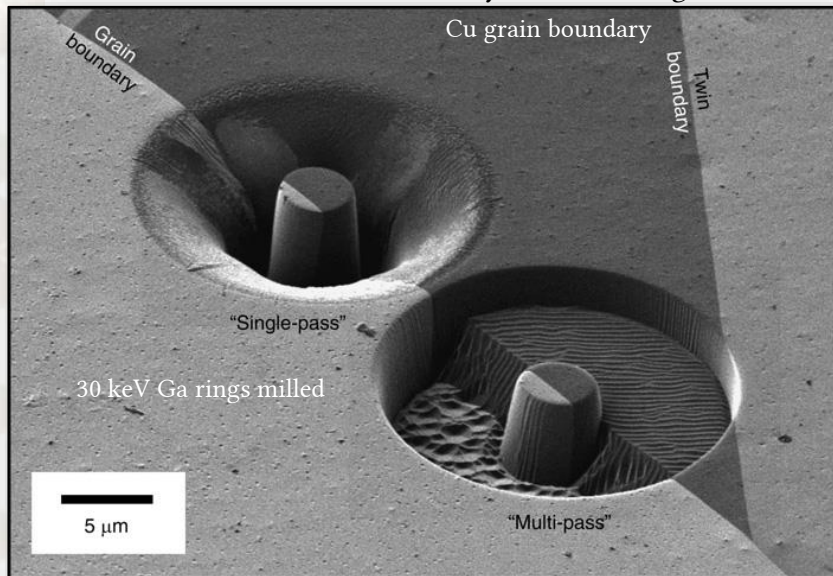


Yamaguchi *et al.*, J. Vac. Sci. Technol. B, 3, 71 (1985)

Ion-beam lithography

Ion/Matter interaction

Ion-induced secondary electron image



Single-pass ring:

- enhanced sputtering yield (deeper trough)
- redeposition (sloped, thicker sidewalls)

Multi-pass ring:

- channeling effects
- surface roughening

C.A.Volkert and A.M.Minor, MRS Bull., **32**, 389 (2007)

Advantages

- Local nanopatterning
- High resolution
- Maskless and resistless process
- (Specific nonpolluting ion sources)

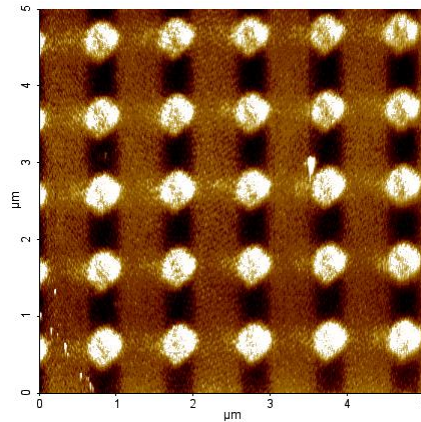
Drawbacks

- Swelling
- Amorphisation
- Redeposition
- Defects (vacancies, interstitials)
- (Implantation)

Ion-beam lithography

FIB patterning: dose

Dose: 0.05×10^{15} ions/cm²

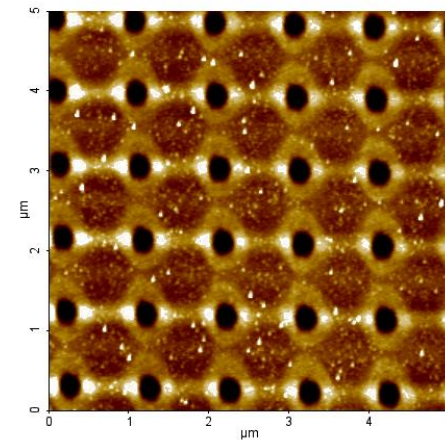


- Implantation
- Amorphization



Swelling

Dose: 4.09×10^{15} ions/cm²



- Implantation
- Amorphization
- Sputtering



Excavation

Ions	Ga ⁺
Acceleration voltage	30 kV
Beam current	2.4 μA
Probe current	1-500 pA

Si wafer (001)

Chemical cleaning:

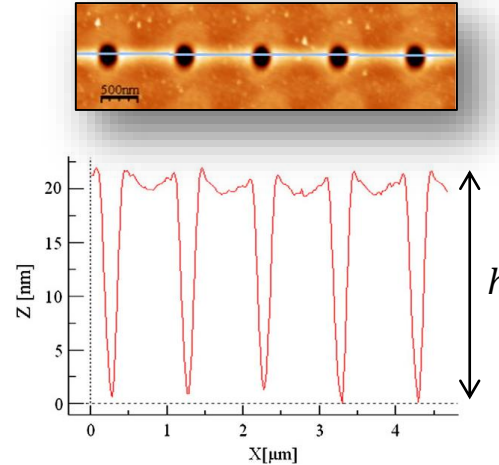
1. HNO₃ (65%) / 70°C / 5 min
2. H₂O (DI) / RT / 1 min
3. HF (49%) : H₂O (1:10) / RT / 30 s

COBRA-FIB from Orsay Physics system integrating SEM Tescan Lyra

Ion-beam lithography

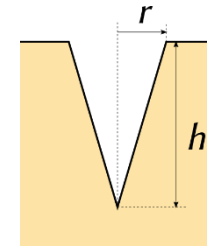
FIB patterning: excavation rate

Ions	Ga ⁺
Acceleration voltage	30 kV
Beam current	2.4 μA
Probe current	1-500 pA

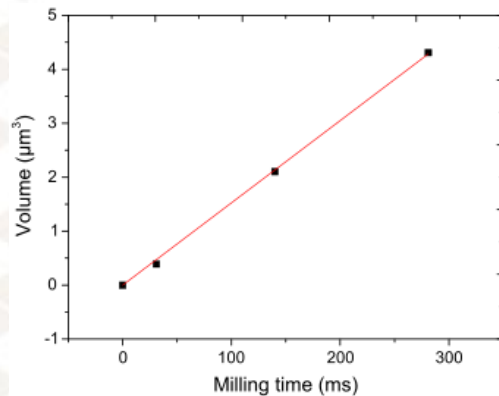


h : AFM measurement
 r : SEM measurement

Holes:
conical shape



Excavated volume: $v = \frac{1}{3}\pi r^2 h$



Excavated volume proportional to the time

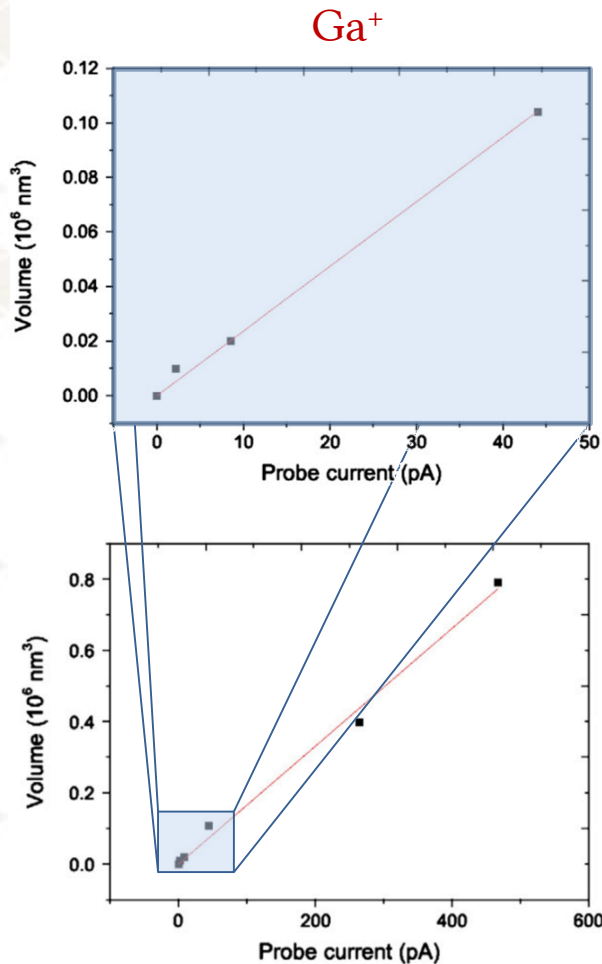
$$V_{\text{Ga}} = K \cdot t$$

with $K = 15.02 \mu\text{m}^3/\text{ms}$

COBRA-FIB from Orsay Physics system integrated SEM Tescan Lyra

Ion-beam lithography

FIB patterning: excavation rate



Excavated volume proportional to probe current

$$v_x = K_x \cdot i_p$$

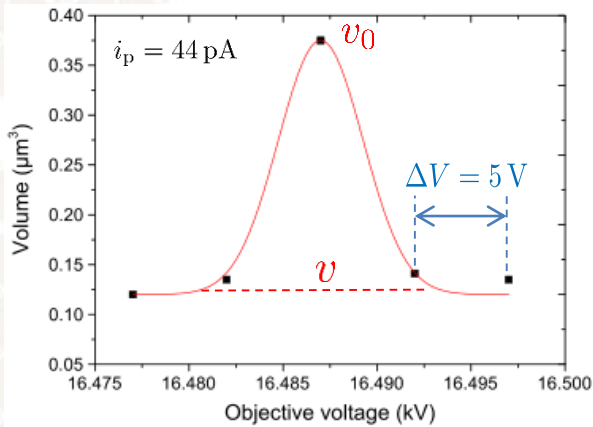
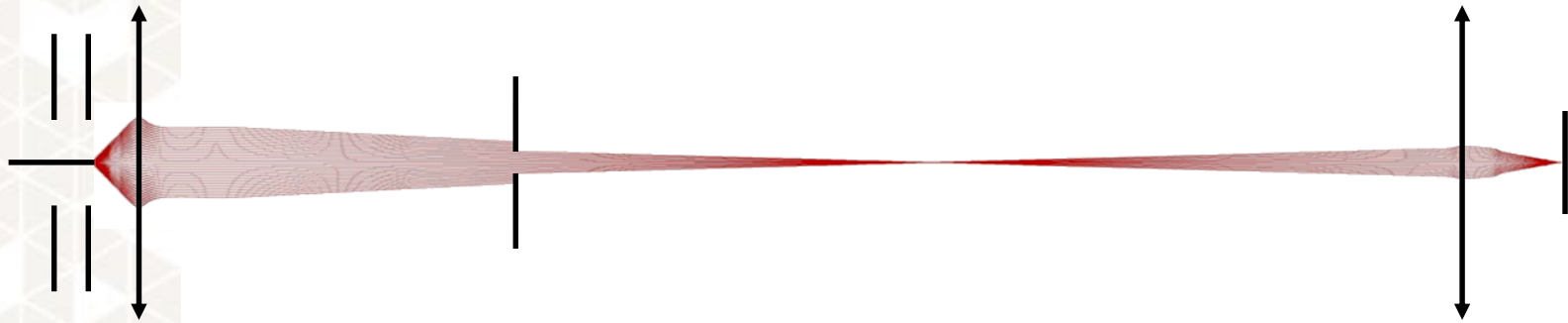
$$K_{\text{Ga}^+} = 2.37 \cdot 10^3 \text{ nm}^3/\text{pA}$$

$$K_{\text{Au}^{2+}} = 19.7 \cdot 10^3 \text{ nm}^3/\text{pA}$$

Au cross-section > Ga cross-section

Ion-beam lithography

FIB patterning: focus



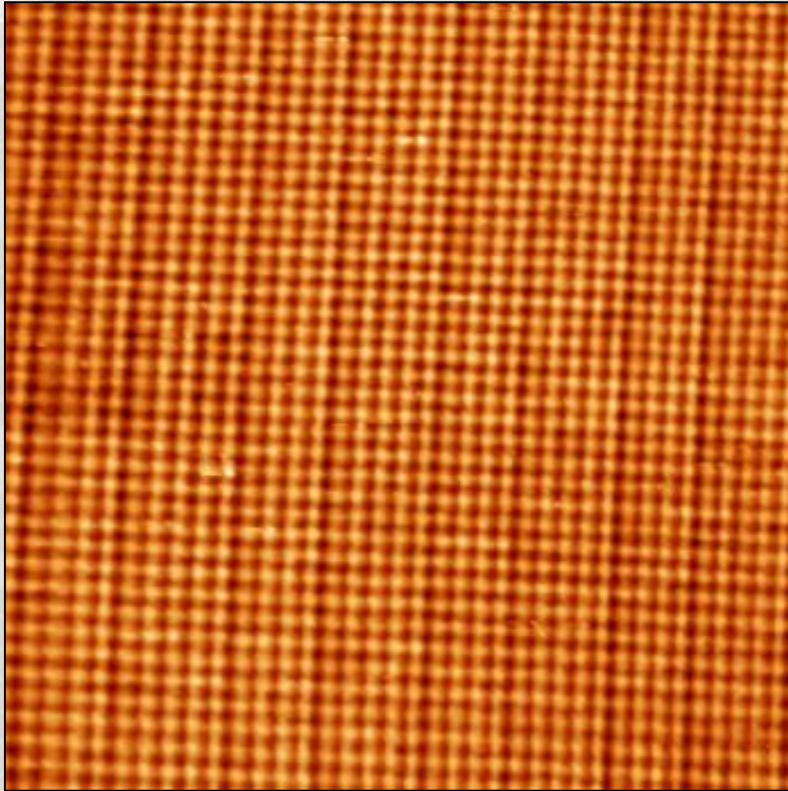
Collective ions interaction during milling process

By defocusing, local dose decrease:
amorphization and swelling before excavation

$$\frac{v_0}{v} = 3$$

Ion-beam lithography

Ion lithography optimized parameter



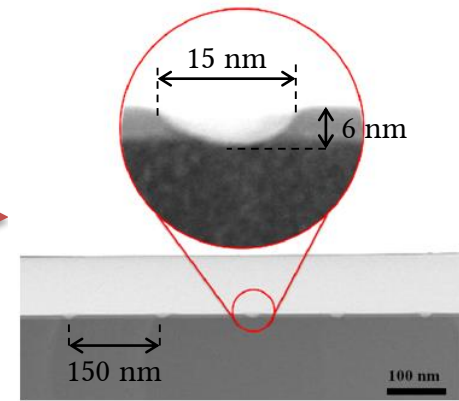
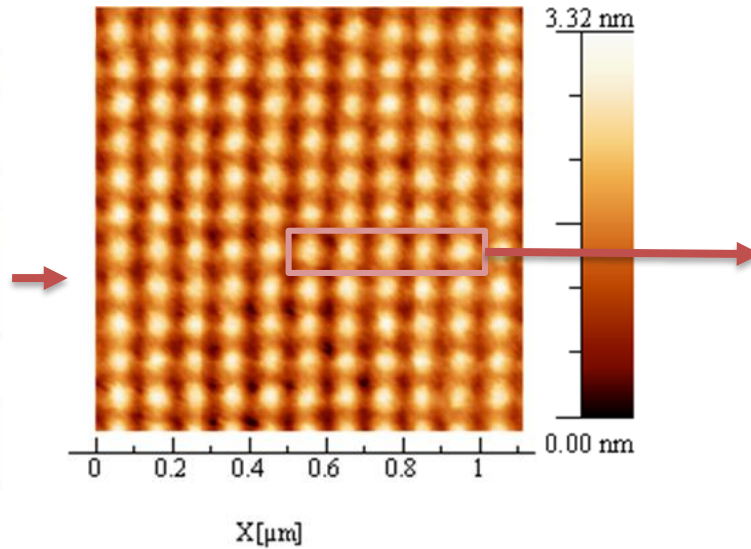
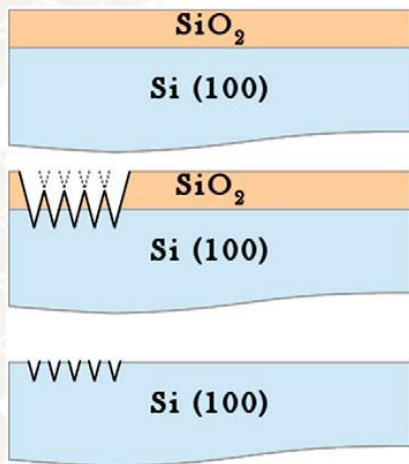
Using optimized experimental parameters:

- $\lambda = 24 \text{ nm}$
- $\varnothing = 15 \text{ nm}$
- $D \approx 2 \cdot 10^{11} \text{ cm}^{-2}$

Ga⁺ or Au²⁺:
Ultimate hole diameter: 15 nm

Ion-beam lithography

Ultimate FIB patterning: sacrificial ultra-thin oxide film



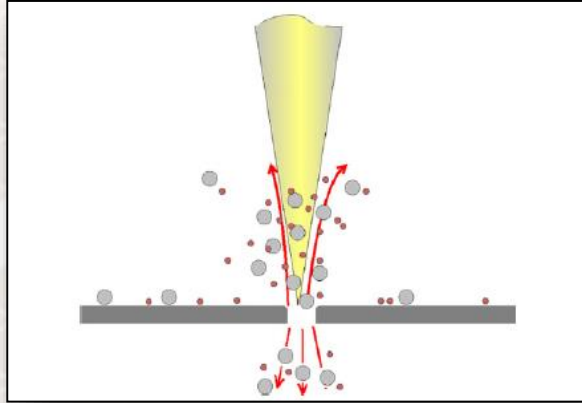
Ions	Ga ⁺
Acceleration voltage	30 kV
Beam current	2.4 μA
Probe current	1-6 pA

Reproducible depth penetration of 1 nm

Ion-beam lithography

FIB patterning: creating structures smaller than 5 nm

FIB processing method for ultra-thin membrane processing

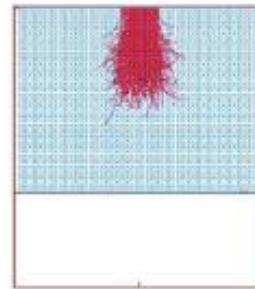


TRIM simulations

35 keV Ga⁺

SiC
membranes

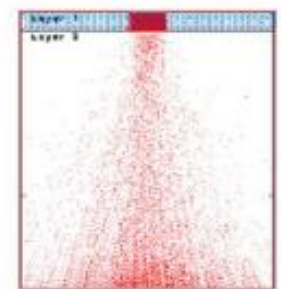
Membrane
thicknesses



100 nm

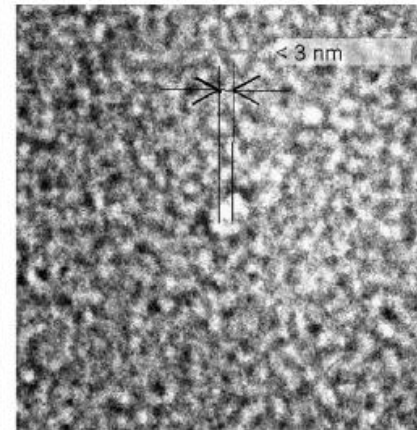
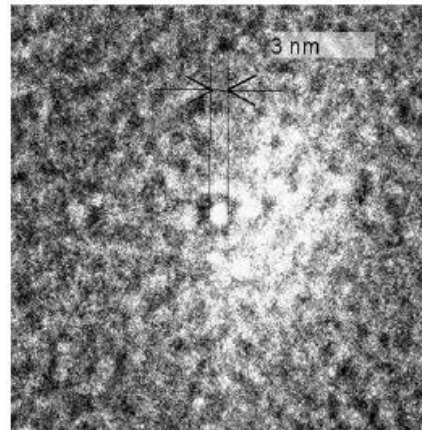


50 nm



10 nm

20 nm thick membrane
dose 10⁶ ions



50 x 50 nm TEM images

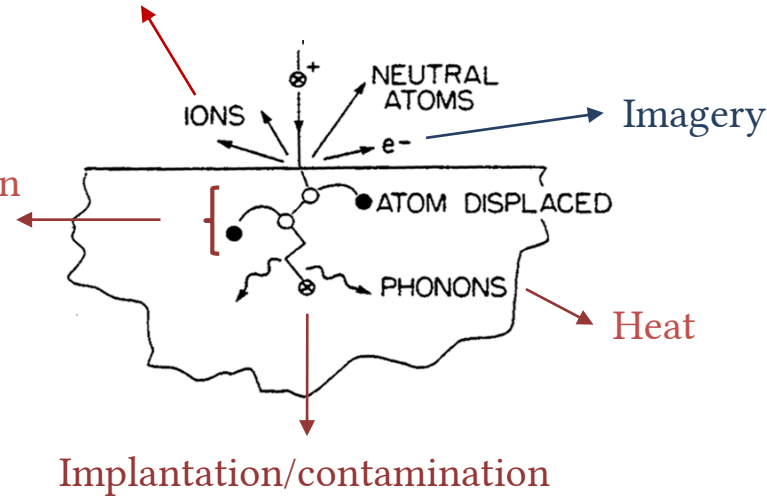
Ion-beam lithography

Ion/Matter interactions

- Lithography
- Analysis (SIMS)

Defects:

- Amorphisation
- Interstitials
- Vacancies

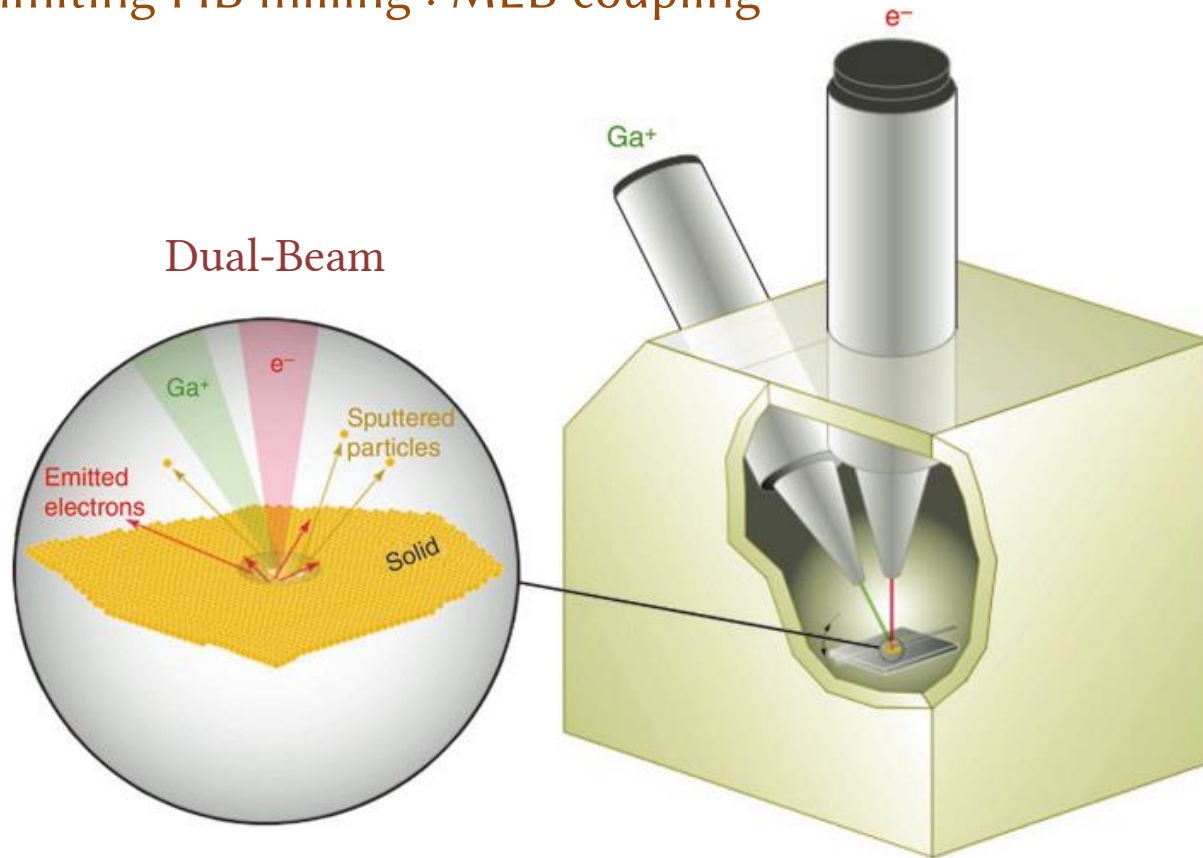


Ion cross section $>$ e^- cross section \Rightarrow Low ion diffusion

Probe size: Ion lithography $<$ e^- lithography

Ion-beam lithography

Limiting FIB milling : MEB coupling

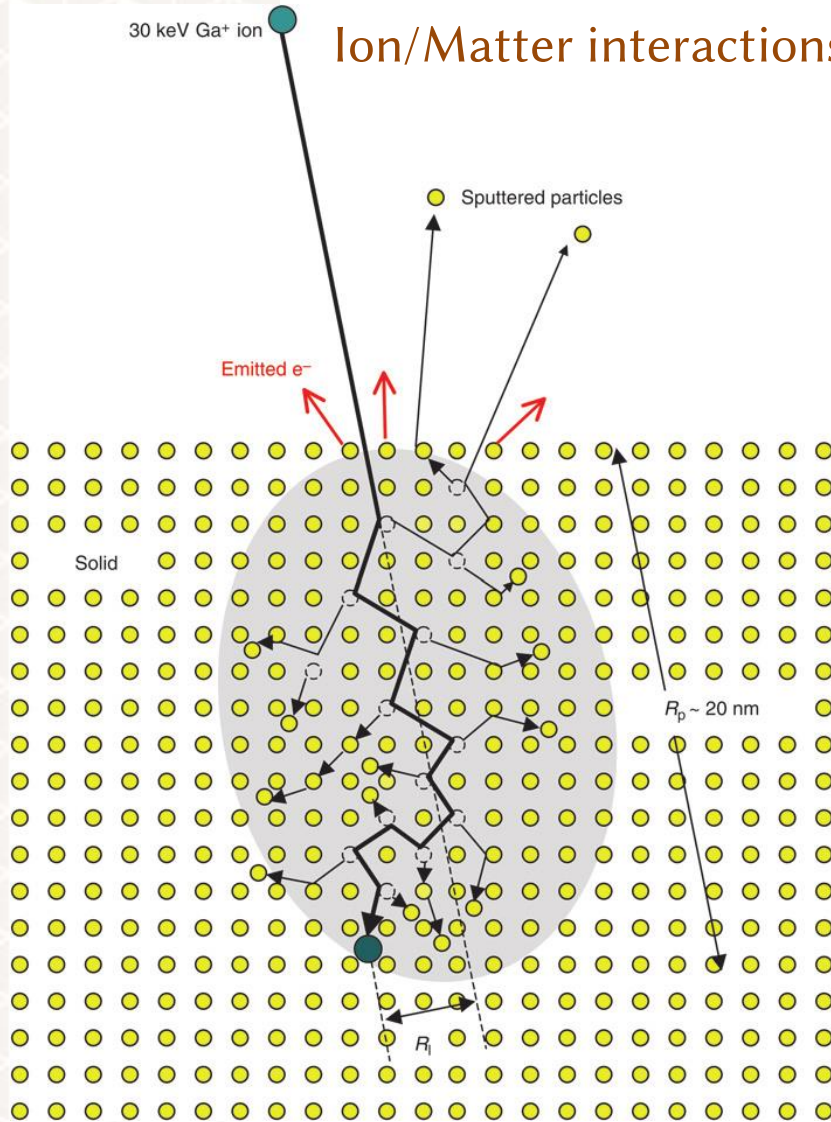


C.A.Volkert and A.M.Minor, MRS Bull., 32, 389 (2007)

Imaging without milling: dual beam

Ion-beam lithography

Ion/Matter interactions: 3 regimes



C.A.Volkert and A.M.Minor, *MRS Bull.*, **32**, 389 (2007)

Main parameters:

- incident ion energy (E_0)
 - atomic masses (M_1 and M_2)
 - atomic numbers (Z_1 and Z_2)
- (1): ion, (2) target atoms

• Regime I (knock-on regime):

$M_1 < M_2$ or E_0 is low, minimum sputtering

• Regime II (linear cascade regime), where FIB operates:

$M_1 \sim M_2$, E_0 is moderate, governed by nuclear effects

• Regime III (spike-on regime):

$M_1 > M_2$ and/or E_0 is high, majority of atoms move in collision cascade

For typical FIB usage:

$$R = \frac{6E_0(\text{keV})}{\rho(\text{g cm}^{-3})} \frac{M_2}{Z_2} \frac{M_1 + M_2}{M_1} \frac{Z_1^{\frac{2}{3}} + Z_2^{\frac{2}{3}}}{Z_1}$$

Ion-beam lithography

Ion/Matter interactions

On first approximation

$$\nu(E) = \begin{cases} 0 & E < E_d \\ 1 & E_d \leq E < 2,5E_d \\ \frac{k(E)E_a}{2E_d} & 2,5E_d \leq E \end{cases}$$

E : Primary Knock-on Atom (PKA)

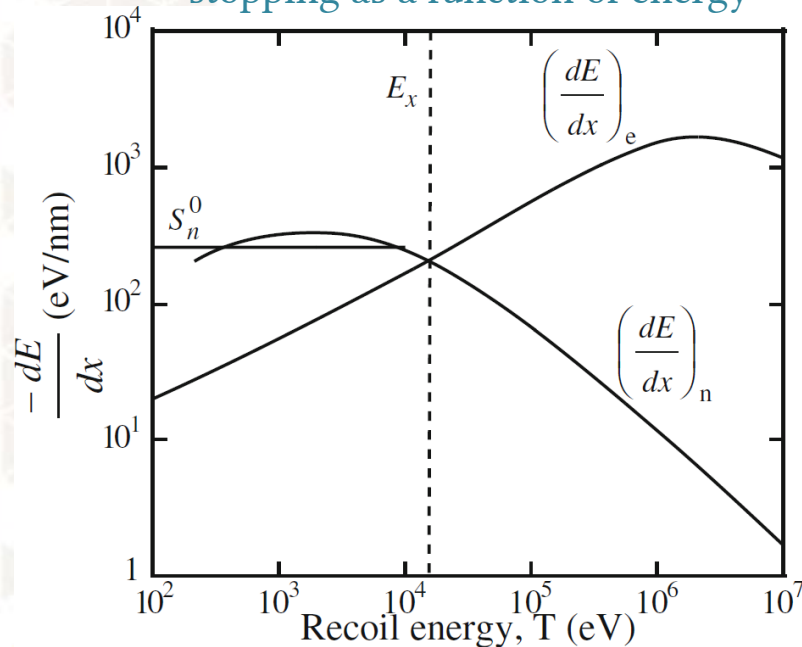
$\nu(E)$: atomic displacements

$\kappa(E) \approx 0.8$

E_d : displacement energy

E_a : damage energy

Energy loss from electronic and nuclear stopping as a function of energy



Dominant mechanism for FIB process (1-50 keV):
Nuclear stopping

G.H. Kinchin and R.S. Pease,
"The Displacement of Atoms in Solids by Radiation,"
Reports on Progress in Physics, vol. 18, pp. 1-51, 1955.

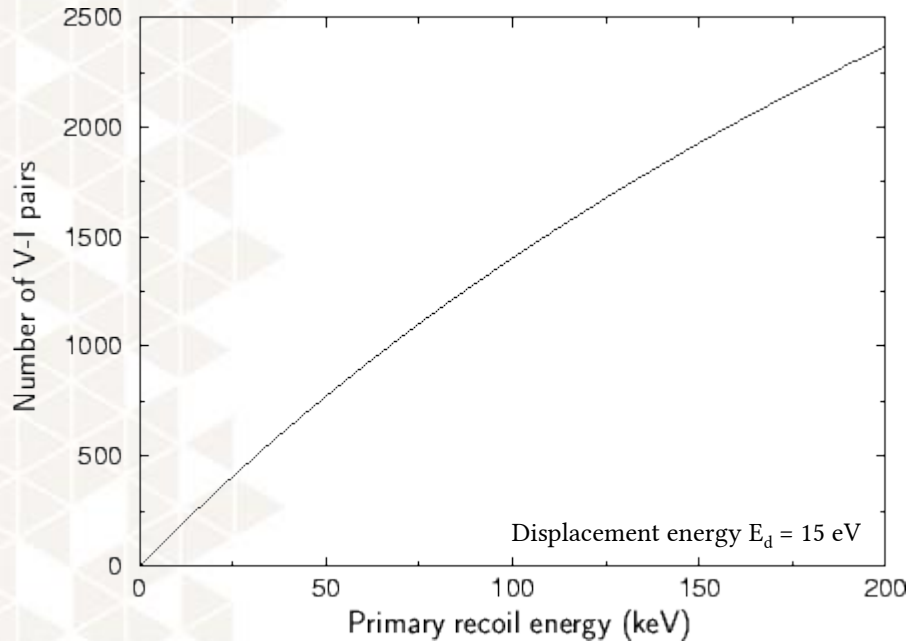
Ion-beam lithography

Ion/Matter interactions

Stopping power

$$S(E) = 2\pi \frac{4M_1M_2}{(M_1 + M_2)^2} E_0 \int_0^{p_{\max}} \sin^2 \frac{\theta}{2} p dp$$

Number of silicon vacancies and interstitials generated by the primary recoil

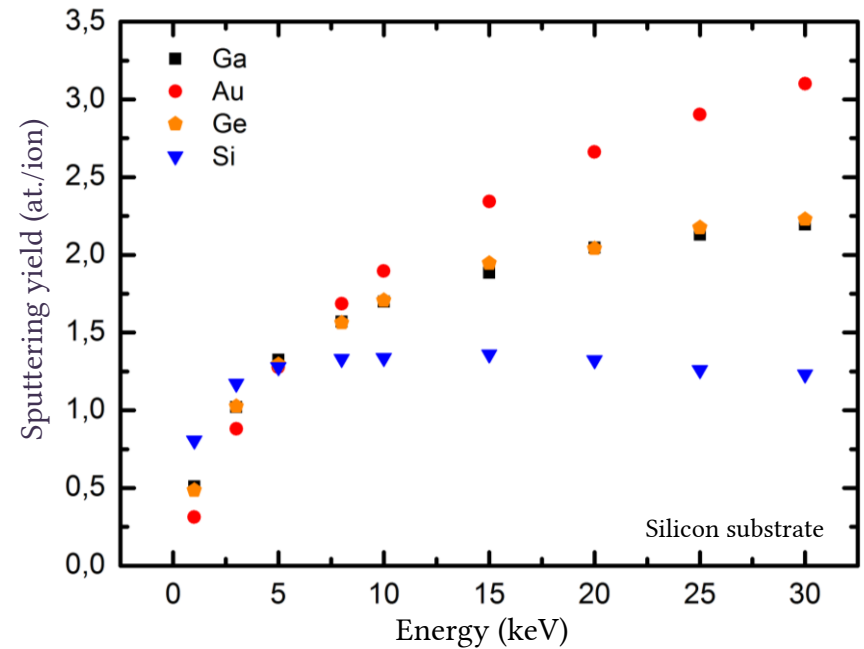


M. T. Robinson, *Defects and radiation damage in metals.*,
Cambridge University Press, Cambridge (1969)

Sputtering yield

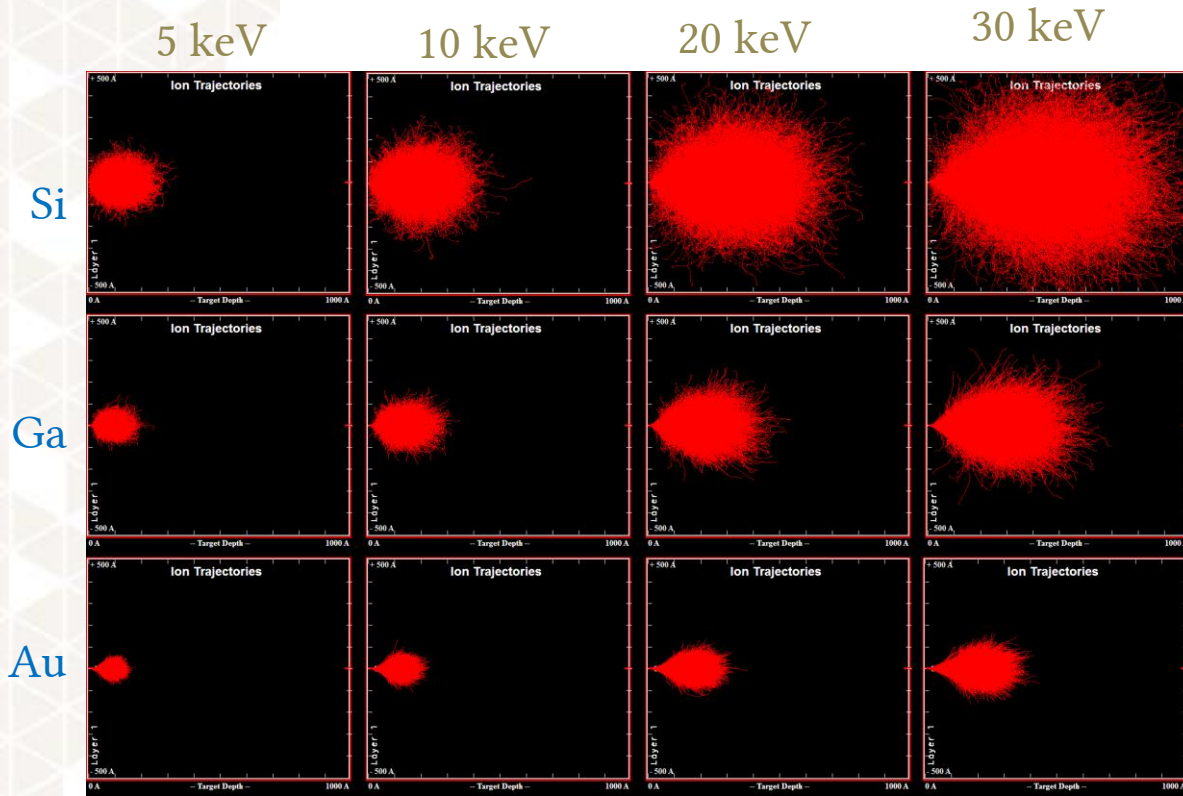
$$Y(E_0) \approx \frac{1}{4} \Gamma_m \frac{\alpha N S_n(E) \Delta x_n}{E_0}$$

Sputtering yields for typical FIB energies
 $10^{-1} < Y < 10^2$



Ion-beam lithography

Ion/Matter interactions



Box: 100 x 100 nm

Ion	Max. depth (nm)	Max. width (nm)
Si	90	70
Ga	60	36
Au	40	20

Ion energy: 30 keV

Ion-beam lithography

Ion/Matter interactions

Principles

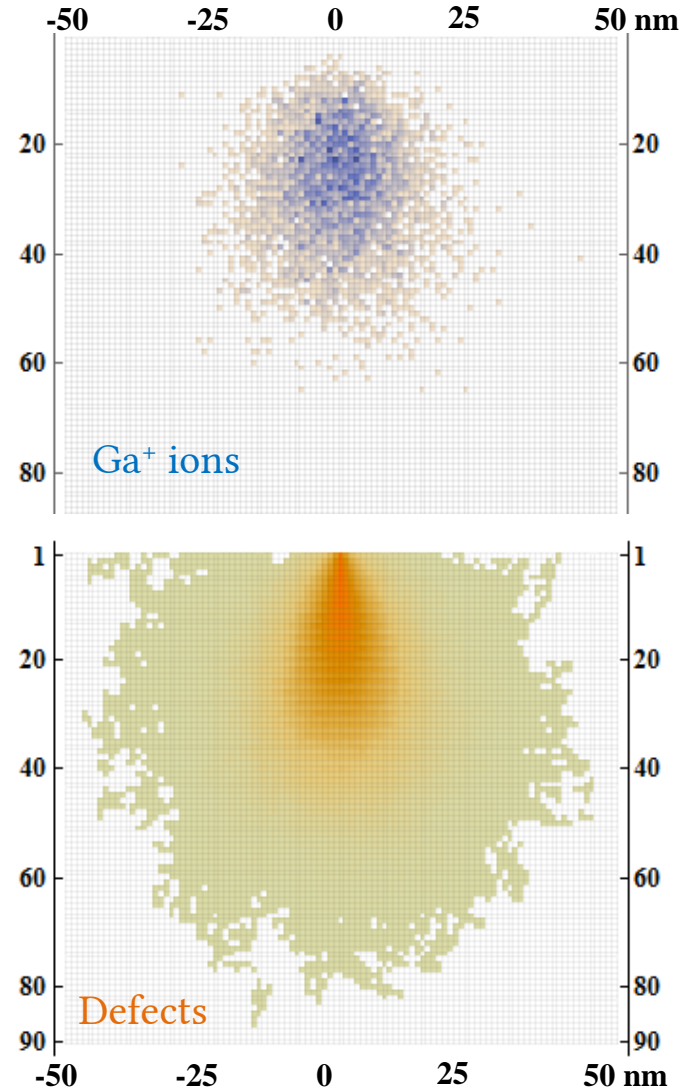
Monte Carlo simulations:

- Incident ion trajectories
- Recoil atoms trajectories
- Phonons
- Nuclear and electronic energy loss

Drawbacks

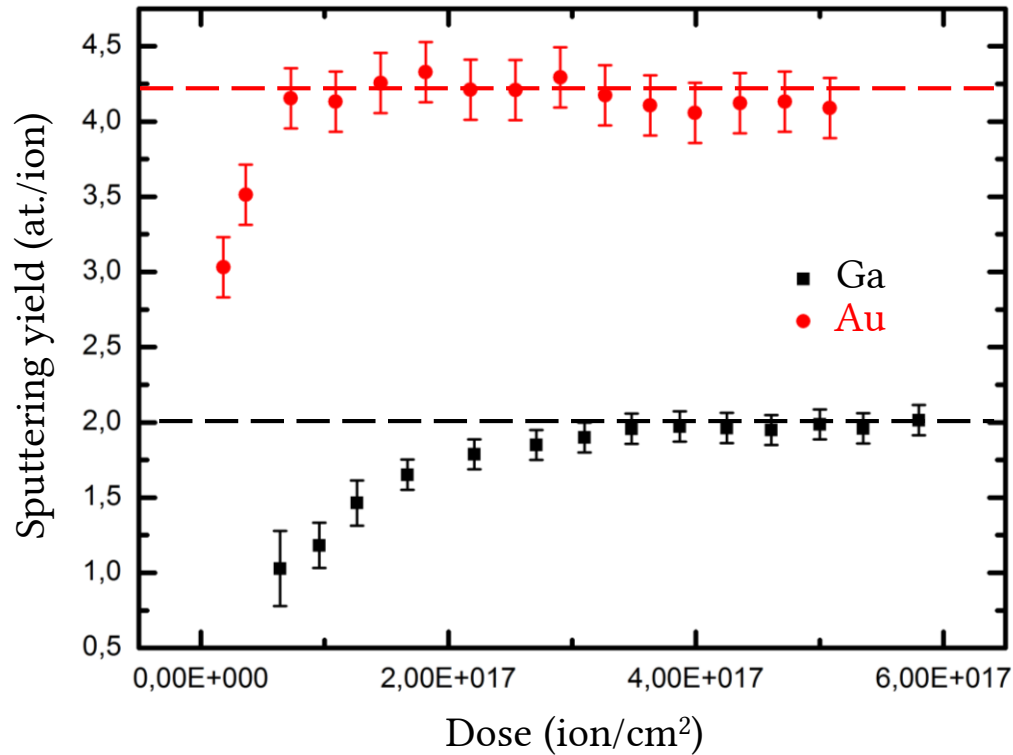
- No crystalline orientation (channeling)
- No defects accumulation
- Punctual probe

SRIM - Ga⁺ - 30 kV



Ion-beam lithography

Ion/Matter interactions



Ion	Simulations (at./ion)	Experiments (at./ion)
Ga ⁺	2,2	2
Au ⁺	3,1	4,2

Ion-beam lithography

Ion/Matter interactions

- **Amorphization** of sample surface
- Ion Implantation: incident atoms remain in the sample target
 - **Dopping** effect
 - May reach critical composition for **second phase formation**
- Lattice defects
 - **Vacancies** – displaced or “missing” atoms from their equilibrium lattice positions
 - **Interstitials** – atoms which are positioned in between equilibrium lattice positions
 - **Dislocations** – a missing “half-plane” of atoms
- **Local heating** due to large displacement of atoms that may occur within the collision cascade (10's of nanometers from surface)

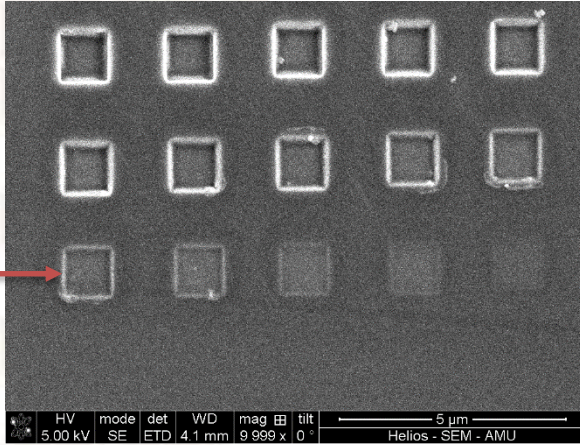
Example: Ga⁺ ion lithography

- Concentration of primary defects (knock-outs from lattice sites): average 1000 defects per ion
- Ga in most semiconductors is acceptor, affecting electronic, optical, magnetic and thermal properties
- Concentration of Ga in the irradiated zone can be given by: $C_{\text{Ga}} = 1/(1+\gamma)$, where γ is sputter yield

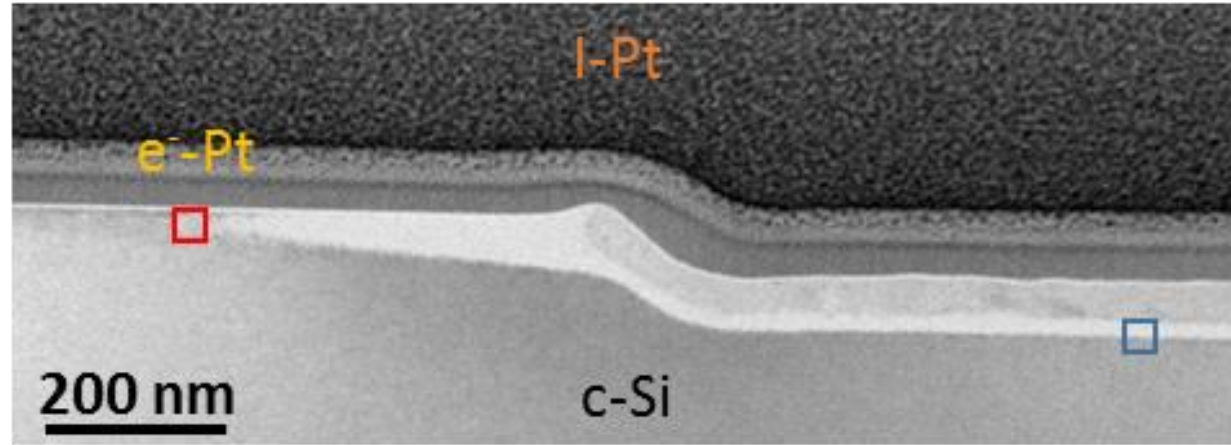
Ion-beam lithography

Square patterns: Ga⁺ (30 keV) on Si substrate

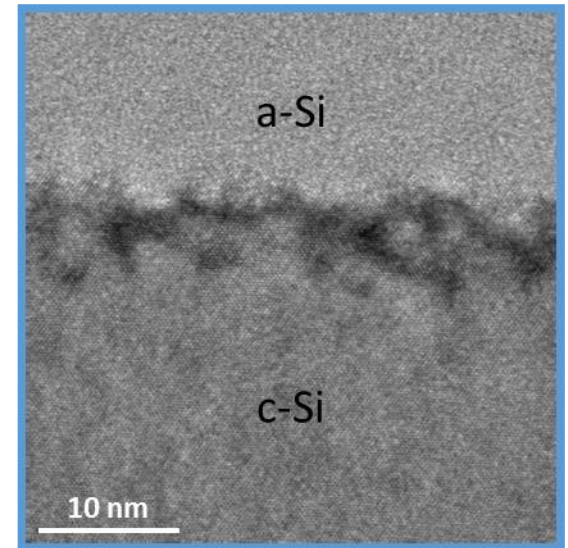
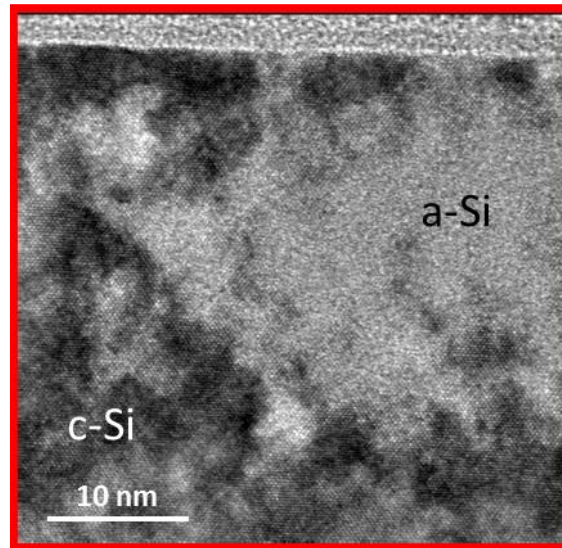
$D = 3 \cdot 10^{17}$ at./cm²



$D = 3 \cdot 10^{16} - 5 \cdot 10^{17}$ at./cm²



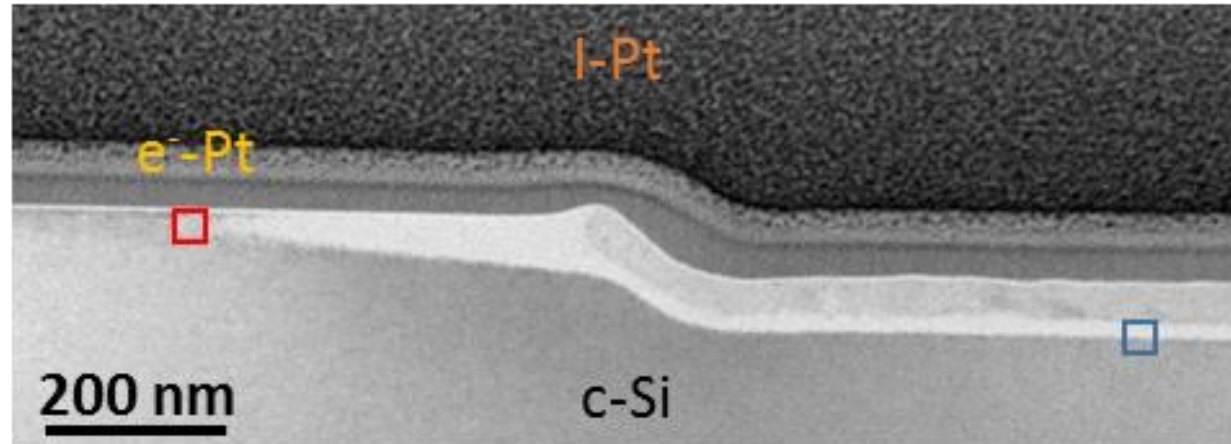
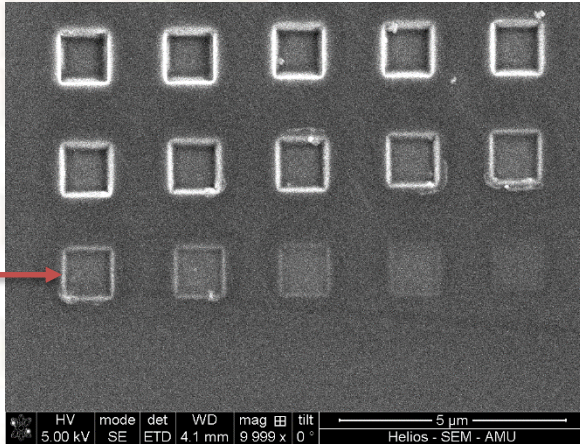
Structural
characterization



Ion-beam lithography

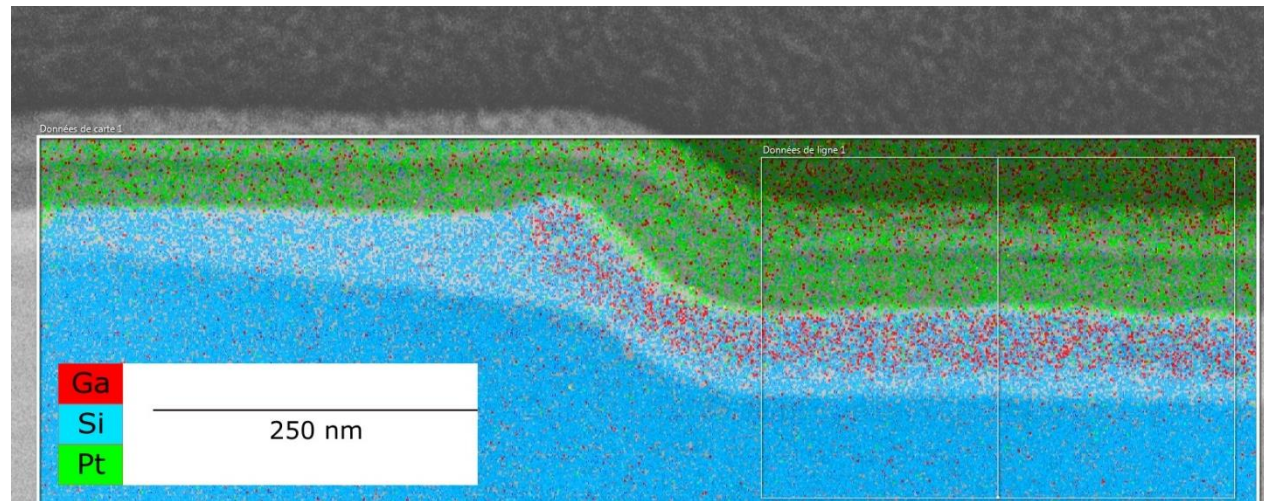
Square patterns: Ga⁺ (30 keV) on Si substrate

$D = 3 \cdot 10^{17}$ at./cm²



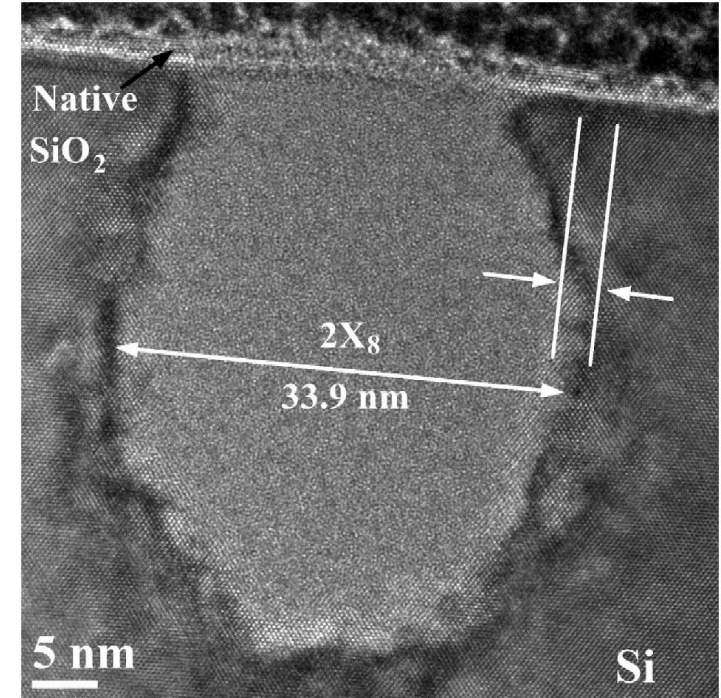
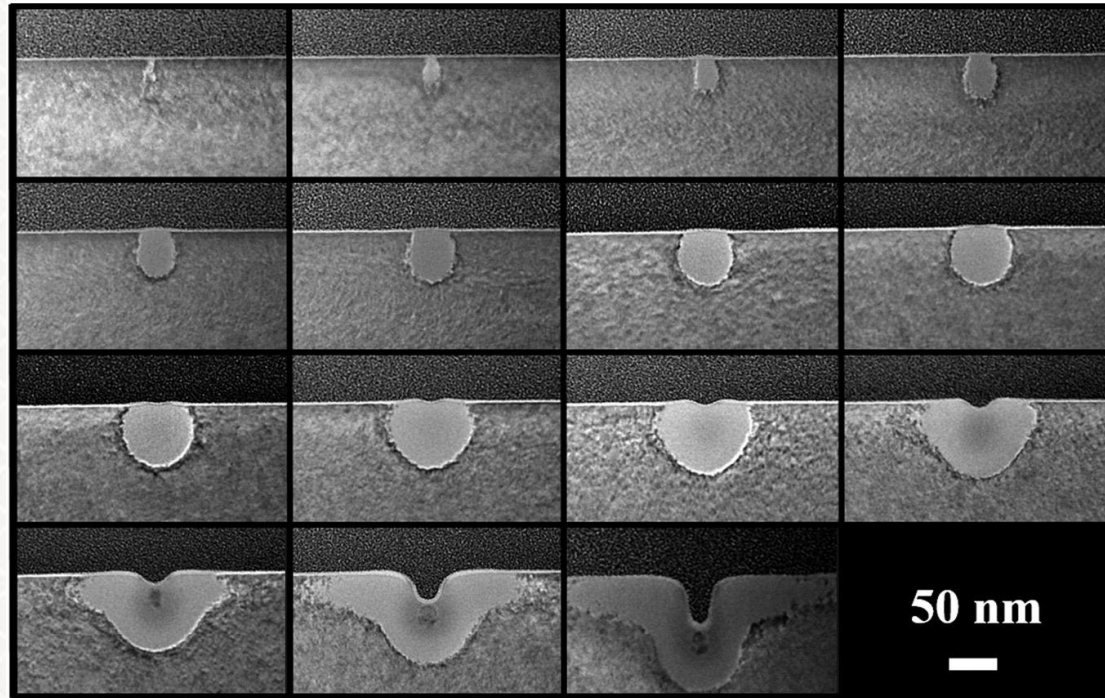
$D = 3 \cdot 10^{16} - 5 \cdot 10^{17}$ at./cm²

Caractérisation chimique (EDX)



Ion-beam lithography

Ion/Matter interactions

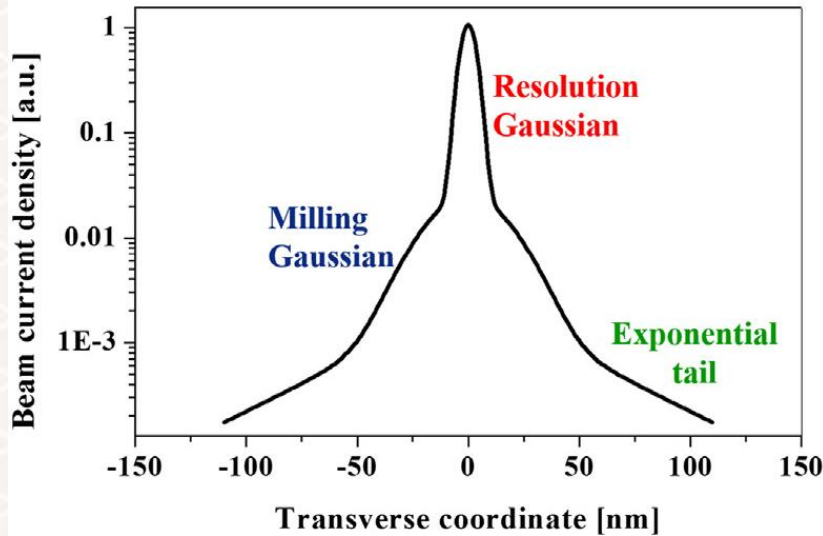


$$D = 1.1 \cdot 10^{-9} \text{ C/m to } 1.8 \cdot 10^{-5} \text{ C/m}$$

Y. Greenzweig *et al.*, *Microelectronic Engineering*, 155, 19 (2016)

Ion-beam lithography

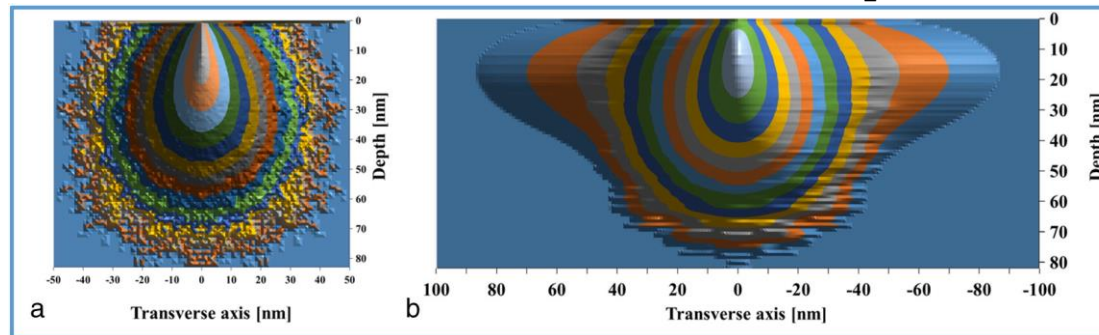
Ion/Matter interactions



$$J_B(x) = \frac{I_1}{\sqrt{2\pi a_1^2}} \exp\left(-\frac{x^2}{2a_1^2}\right) + \frac{I_2}{\sqrt{2\pi a_2^2}} \exp\left(-\frac{x^2}{2a_2^2}\right) + \frac{I_3}{2a_3} \exp\left(-\frac{|x|}{a_3}\right)$$

SRIM

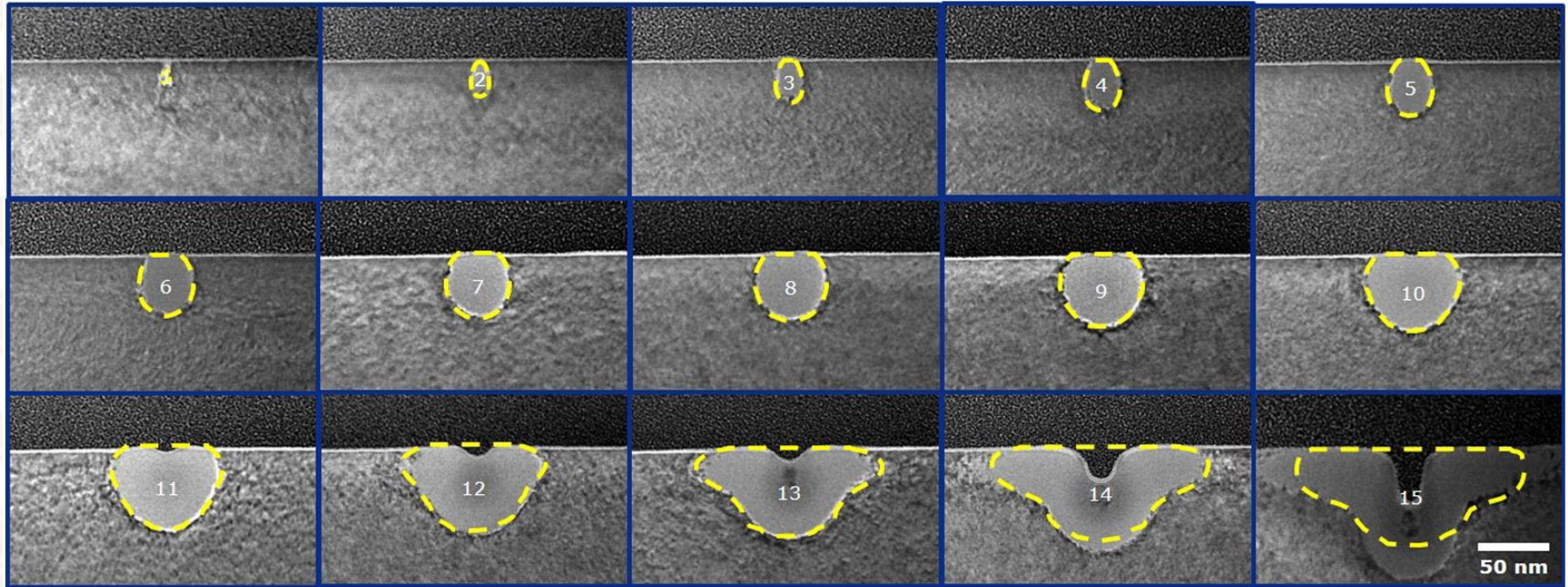
SRIM \otimes Probe profil



Y. Greenzweig *et al.*, *Microelectronic Engineering*, 155, 19 (2016)

Ion-beam lithography

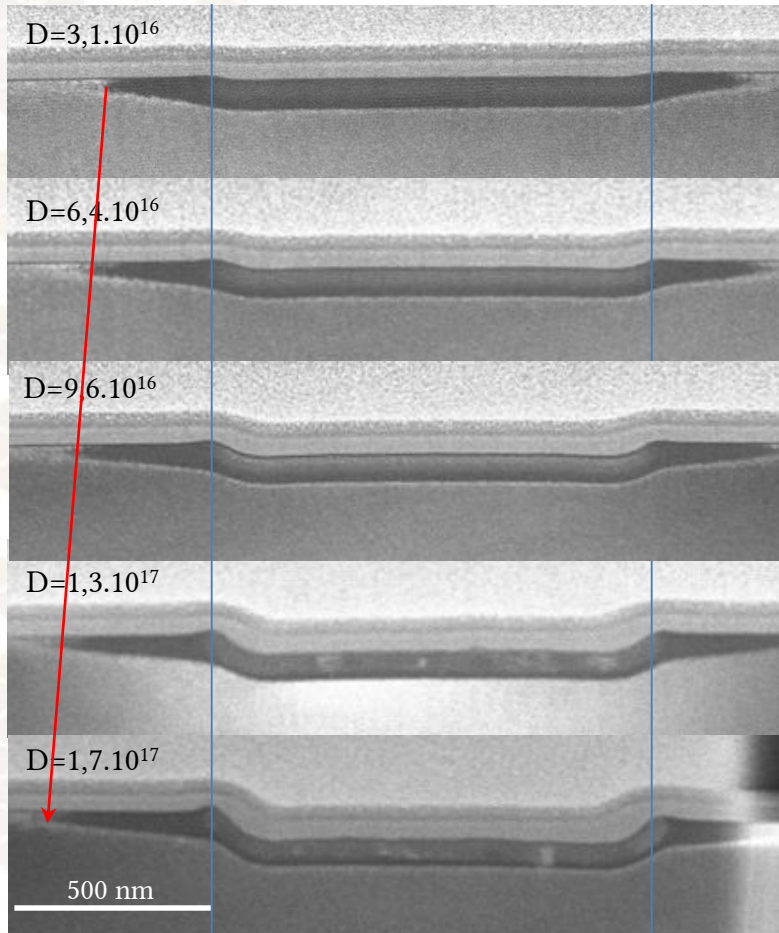
Ion/Matter interactions



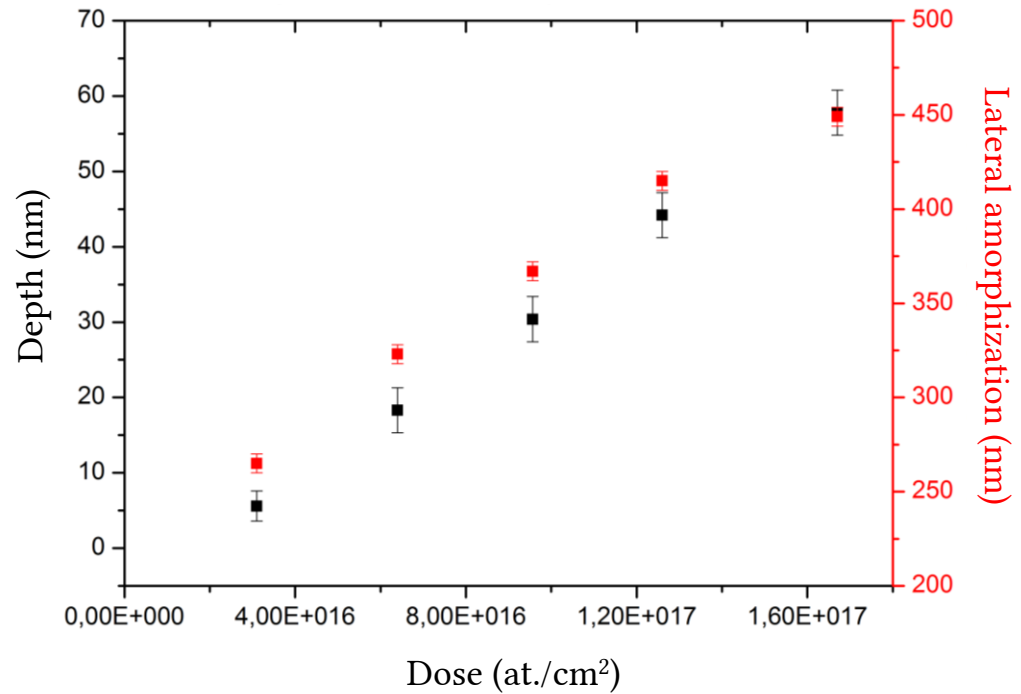
Y. Greenzweig et al., *Microelectronic Engineering*, 155, 19 (2016)

Ion-beam lithography

Square patterns: Ga⁺ (30 keV) on Si substrate



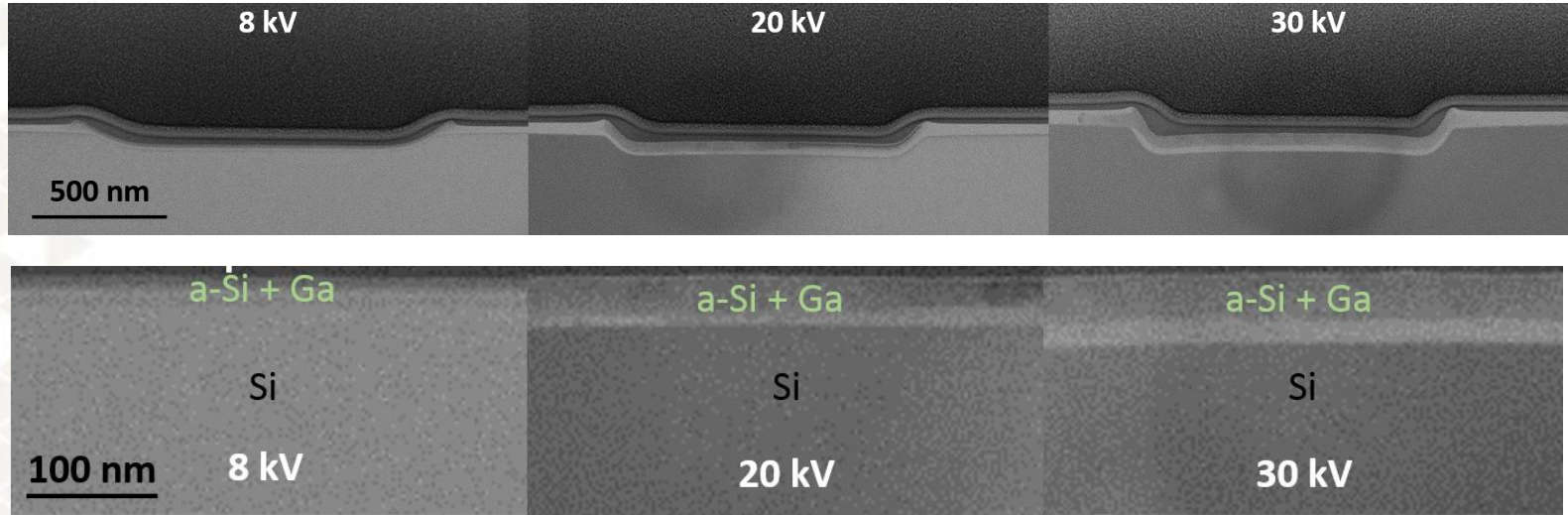
$D = 3 \cdot 10^{17}$ at./cm²



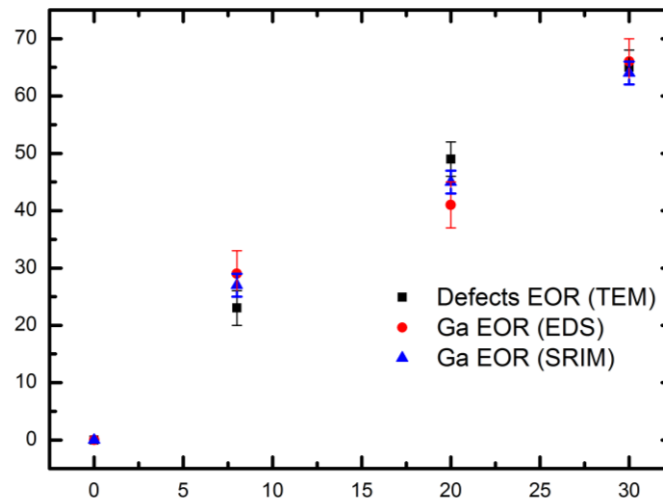
Depth and width of amorphized layer proportional to ion dose

Ion-beam lithography

Square patterns: Ga⁺ (8, 20 and 30 keV) on Si substrate



$D = 3 \cdot 10^{17}$ at./cm²



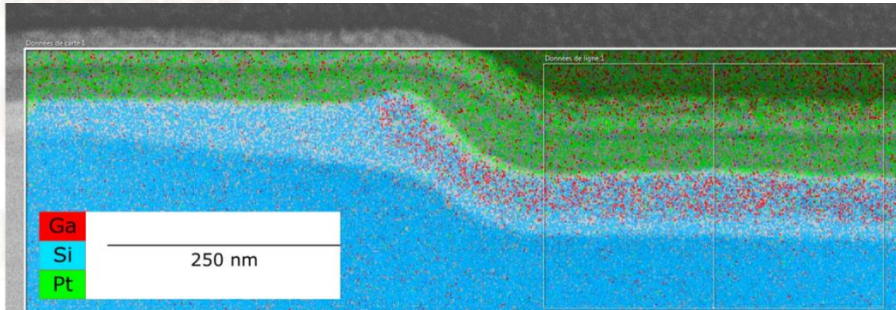
Working at low energy

Ion-beam lithography: defects healing

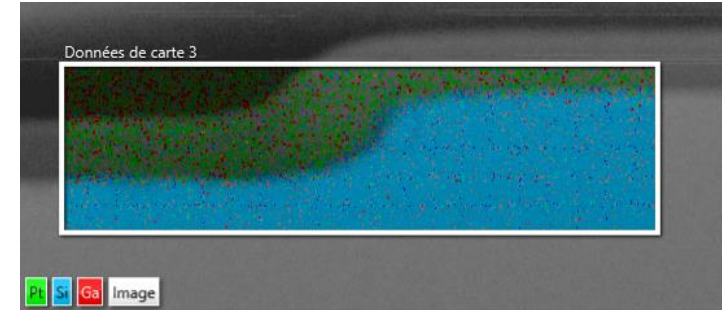
Square patterns: Ga⁺ (8, 20 and 30 keV) on Si substrate

Defects healing by annealing

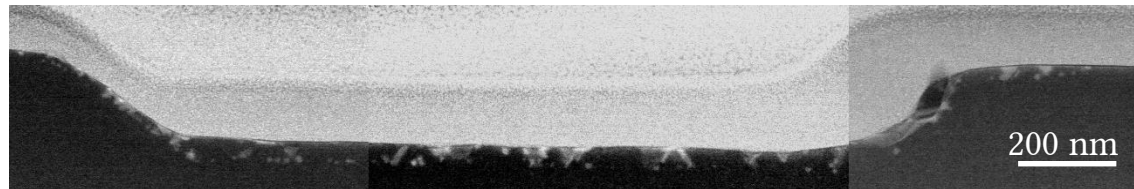
Before



After



600°C / 10 min. then 850°C / 5 min



No gallium contamination detectable: Ga diffusion

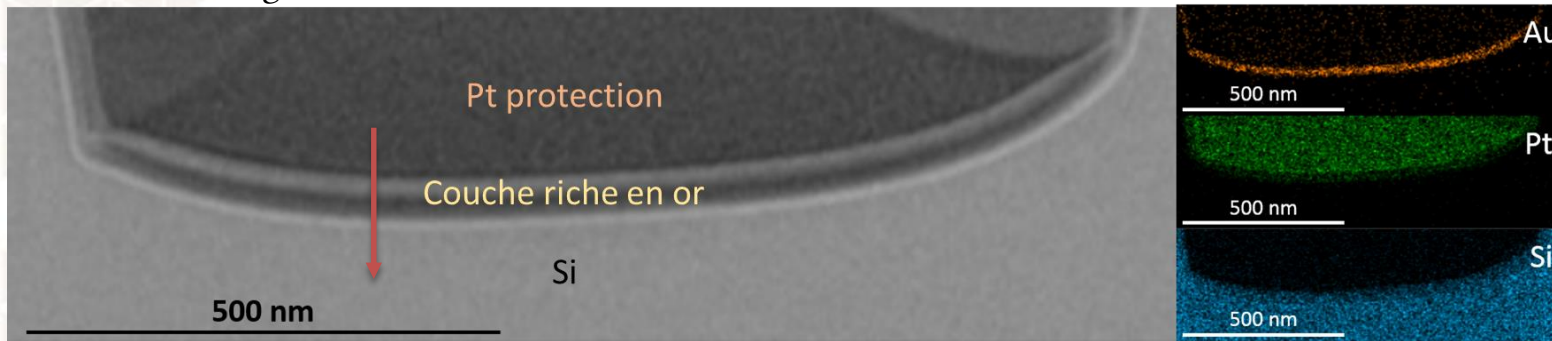
Lot of structural defects: stacking faults, dislocations

Ion-beam lithography: defects healing

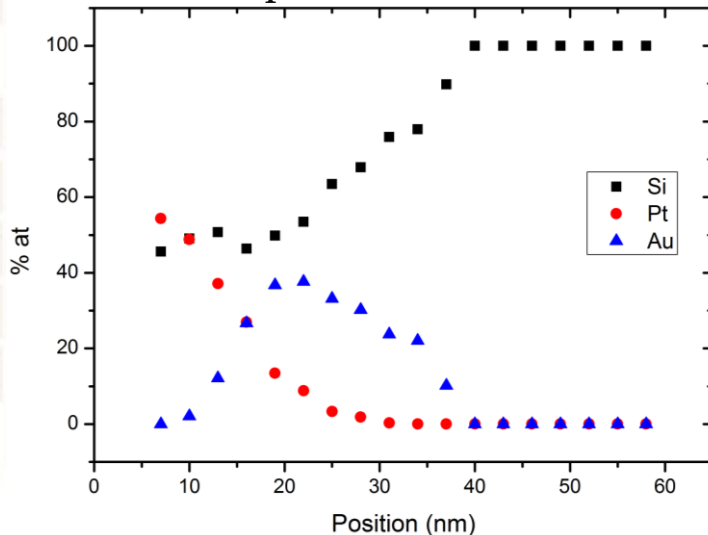
Square patterns: Au⁺ (8, 20 and 30 keV) on Si substrate

Defects healing by annealing

Before annealing



EDX line profile



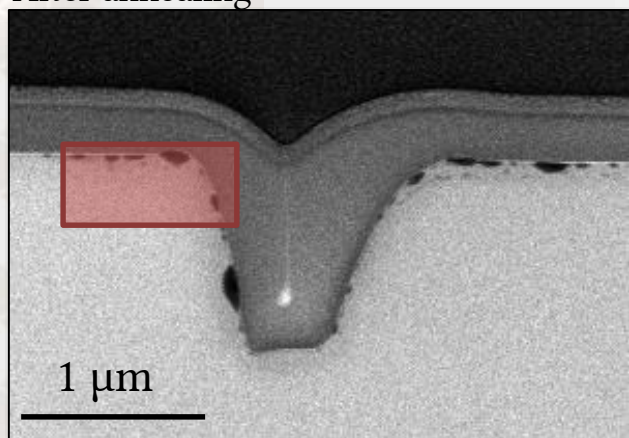
Gold-silicon alloy layer

Ion-beam lithography: defects healing

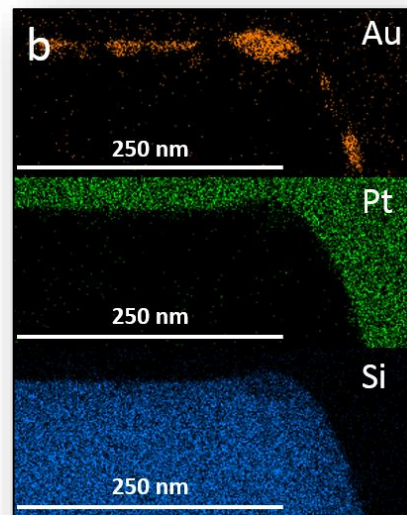
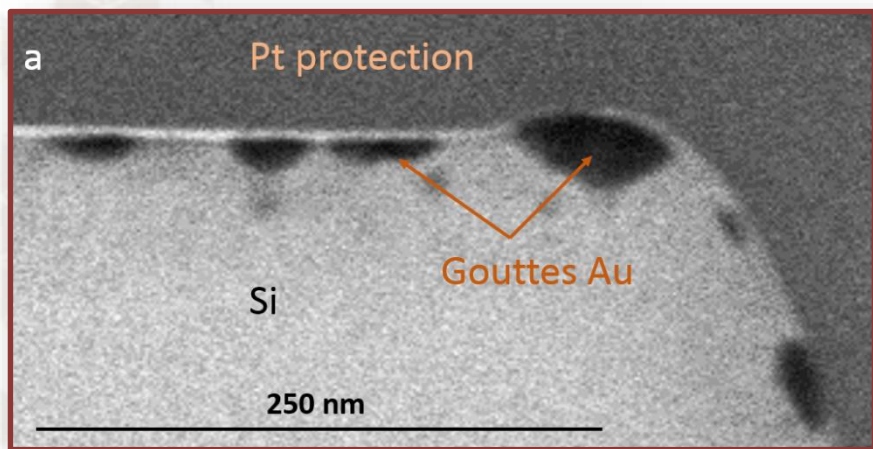
Square patterns: Au⁺ (8, 20 and 30 keV) on Si substrate

Defects healing by annealing

After annealing



600°C / 10 min. then 850°C / 5 min



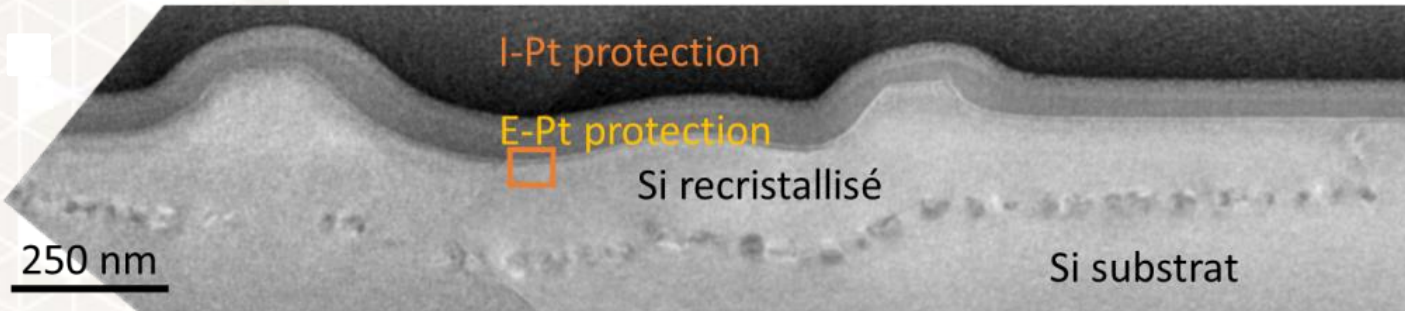
Gold segregation

Ion-beam lithography: defects healing

Square patterns: Si⁺ (8, 20 and 30 keV) on Si substrate

Defects healing by annealing

After annealing

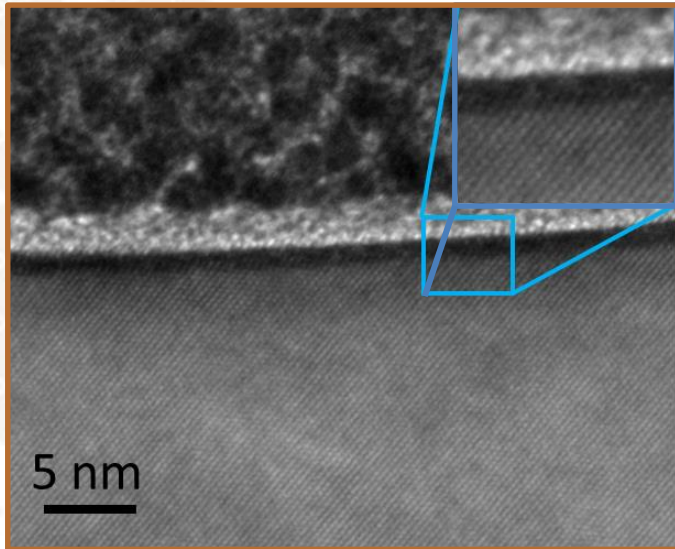


600°C / 10 min. then 850°C / 5 min

Si in Si: no contamination

Fully recrystallized lattice

End of Range defects (interstitials atoms):
thickness 40-70 nm



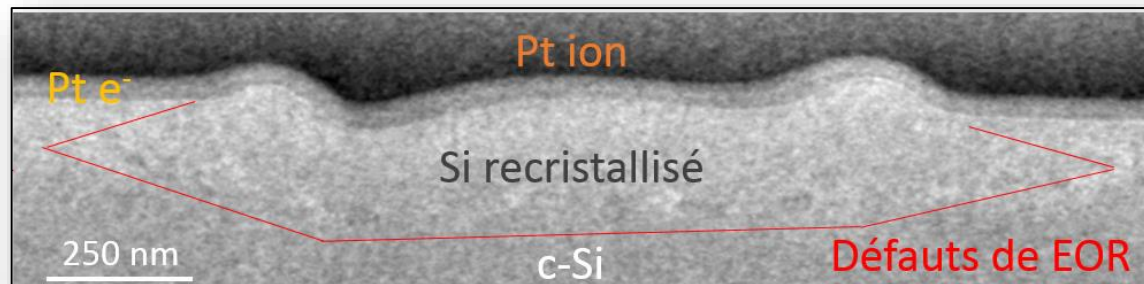
Ion-beam lithography: defects healing

Square patterns: Si^+ (8, 20 and 30 keV) on Si substrate

Defects healing by annealing

Previous recipe 600°C / 10 min. then 850°C / 5 min

New recipe 700°C / 30 min



Reduction of End of Range defects (thickness 10-20 nm)

Synopsis

Introduction

- Usual Si cleaning process
- Bottom-up is not, yet, efficient

Ion beam lithography

- Fast review of lithography technics
- Focused Ion Beam principles
- High resolution
- Limits: surface and volume defects

Using Focus Ion Beam for surface preparation

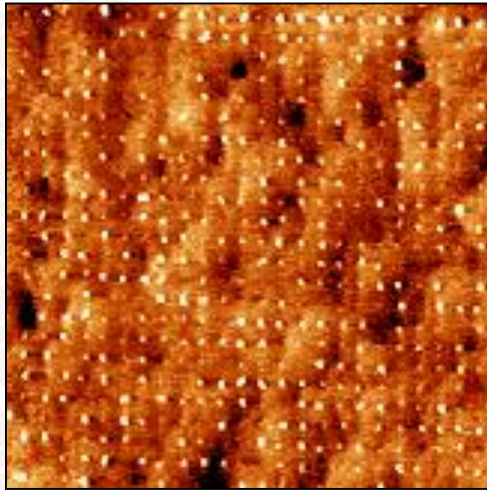
- Gold dewetting
- Au dots
- Silicon dewetting

Conclusion

Ion-beam lithography: Ge dots

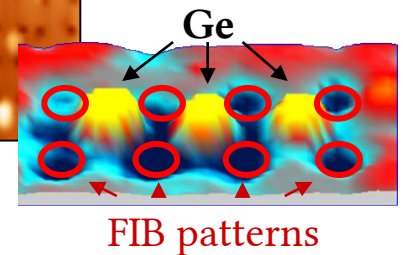
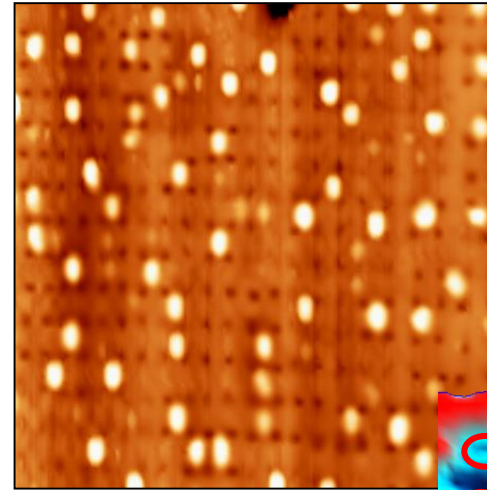
8 Ge monolayers growth on FIB patterned Si(100) substrate

550°C: metastable state



$d = 50 \text{ nm}$
 $D = 4 \cdot 10^{10} / \text{cm}^2$

750°C: stable state



⇒ Scattering controlled diffusion

$$k(E, T) = k_0 \exp\left(-\frac{E}{k_B T}\right) \frac{E_{D1}}{E_{D2}}$$

$$E = E_D(x, y) + nE_N \quad E_{D1}(x, y) > E_{D2}(x, y)$$

Scattering barrier higher inside holes (E_{D1})
 than on terrace (E_{D2})

⇒ Preferential nucleation inside patterned holes

$$k_1(E_1, T) = k_2(E_2, T) \exp\left(-\frac{E_{D1} - E_{D2}}{k_B T}\right)$$

Scattering barriers negligible at high temperature

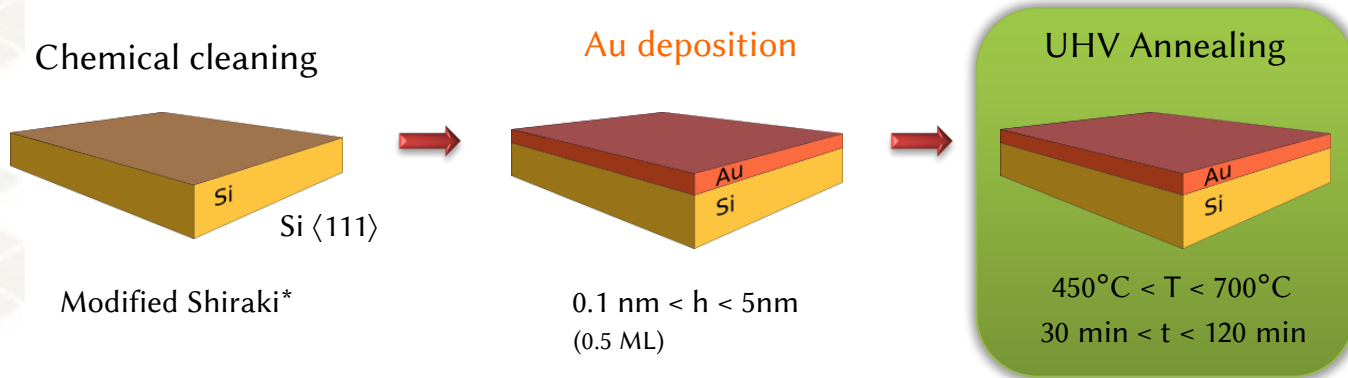
⇒ Nucleation controlled by elastic energy

⇒ Preferential nucleation on terraces

Pascale *et al.* Phys. Rev. B 77, 075311 (2008)

Ion-beam lithography: gold dewetting

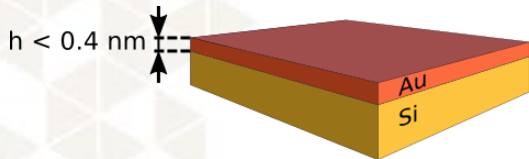
Gold Dewetting process



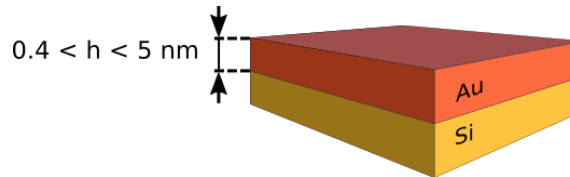
- * Modified Shiraki:
1. $\text{HNO}_3(65\%) / 70^\circ\text{C} / 10 \text{ min}$
 2. $\text{H}_2\text{O}(\text{DI}) / \text{RT} / 1 \text{ min}$
 3. $\text{HF}(49\%) : \text{H}_2\text{O} (1:10) / \text{RT} / 30 \text{ s}$

Dewetting process depends on Au layer thickness h

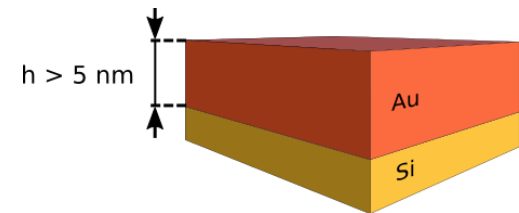
I. $h_{\text{Au}} \leq 0.4 \text{ nm}$



II. $0.4 \leq h_{\text{Au}} \leq 5 \text{ nm}$



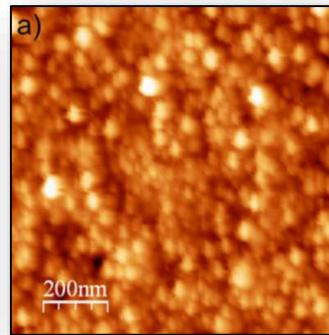
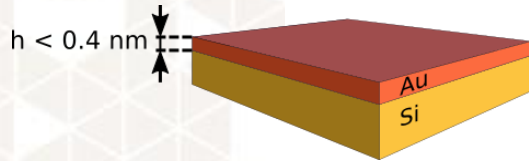
III. $h \geq 5 \text{ nm}$



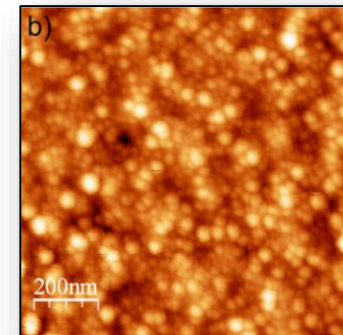
Ion-beam lithography: gold dewetting

Gold dewetting regimes

I. $h_{\text{Au}} \leq 0.4 \text{ nm}$



Annealing
($T = 550^\circ\text{C}$)



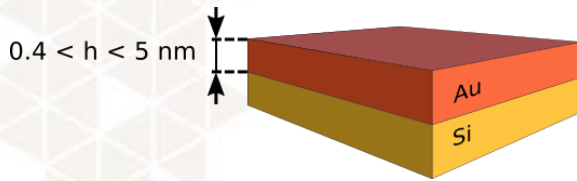
- Same RMS roughness: 0.5 nm
 - Close Au nanocrystalline mean grains size
- } no significant difference

Consistent with the standard morphology of ultra-thin Au films on Si(111):
nanocrystalline grains on top of 2D sub-ML structures

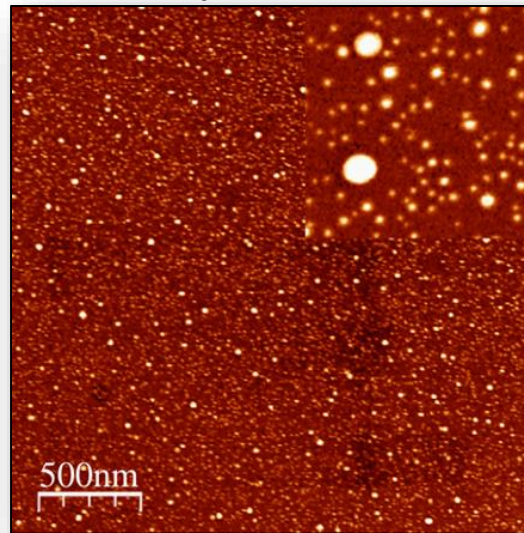
Ion-beam lithography: gold dewetting

Gold dewetting regimes

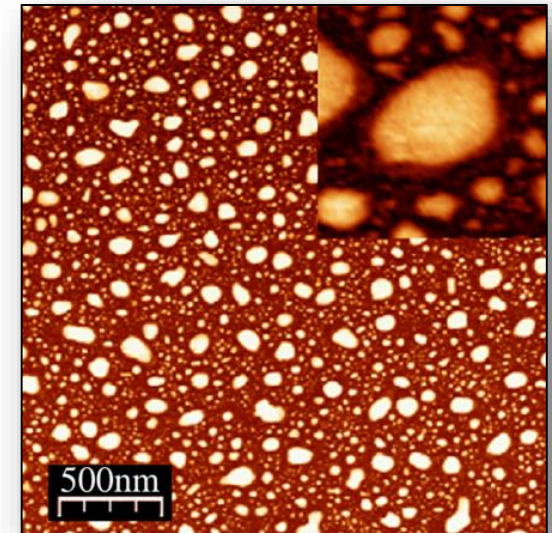
II. $0.4 \leq h_{\text{Au}} \leq 5 \text{ nm}$



$h_{\text{Au}} = 1 \text{ nm}$



$h_{\text{Au}} = 5 \text{ nm}$

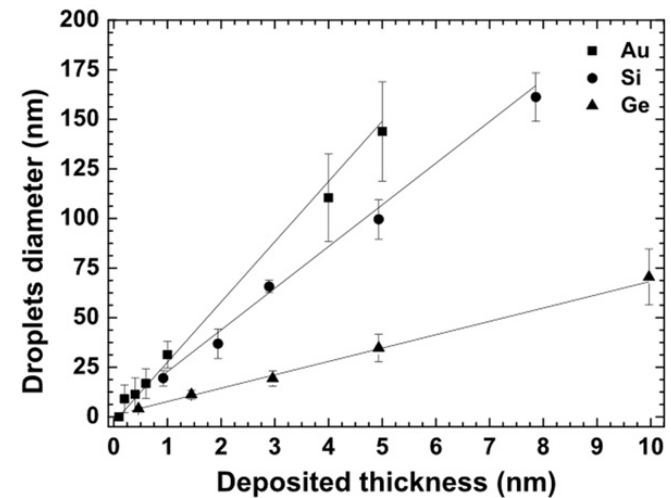
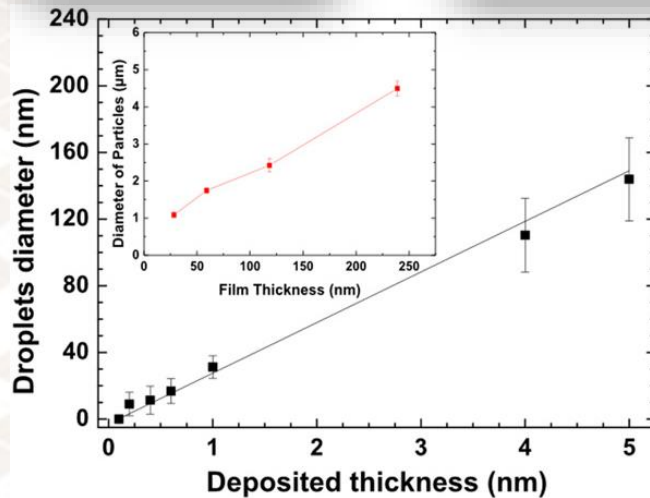
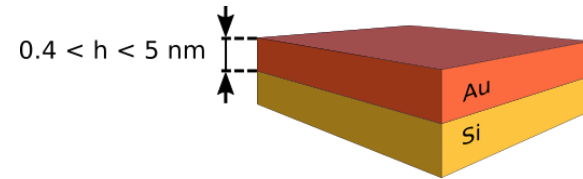
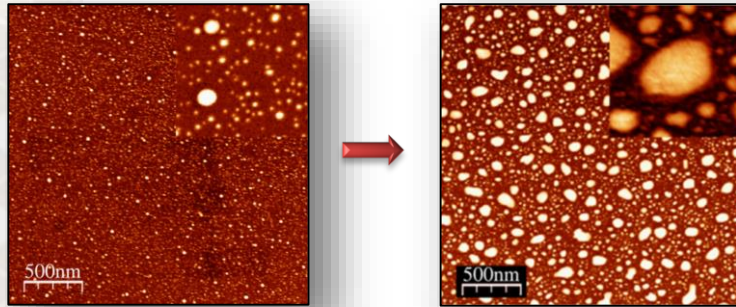


- Au droplets formation
- Bimodal distribution

Ion-beam lithography

Gold dewetting regimes

II. $0.4 \leq h_{Au} \leq 5 \text{ nm}$



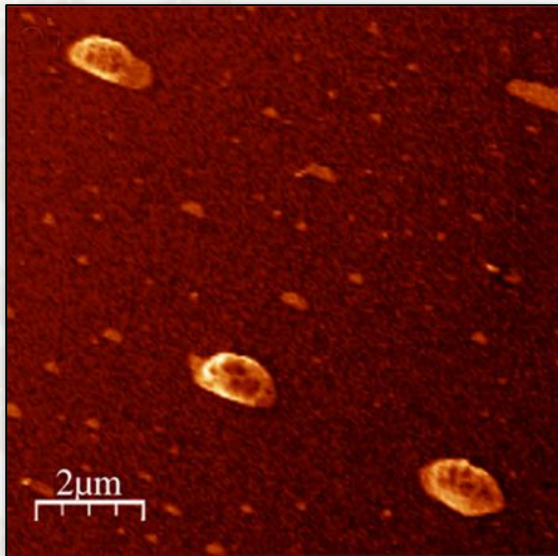
Linear size evolution: $D \approx 30 \times h$

Characteristic of dewetting process

Ion-beam lithography: gold dewetting

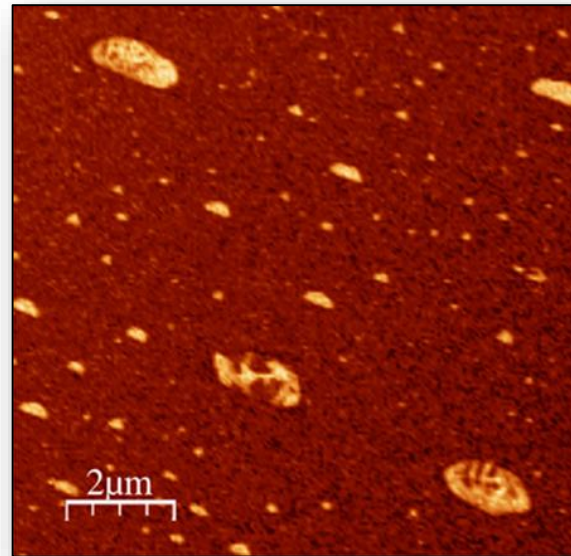
Gold dewetting regimes

$$\text{II. } 0.4 \leq h_{\text{Au}} \leq 5 \text{ nm}$$

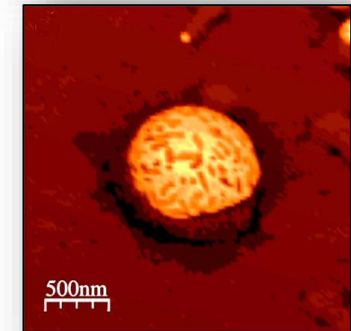
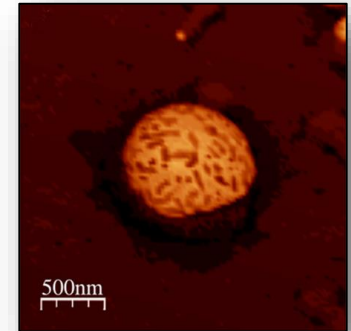


SE

SEM



BSE



SE

Silicon channels into droplets: phase separation of the binary alloy during cooling

Si-Au intermixing results in pinning of the droplets

Ion-beam lithography: gold dewetting

Gold dewetting regimes

III. $h \geq 5 \text{ nm}$

Annealing (30 min)

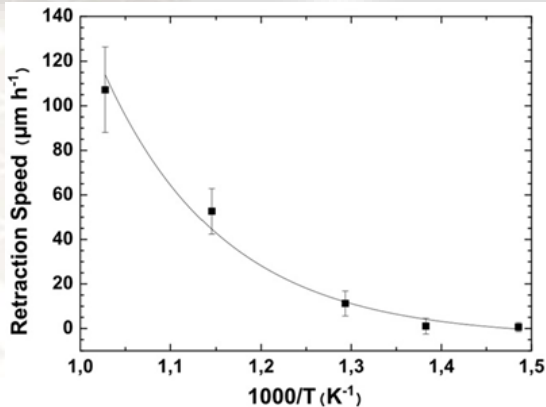
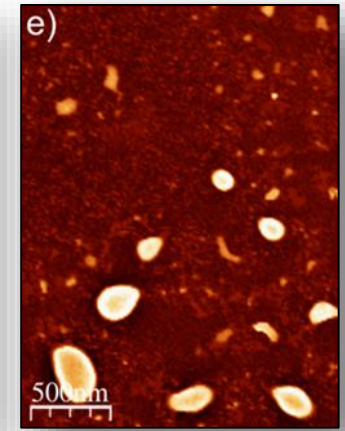
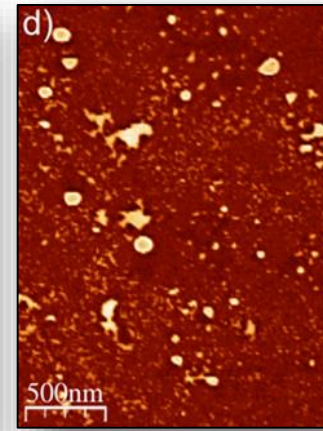
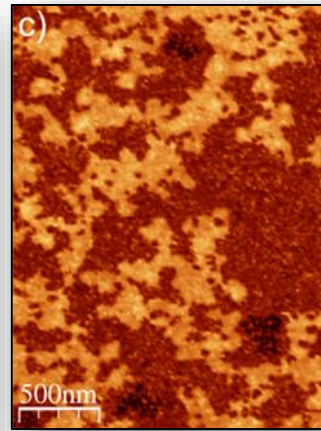
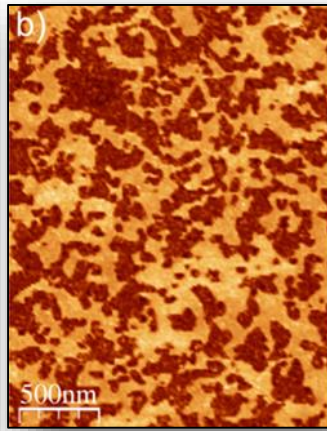
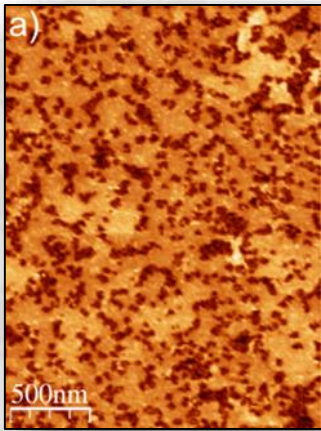
T = 450°C

T = 500°C

T = 550°C

T = 600°C

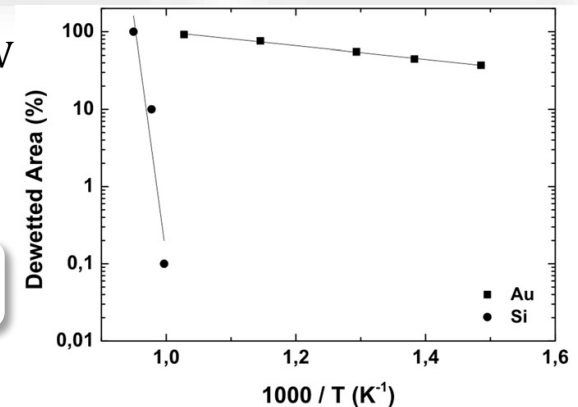
T = 700°C



- Diffusion activation energy: $E = 0.6 \text{ eV}$

Gold self diffusion

Hard Au diffusion control



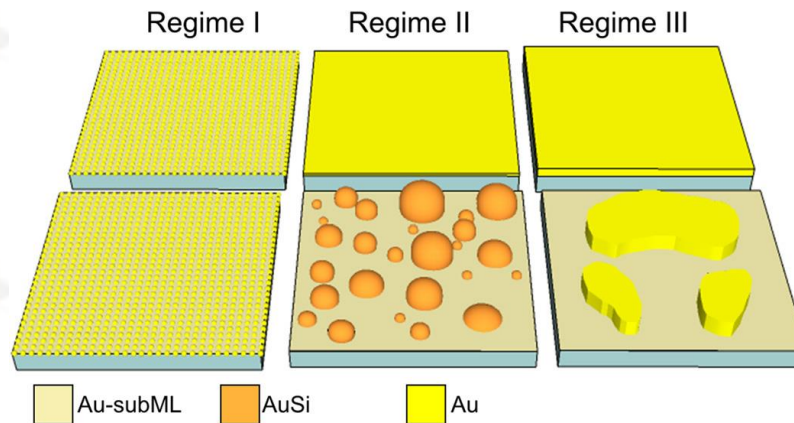
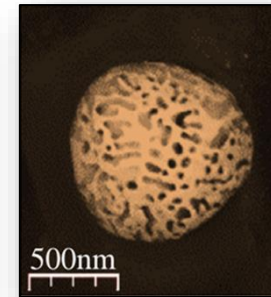
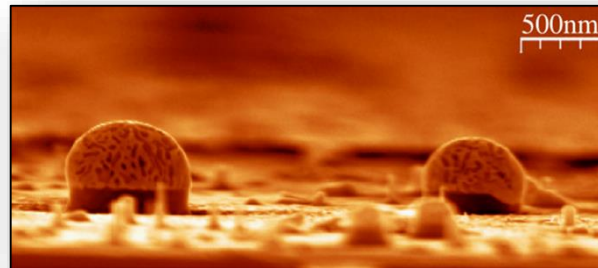
Ion-beam lithography: gold dewetting

Gold dewetting regimes

III. $h \geq 5 \text{ nm}$

No Si channels into droplets... unless additional supply of Si atoms

MBE deposition:
350 nm Si upon
6 nm Au layer



Summary

Ion-beam lithography: gold dewetting

Au layer FIB patterning

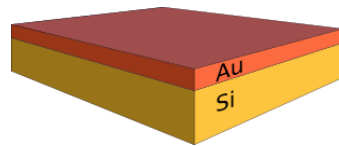
Chemical cleaning



Modified Shiraki*



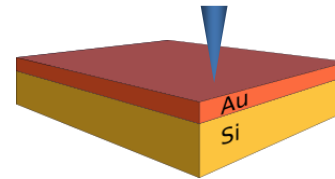
Au deposition



$0.4 \text{ nm} < h < 5 \text{ nm}$
(2 ML)



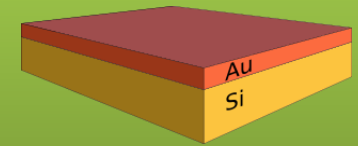
Ion lithography



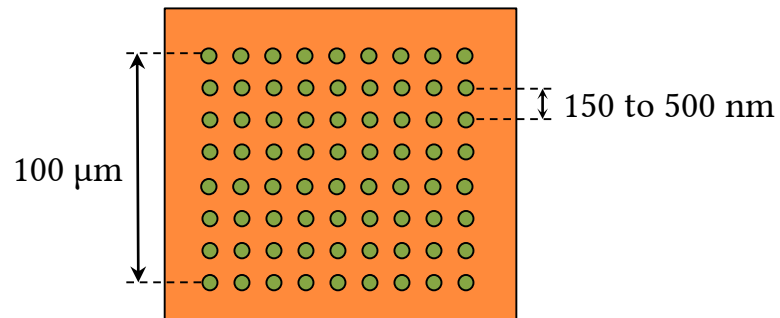
Ions	Ga ⁺ / Au ²⁺ / Ge ²⁺
Acceleration voltage	30 kV
Probe current	10-100 pA



UHV Annealing



$450^\circ\text{C} < T < 700^\circ\text{C}$
 $30 \text{ min} < t < 120 \text{ min}$

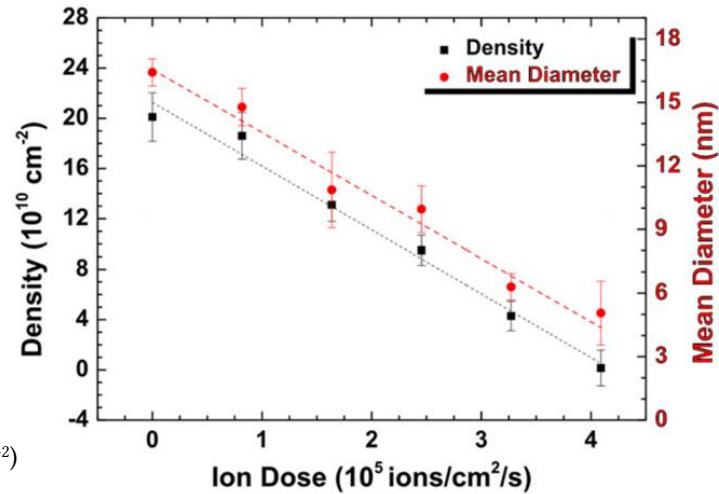
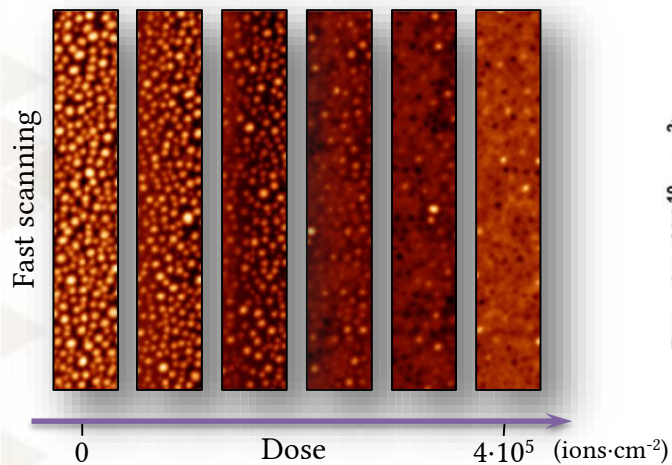


* Modified Shiraki:

1. HNO₃(65%) / 70°C / 10 min
2. H₂O (DI) / RT / 1 min
3. HF(49%) : H₂O (1:10) / RT / 30 s

Ion-beam lithography: gold dewetting

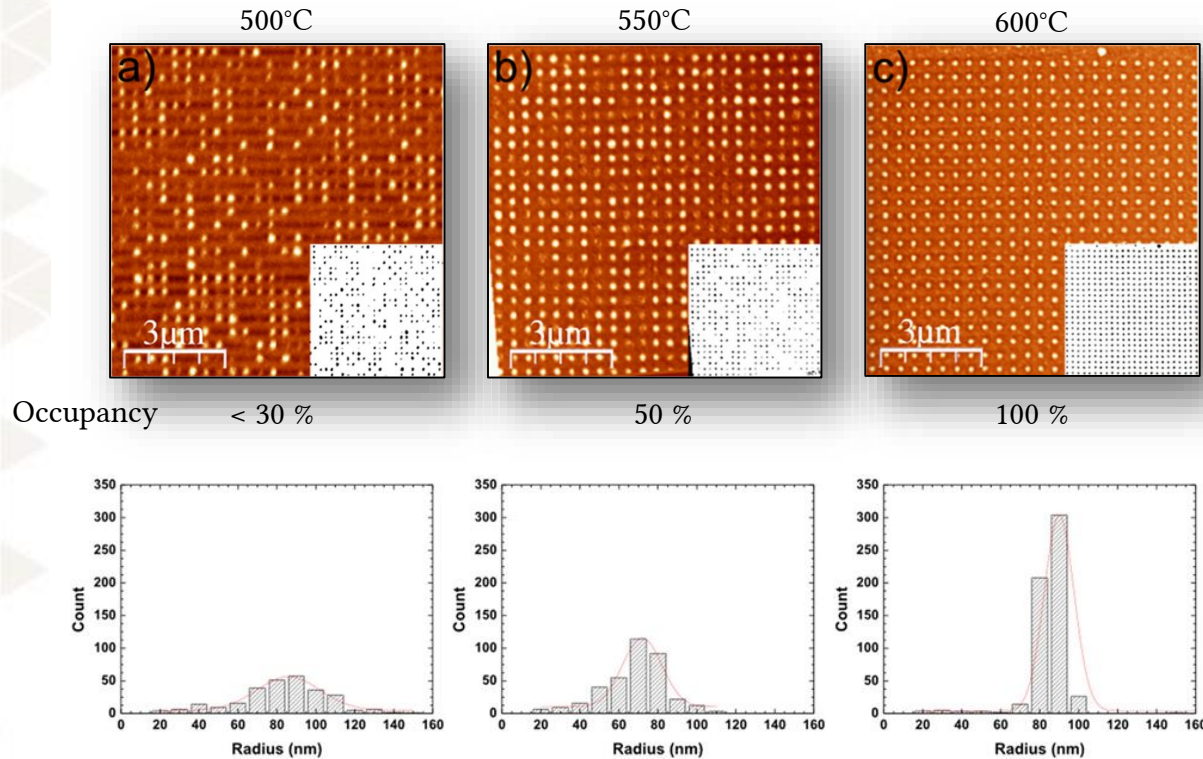
Gold dewetting regimes



Au droplets formation reduced by FIB erosion

Ion-beam lithography: gold dewetting

Dewetting: temperature influence



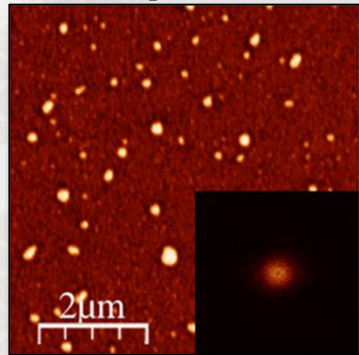
Au – Si interdiffusion induced in FIB patterns \Rightarrow droplets formation

Au diffusion promoted by the temperature

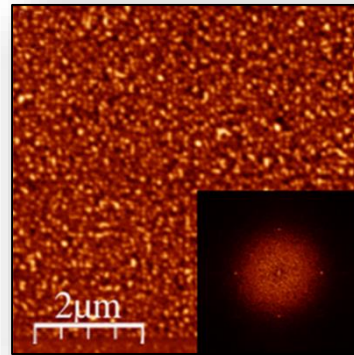
Ion-beam lithography: gold dewetting

FIB patterning: pitch limit

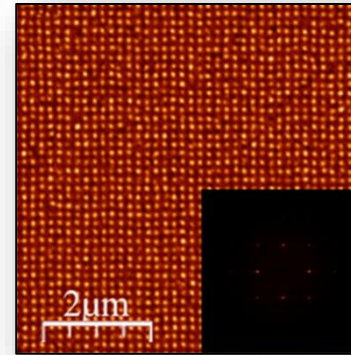
Unpattern



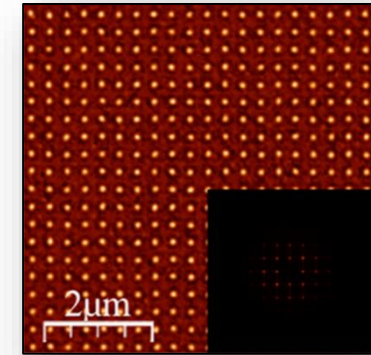
Pitch = 100 nm



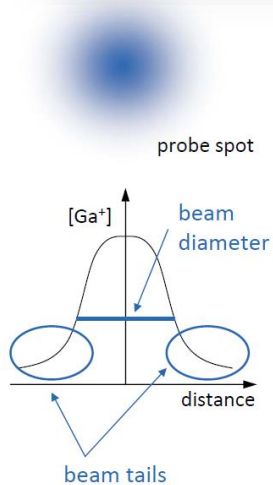
Pitch = 250 nm



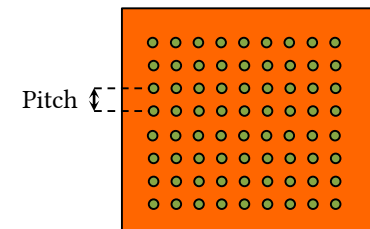
Pitch = 500 nm



Superimposition of the patterned areas:
extended tail of the ion beam



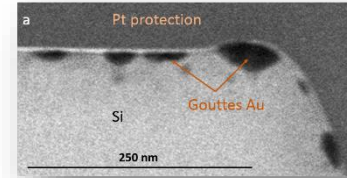
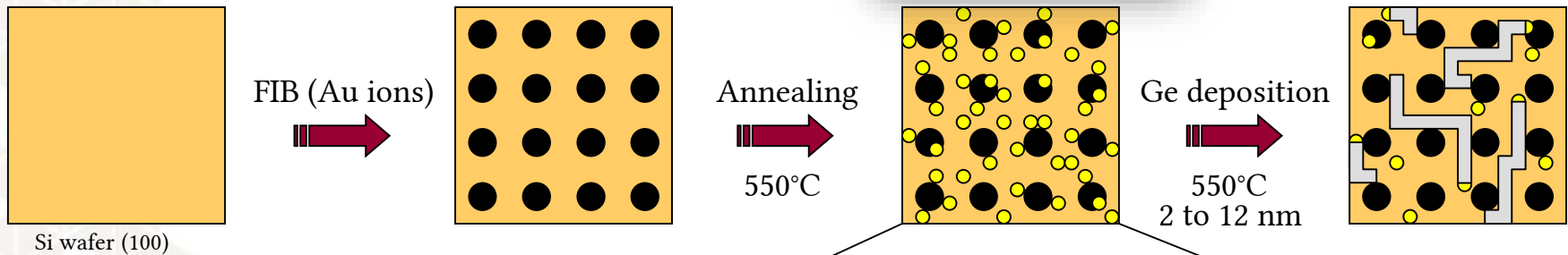
Optimizing FIB pattern



Ion-beam lithography: Au dots

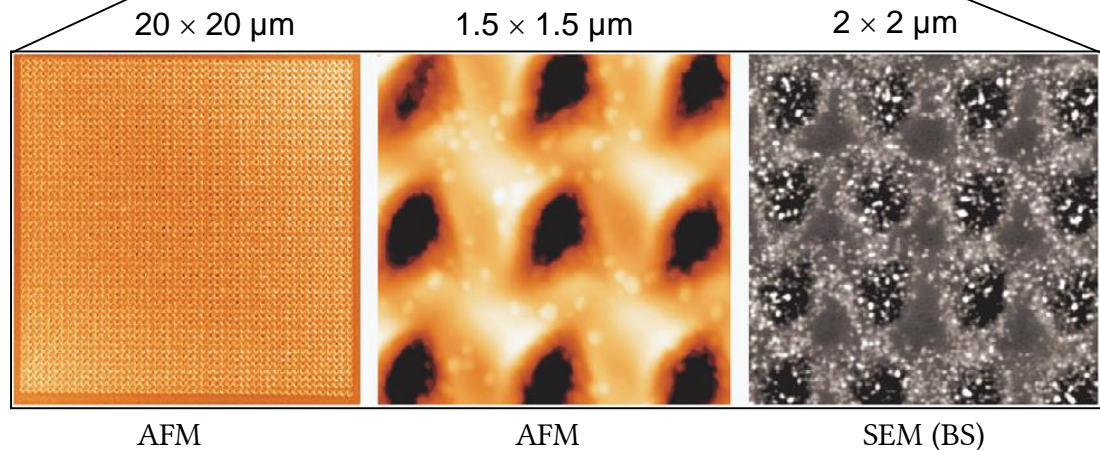
Au patterning + Ge deposition on Si(100)

Fabrication process :



Au lithography + annealing

Formation of Au dots



I. C. Marcus *et al.* Cryst. Growth Des. (2011)

Ion-beam lithography: Au dots

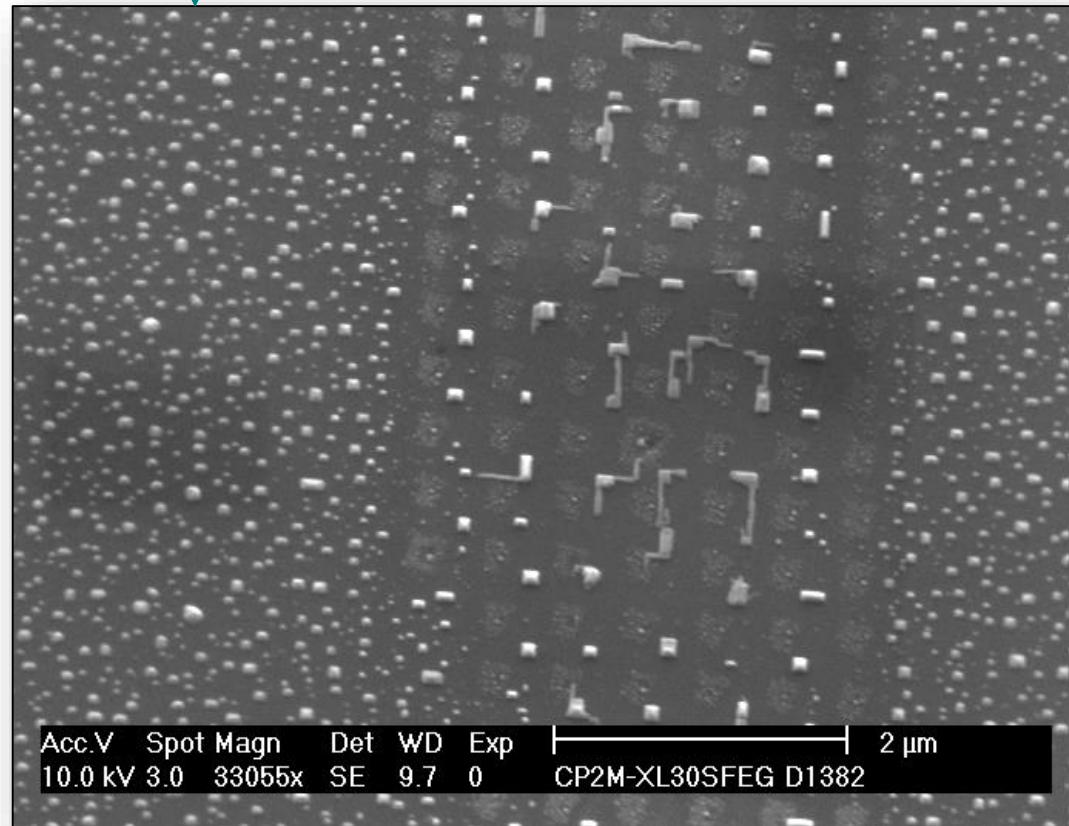
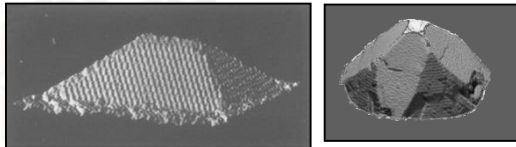
Au patterning + Ge deposition on Si(100)

Unpatterned surface

FIB Patterned surface
⇒ in plane nanowires growth

Bimodal distribution :

- huts
- domes



Nanowire growth speed:

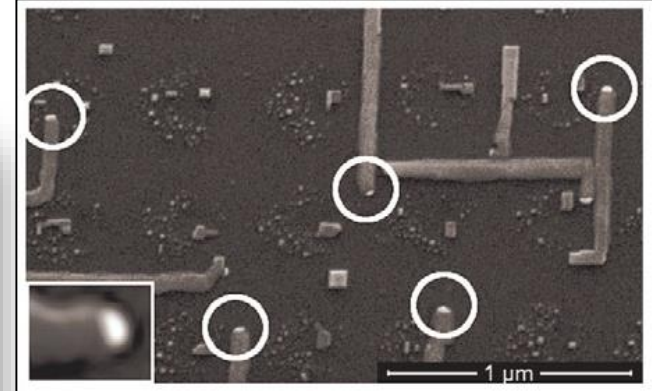
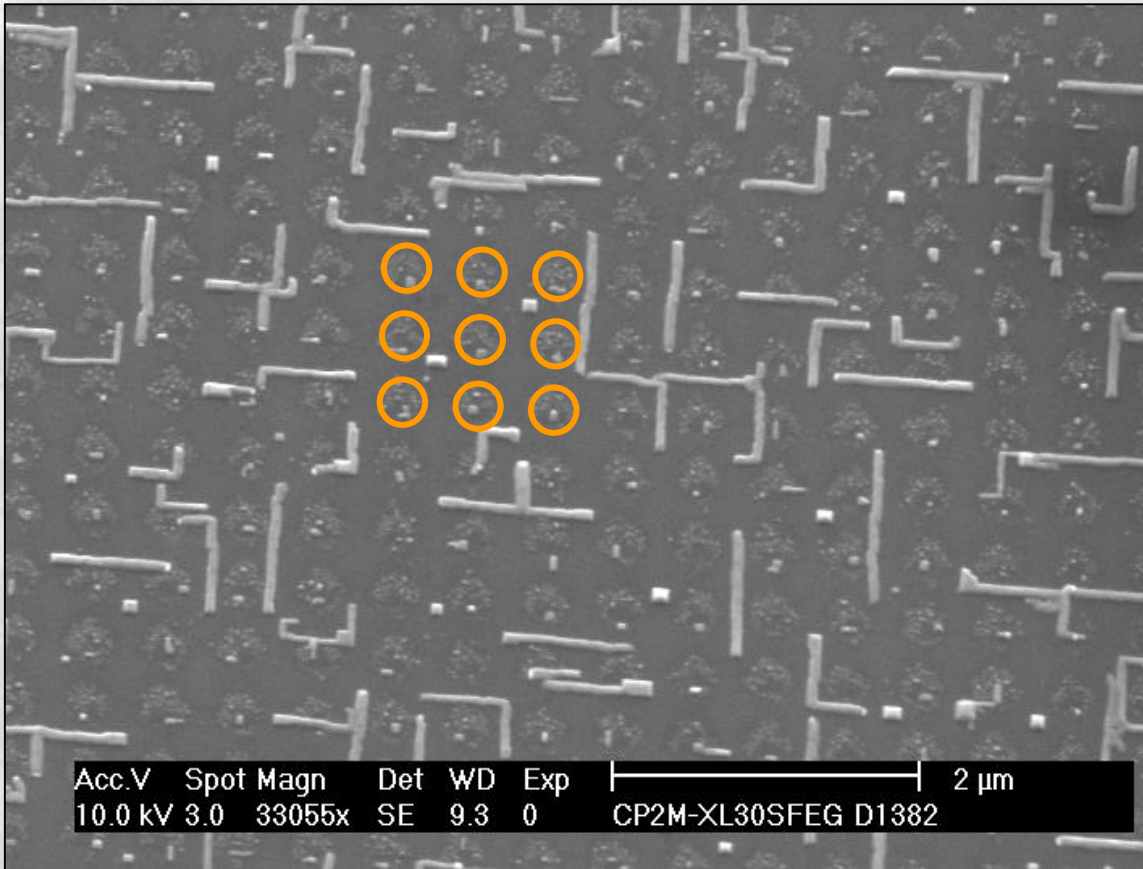
$$v_s = \alpha l_s (\mu_l - \mu_s)$$

I. C. Marcus *et al.* Cryst. Growth Des. (2011)

Ion-beam lithography: Au dots

Au patterning + Ge deposition on Si(100)

FIB pattern



Au catalyst droplets

Ge nanowires :

- Trapezoidal section
- Section square root: 50 nm
- Length: 1000 nm

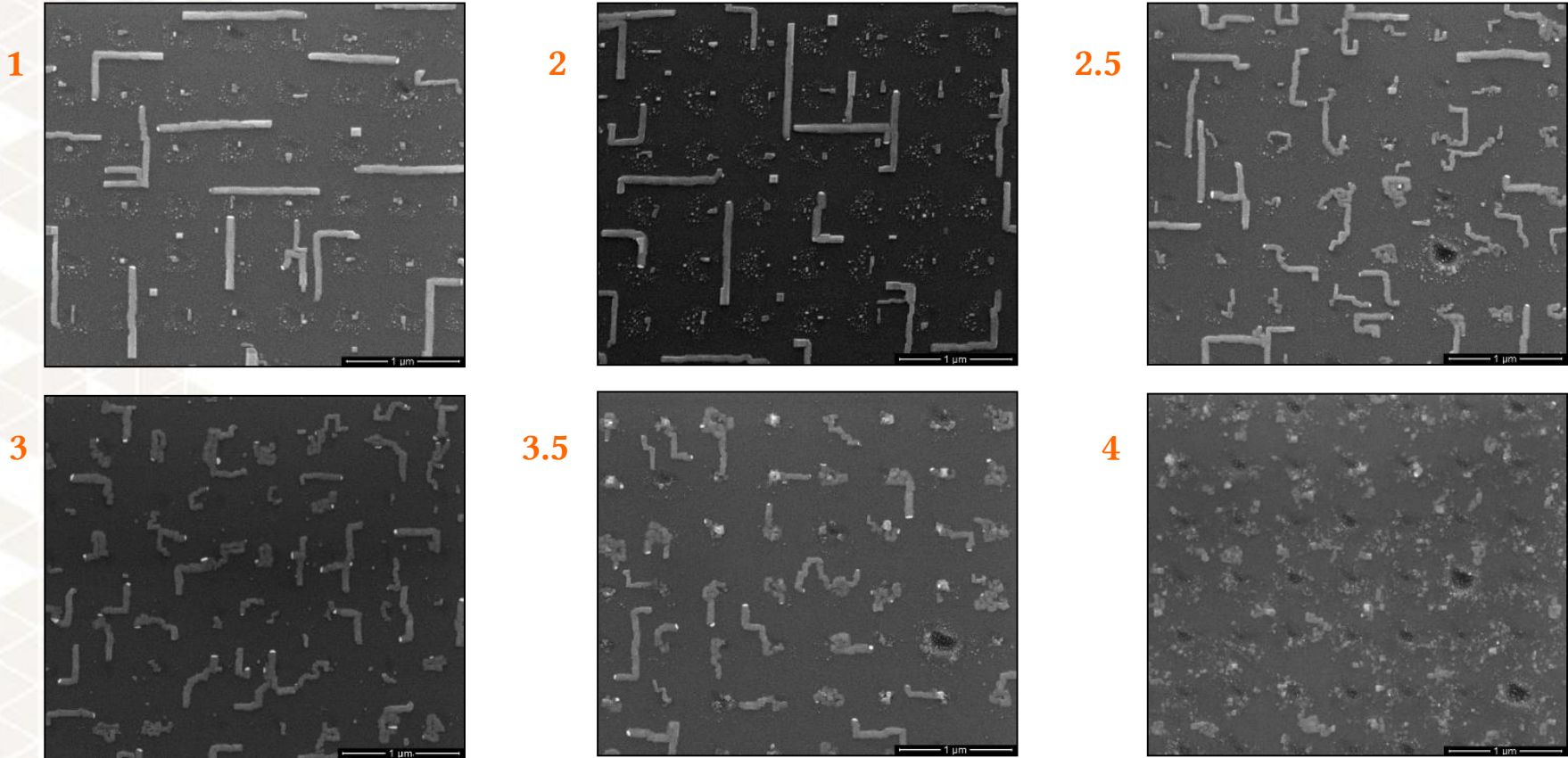
⇒ aspect ratio = 0.05

I. C. Marcus *et al.* Cryst. Growth Des. (2011)

Ion-beam lithography: Au dots

Au patterning + Ge deposition on Si(100)

Influence of ion dose (a. u.)



- Growth along $\langle 110 \rangle$ directions
- Surface roughness acts as a diffusion barrier: waviness growth, almost hindered for higher doses

I. C. Marcus *et al.* Cryst. Growth Des. (2011)

Ion-beam lithography: Au dots

Organised Ge dots on Si(100) using “FIB single” process

Principle: do a single image using Au ions FIB



Single image of the substrate surface

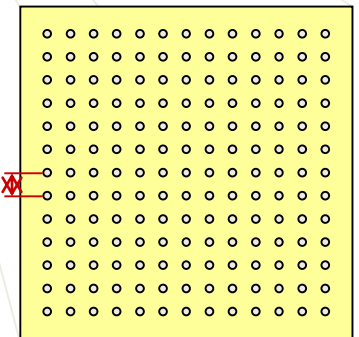
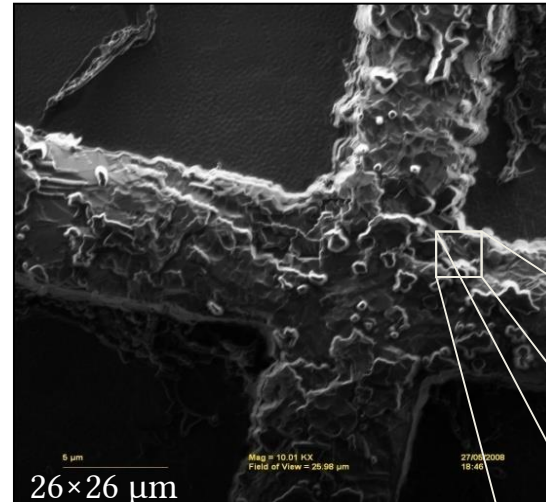


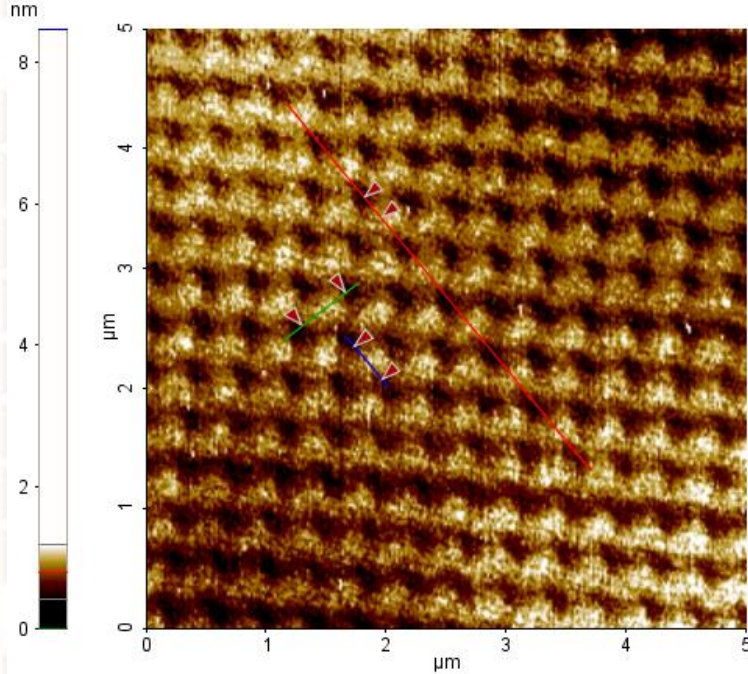
Image resolution
=
Step pattern

Ion-beam lithography: Au dots

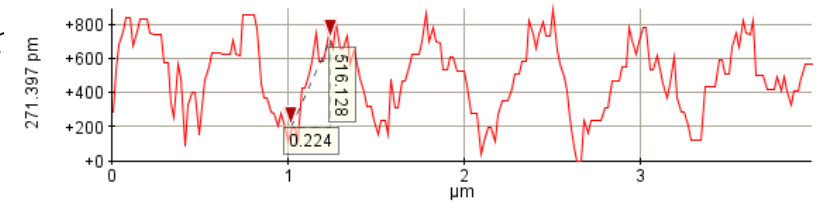
Organised Ge dots on Si(100) using “FIB single” process

Principle: do a single image using Au ions FIB

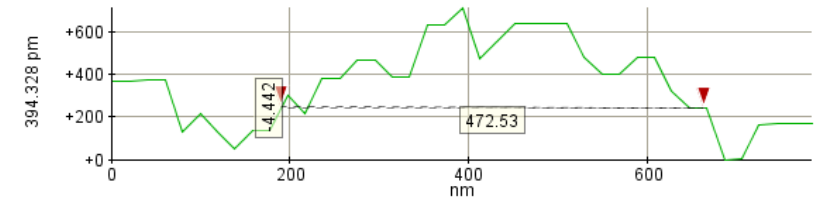
- Specific parameters: Au deposition
- Dots array: step = Magnification + resolution



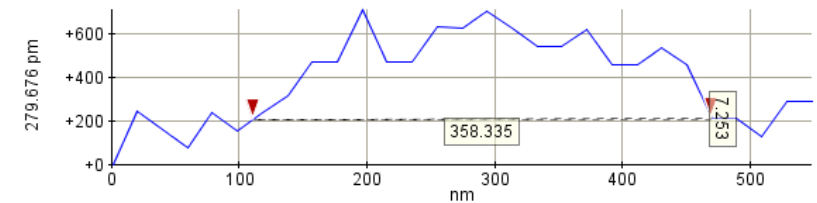
Line Profile: Red



Line Profile: Green



Line Profile: Blue



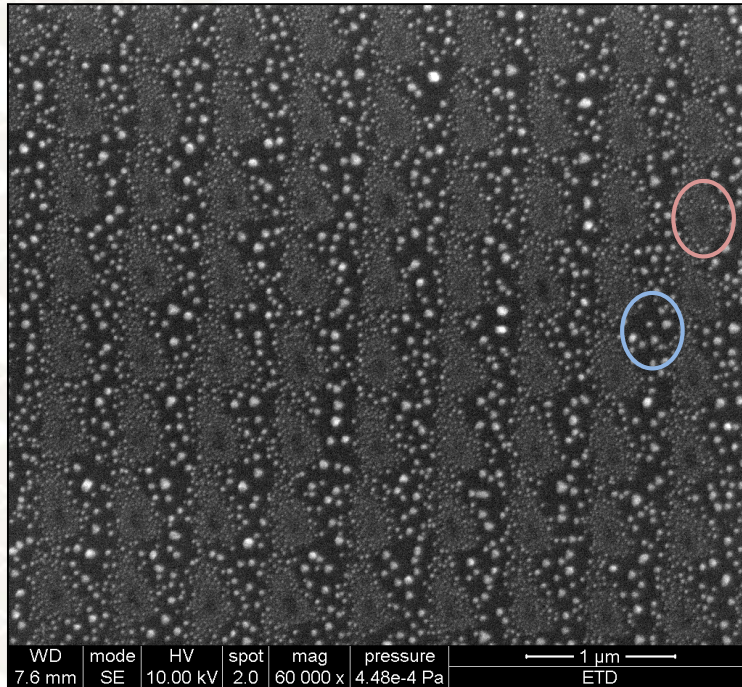
Large structure (≈ 450 nm) and weak height (0.5 nm)

Ion-beam lithography: Au dots

Organised Ge dots on Si(100) using “FIB single” process

Presence of gold on surface during Ge deposition:

- increase number of droplets nucleation centres
- reduce Ge scattering

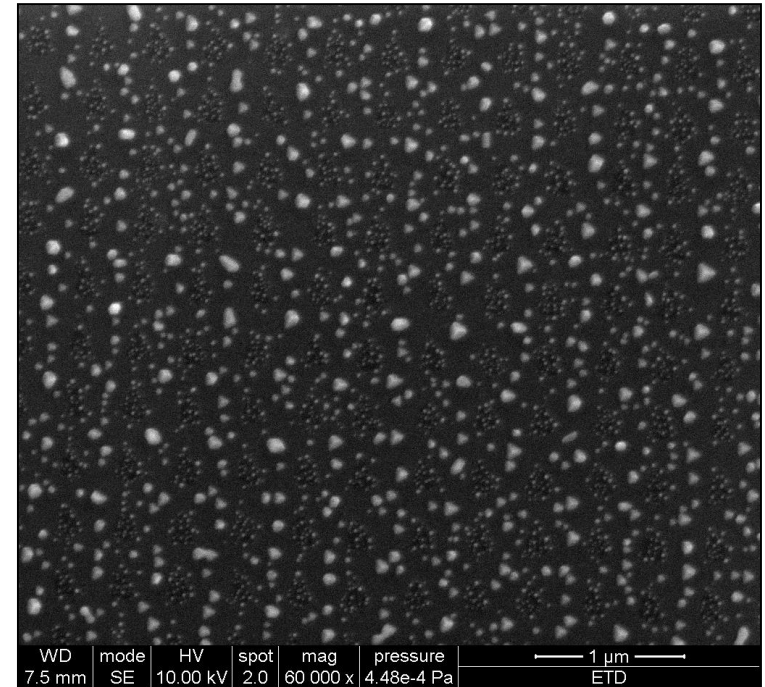


with gold

without gold



FIB step
tuned



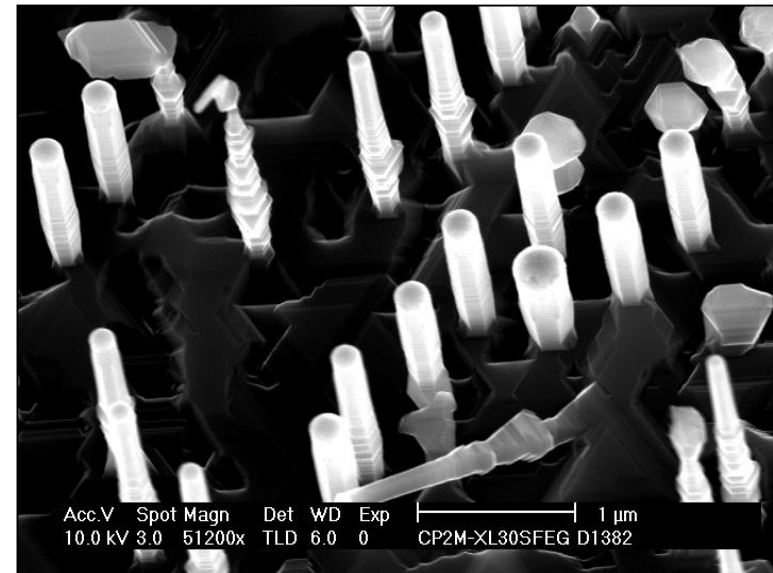
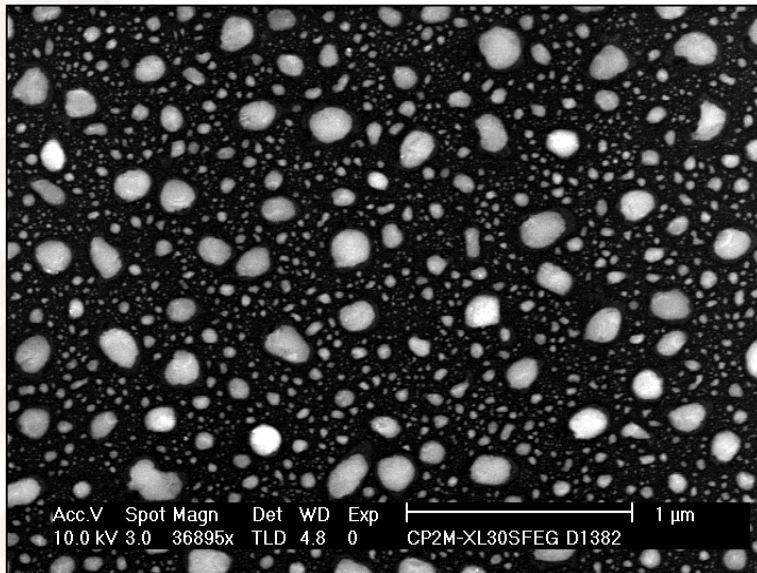
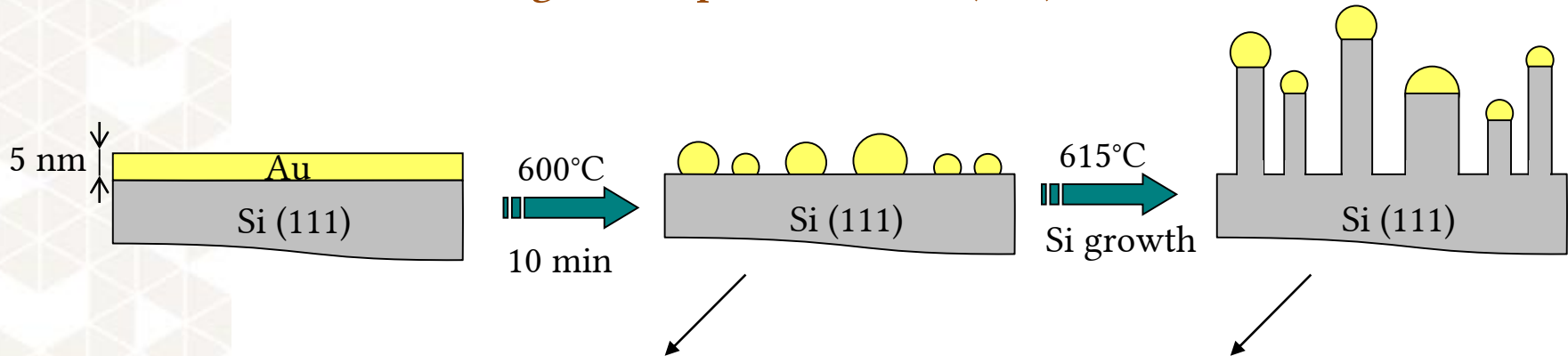
Step = 500 nm (768 × 768)

Array of Ge droplets

Step = 480 nm (1024 × 1024)

Ion-beam lithography: Au catalyst

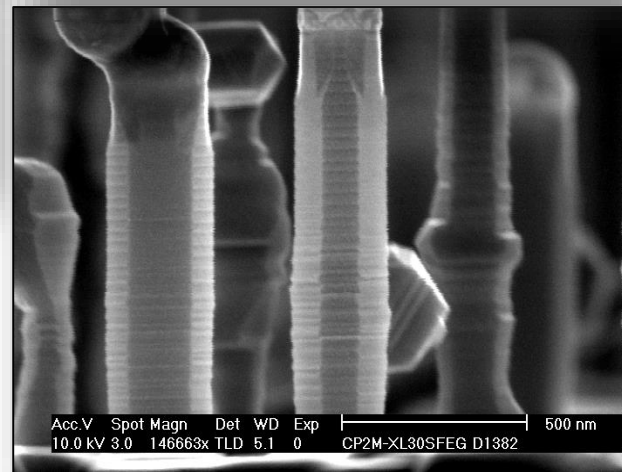
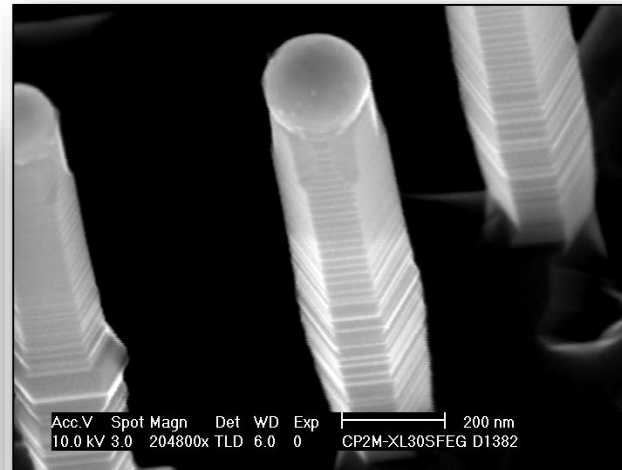
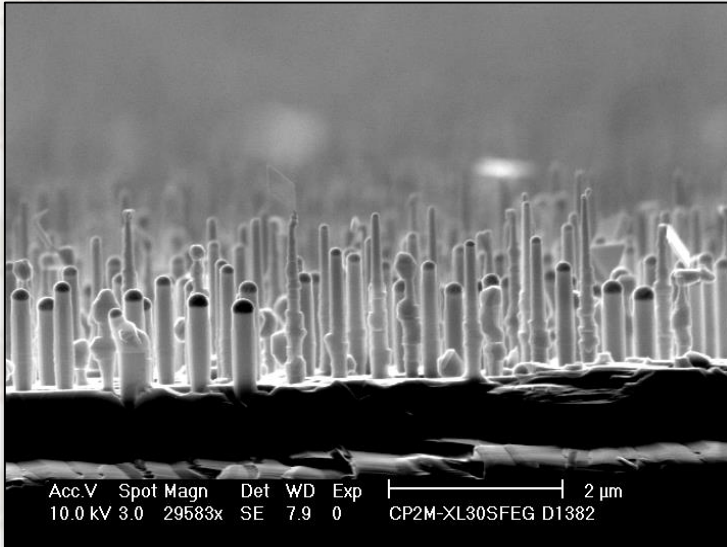
Au dewetting + Si deposition on Si(111)



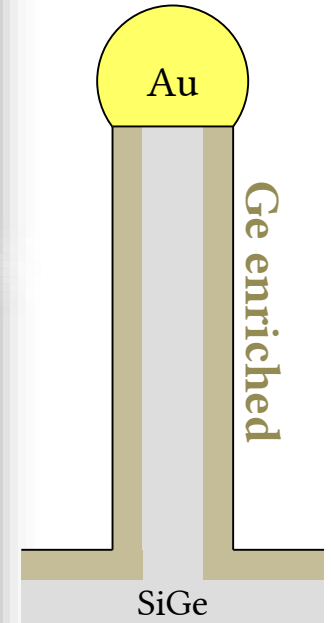
In collaboration with Prof. M. De Crescenzi and his team (University of Roma Tor Vergata, Italy)

Ion-beam lithography: Au catalyst

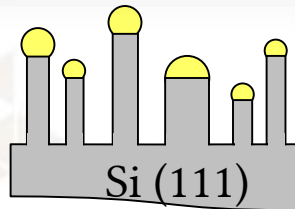
Au dewetting + Si deposition on Si(111)



$\varnothing \approx 200 \text{ nm}$
 $h \approx 1500 \text{ nm}$

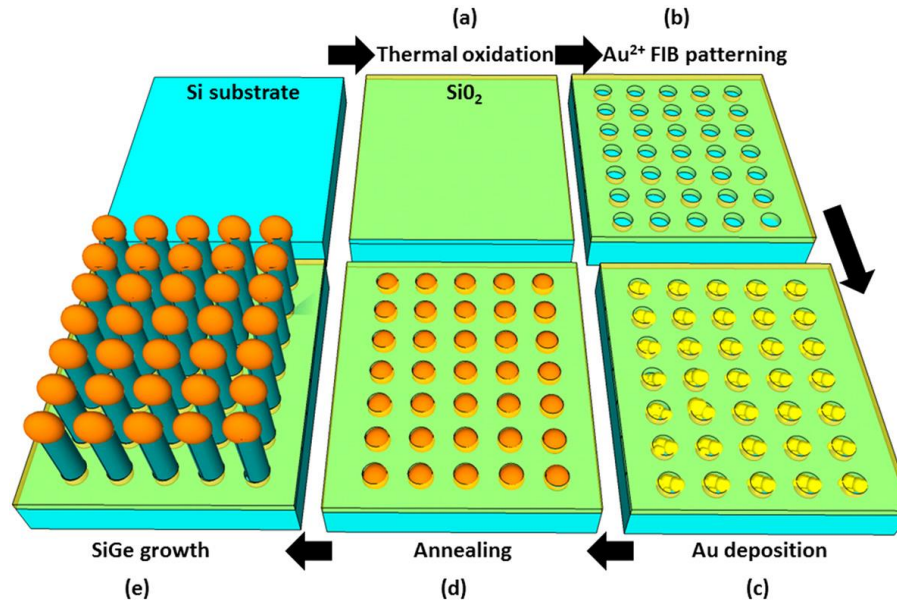


heteroepitaxy



Ion-beam lithography: Au catalyst

Ultimate FIB patterning process



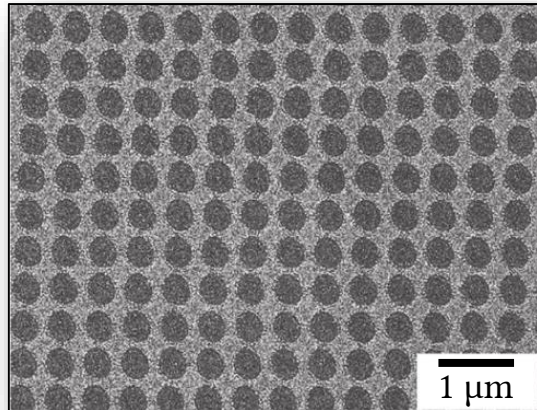
- (a) Formation of SiO_2 ($h_{\text{SiO}_2} = 5 \text{ nm}$) by RTO
- (b) Opening of SiO_2 - free windows by FIB milling
- (c) Au deposition by oxydoreduction of gold salts
- (d) Phase transition of Au in AuSi clusters by annealing (T_A)
- (e) MBE growth of SiGe NWs at T_A

Ion-beam lithography: Au catalyst

Au salts oxidoreduction (galvanic displacement)

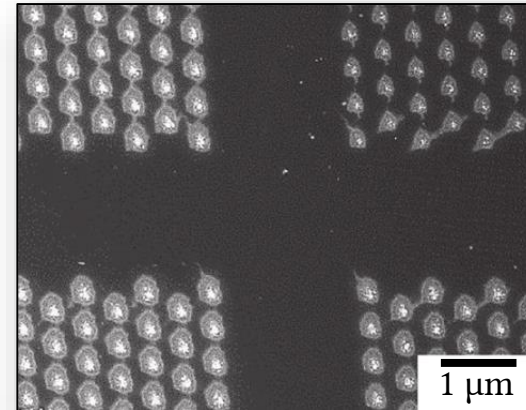


Not selective nor limited



Diluted HF

Selective and self limited

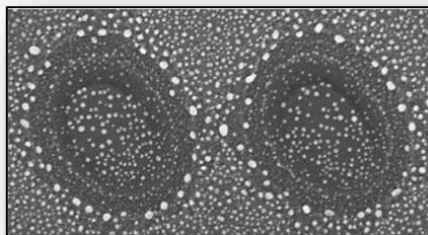


No HF

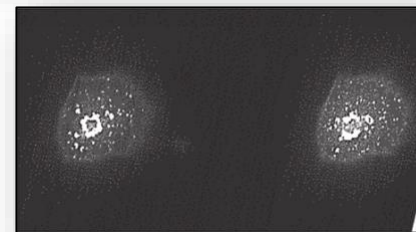
Au thickness ~5nm

Au clusters diameter

- ≈ 20 nm in pattern
- ≈ 40 nm out of pattern



250 nm

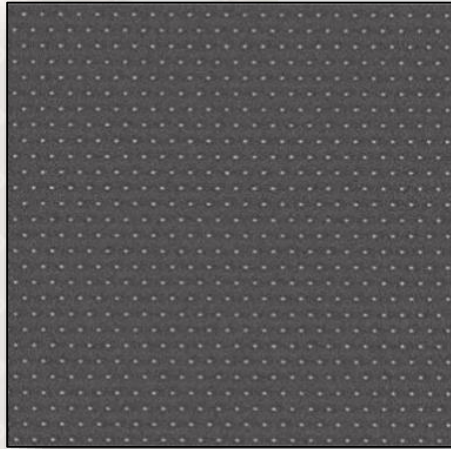


500 nm

Ion-beam lithography: Au catalyst

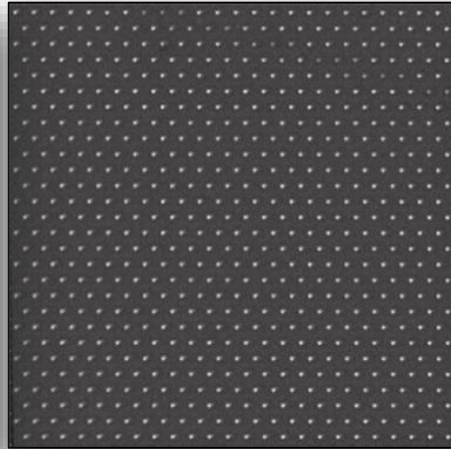
Dose influence (pA/cm^{-2})

2.8×10^{15}



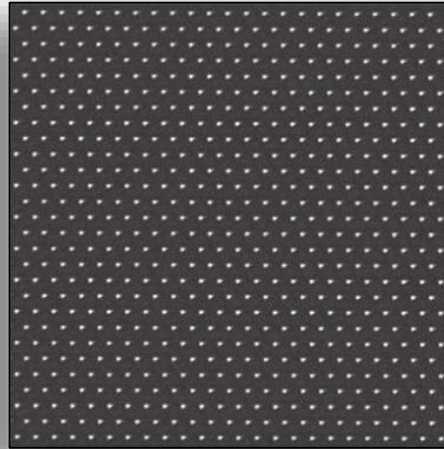
SEM - BSE

5.5×10^{15}



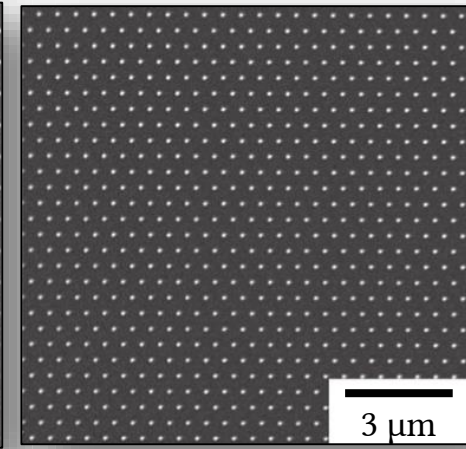
SEM - BSE

8.3×10^{15}



SEM - SE

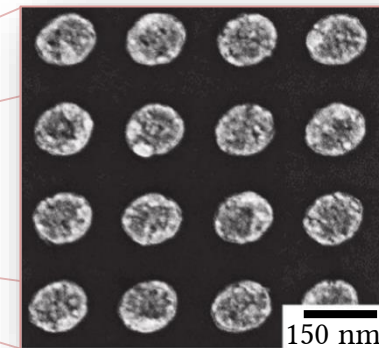
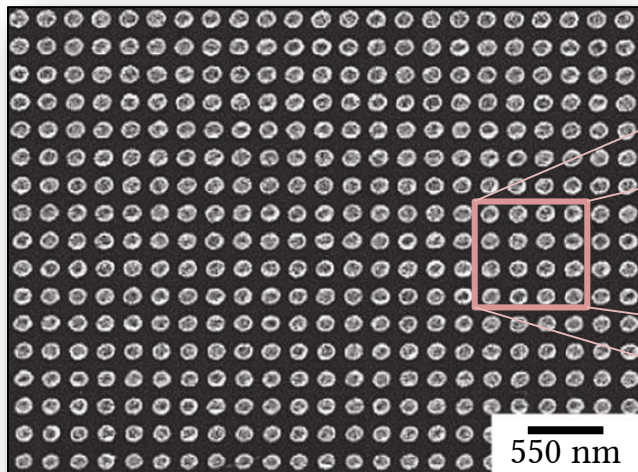
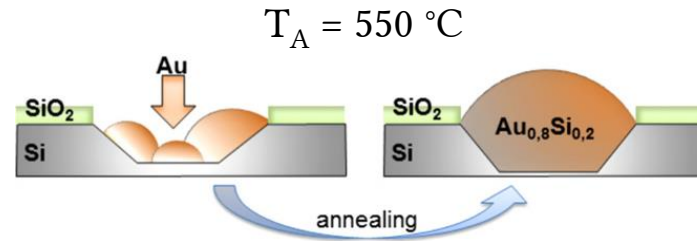
11×10^{15}



3 μm

Ion-beam lithography: Au catalyst

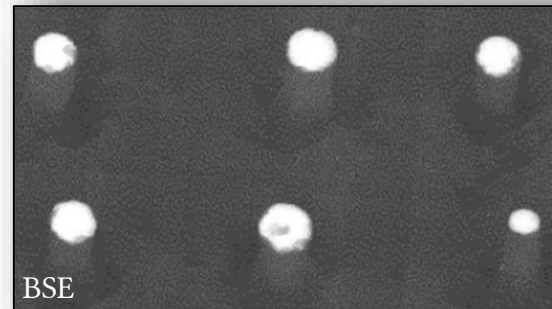
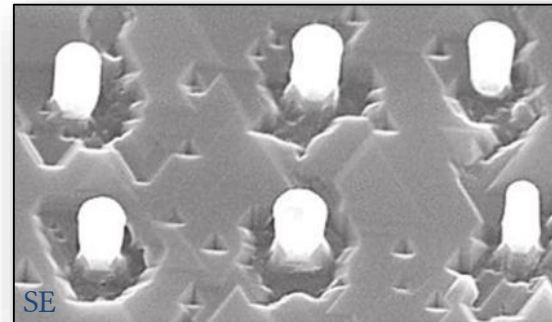
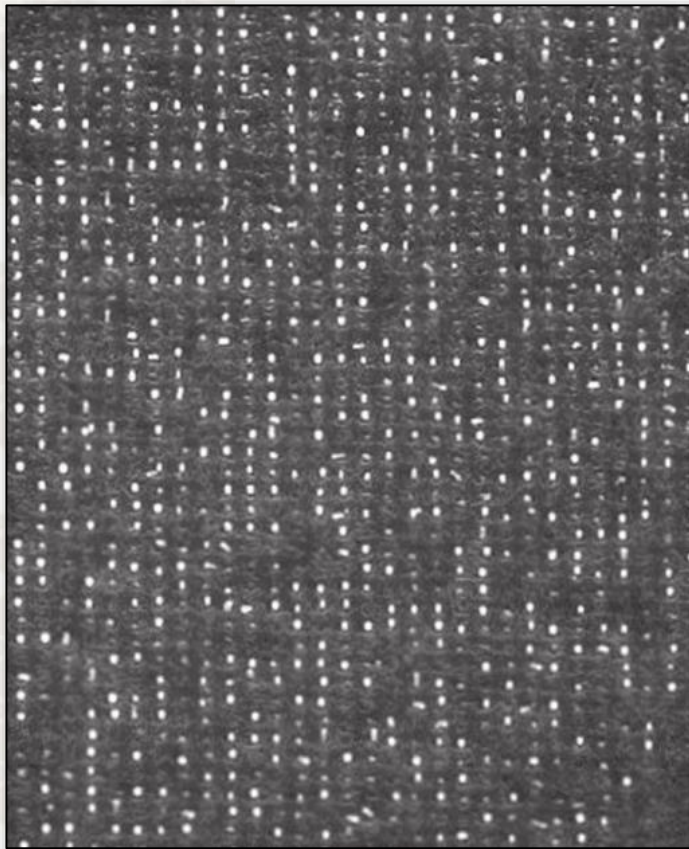
Annealing



Ion-beam lithography: Au catalyst

MBE growth

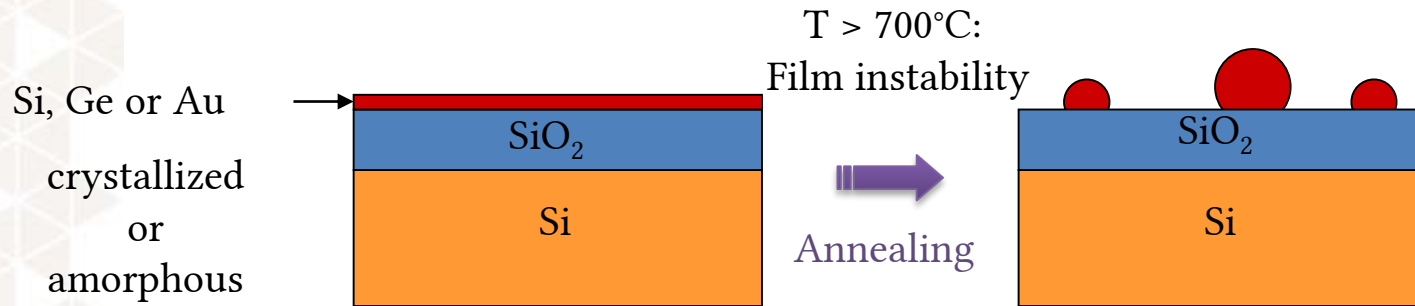
$T_{\text{Growth}} = 550\text{ }^{\circ}\text{C}$



Nanowires height: $h = 200\text{ nm}$

Dewetting process organization

Heating ultra-thin films on oxide



Dewetting process organization

Chemical potential

$$\mu^\beta = \underbrace{\mu_m^\beta(T)}_{\text{Original } \beta \text{ phase chemical potential}} + \underbrace{\frac{1}{2} C^\beta \varepsilon^2}_{\text{Elastic strain energy density}} - \underbrace{\sigma_n \Omega^\beta}_{\text{Work (pV)}} + \underbrace{\left[\left(\gamma^{\alpha\beta} + \frac{d^2 \gamma^{\alpha\beta}}{d\theta_1^2} \right) \frac{1}{R_1} + \left(\gamma^{\alpha\beta} + \frac{d^2 \gamma^{\alpha\beta}}{d\theta_2^2} \right) \frac{1}{R_2} \right]}_{\text{Surface energy (including anisotropy)}} \Omega^\beta$$

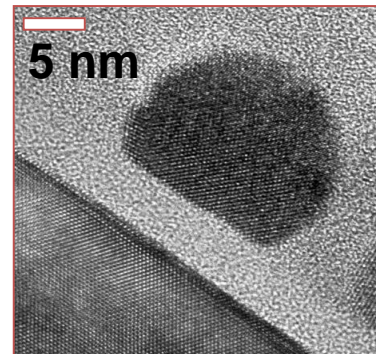
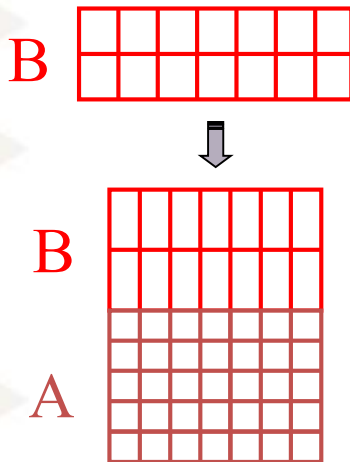
Original β phase
chemical potential

Work (pV)

Surface energy (including anisotropy)

- Ω : atomic volume (or molecular)
- R_i : main radius of curvature
- θ_i : surface orientation

Elastic strain
energy density

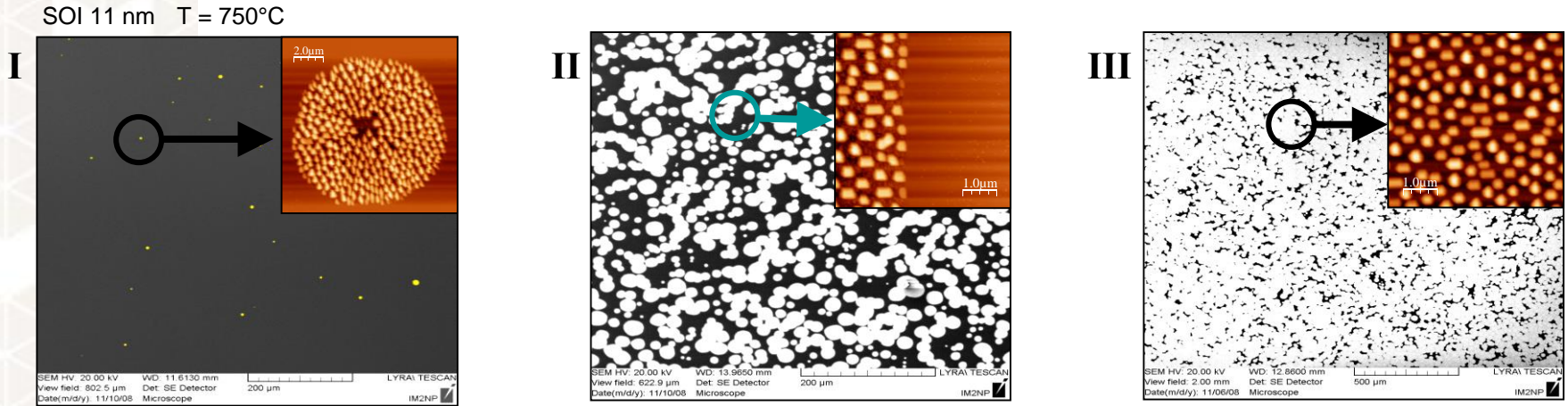


+ Scattering Barriers
Surface defects, steps, ...

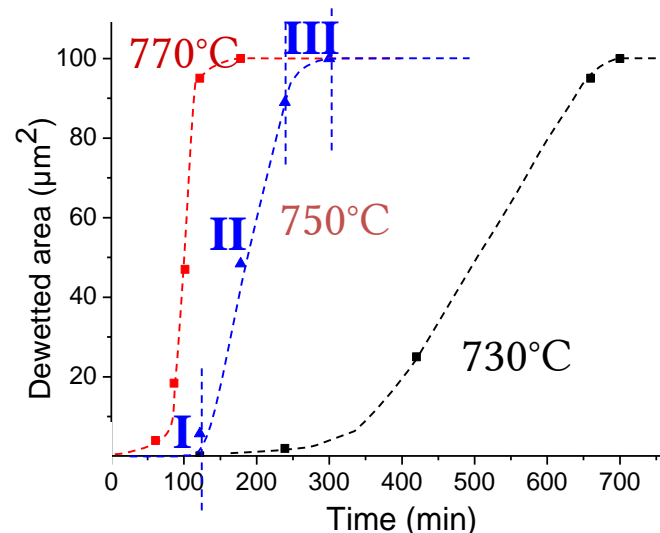
Growth and self-organisation of SiGe nanostructures
J.N. Aqua, I. Berbezier, L. Favre, T. Frisch, A. Ronda, Phys. Rep. (2013)

Dewetting process organization

Dewetting process as a function of temperature (SOI)



- (I) Holes nucleation
- (II) Film retraction and holes nucleation
- (III) Dewetted areas coalescence



M. Aouassa, L. Favre, A. Ronda, H. Maaref and I. Berbezier, *New J. Phys.*, **14**, 063038 (2012)

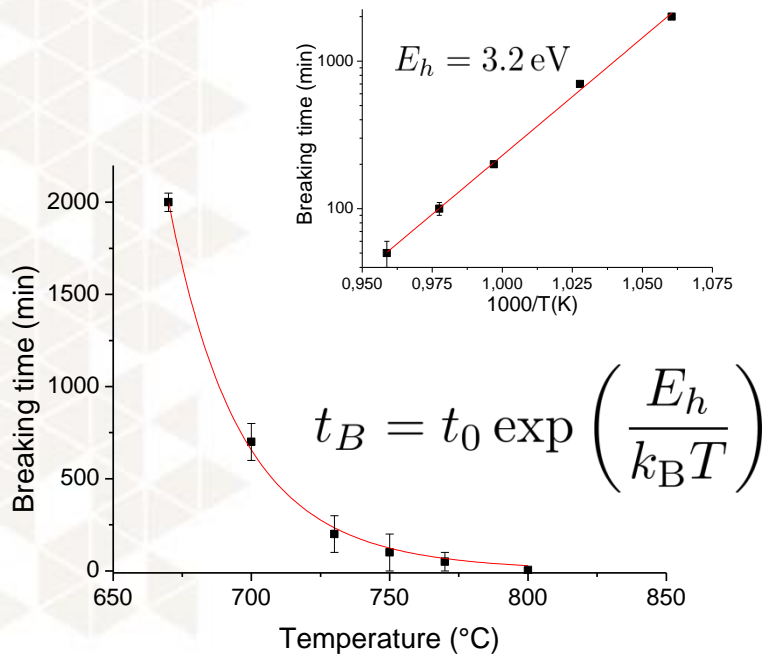
Dewetting process organization

Heating ultra-thin films on oxide

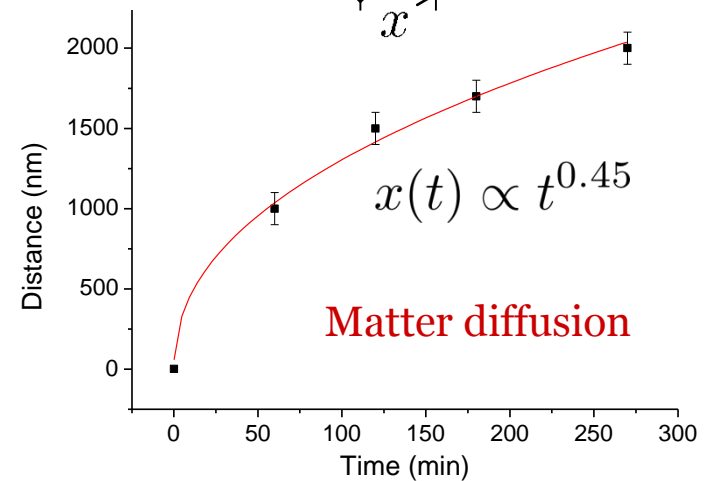
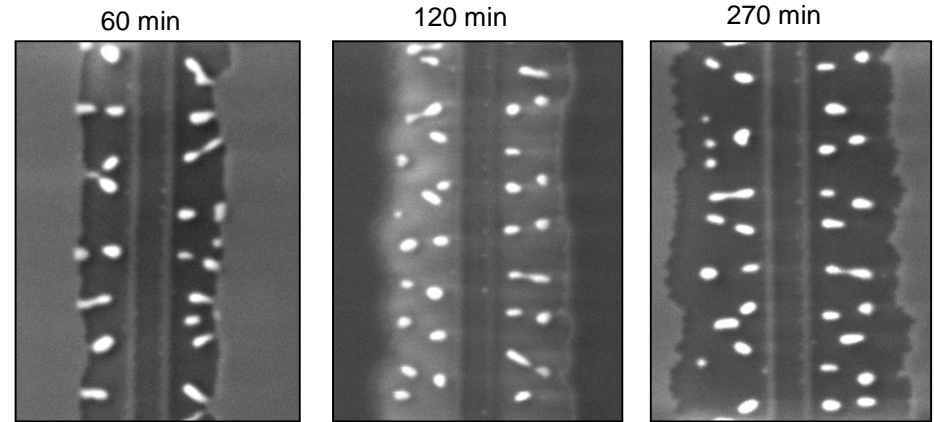
Nucleation rate

$$\tau = \tau_0 \exp\left(-\frac{E_h}{k_B T}\right)$$

G_H : hole nucleation barrier



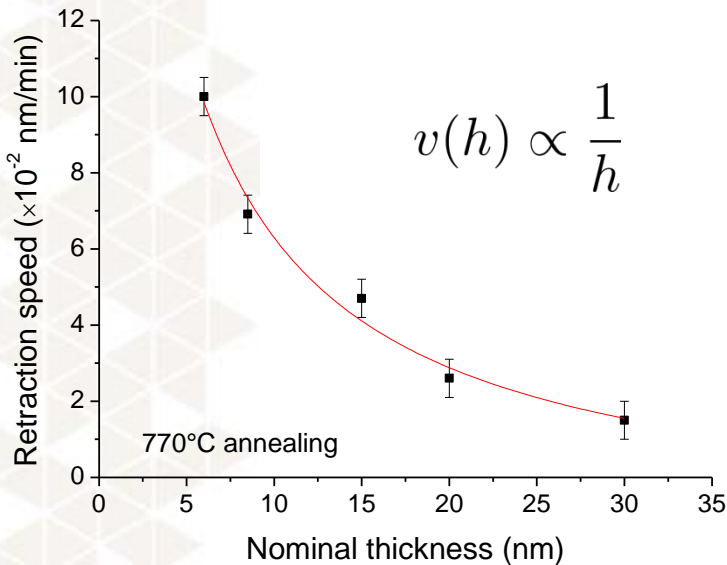
Dewetted front of pa-Si



Dewetting process organization

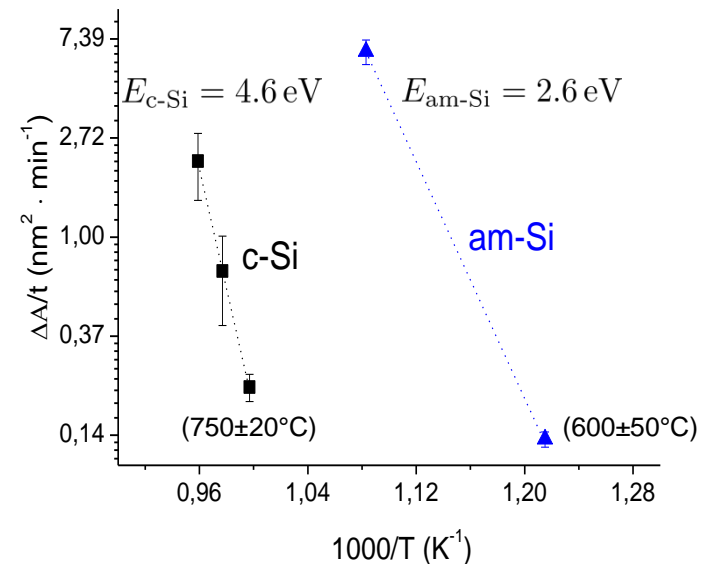
Dewetting process as a function of film thickness and temperature (SOI)

Motion of the dewetted front as a function of the nominal thickness



- Matter conservation law
- Consistent with a diffusion limited dewetting mechanism

Dewetting speed as a function of the temperature for c-Si and am-Si

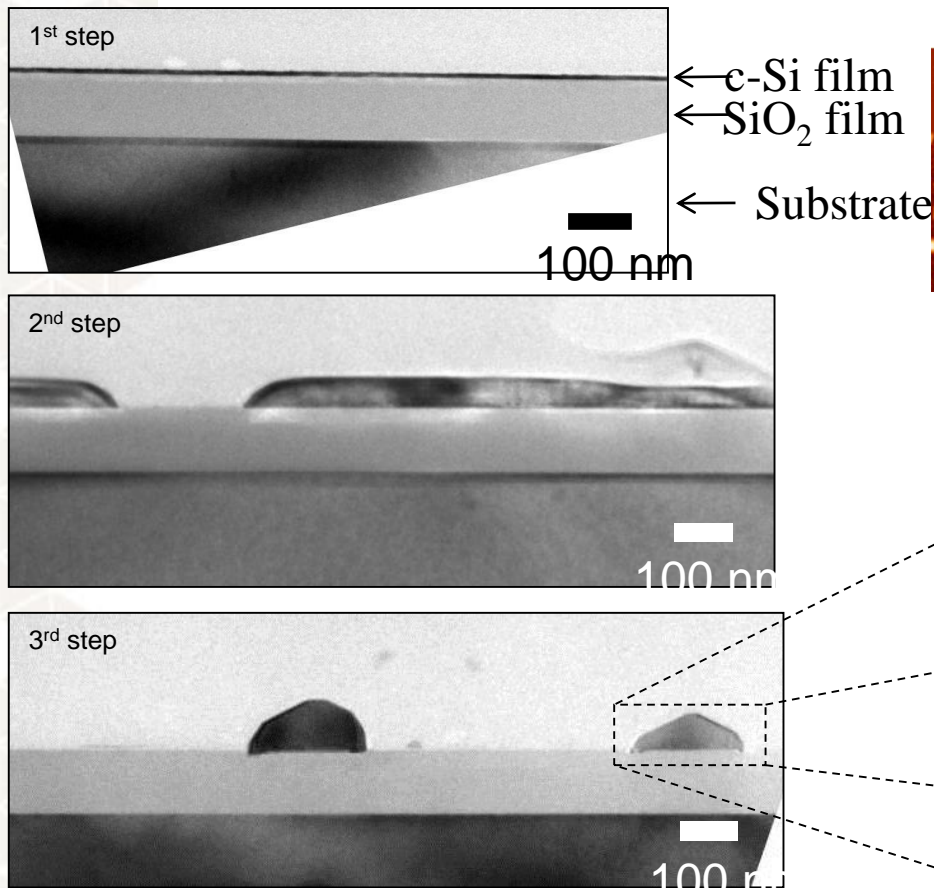


- am-Si films dewet almost instantly: no breaking time
- Activation energy is much smaller for am-Si
- Dewetting speed of am-Si is higher c-Si by two orders of magnitude (at 730°C)

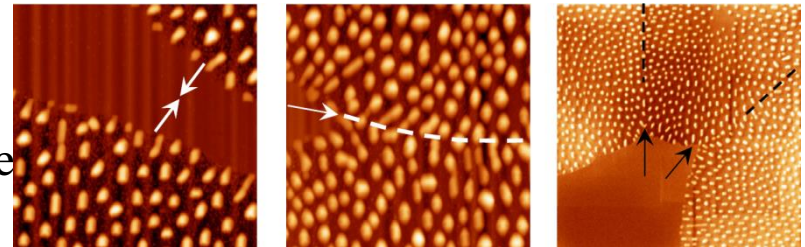
Dewetting process organization

Dewetting process as a function of time (SOI)

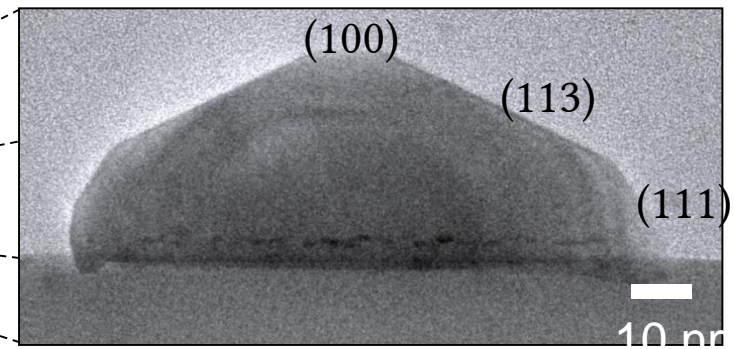
Dewetting process among time



• 3rd step: coalescence

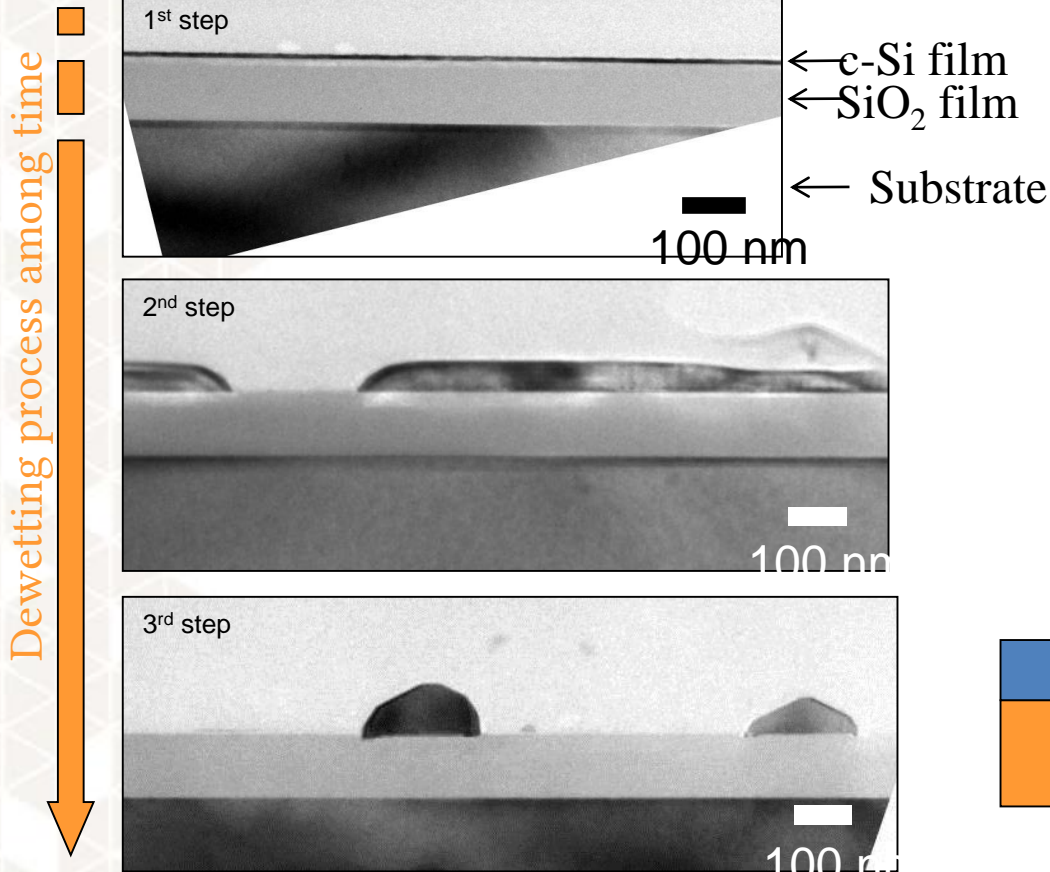


• faceted dots



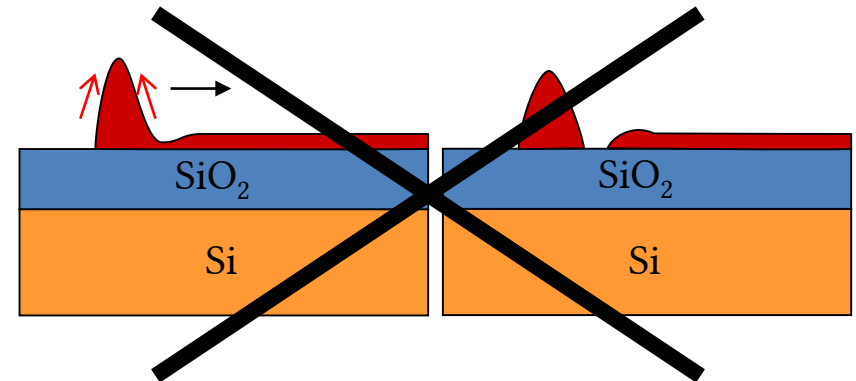
Dewetting process organization

Dewetting process as a function of time (SOI)



• 2nd step: film retraction

- flat film
- thickening on large scale
- no rim (Mullins model)

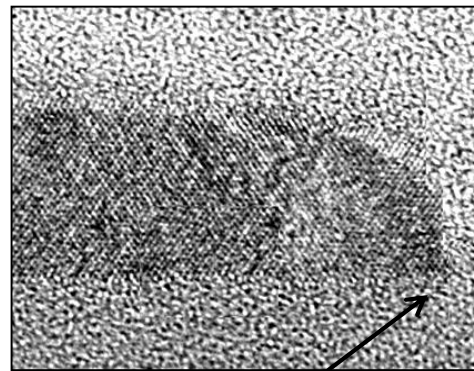
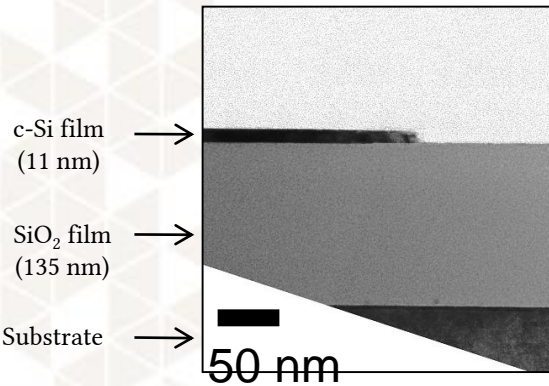
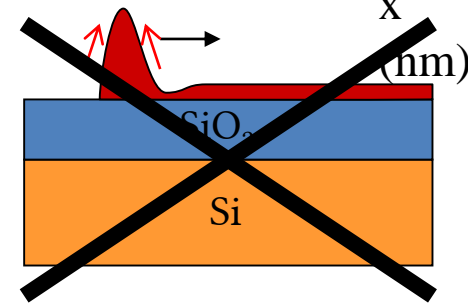
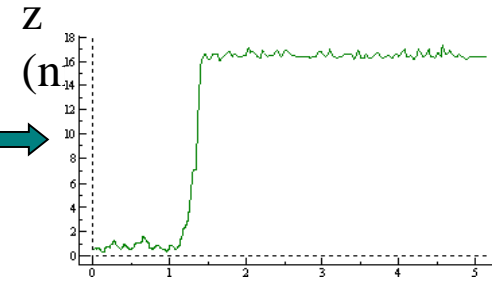
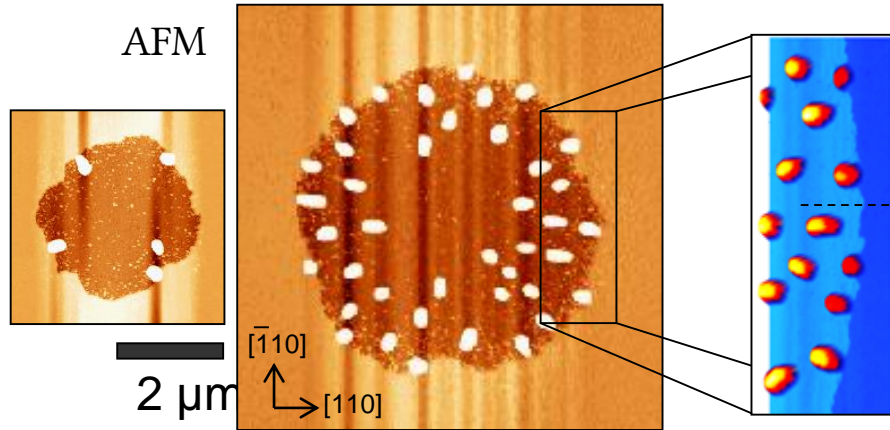


Dewetting process organization

Annealing of SOI layer (11 nm thick)

750°C

Holes nucleation:



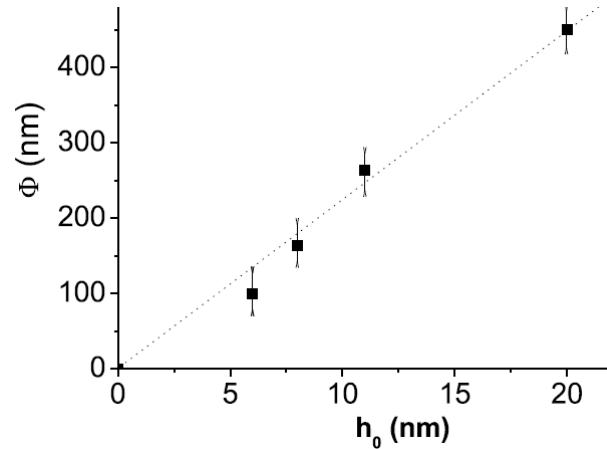
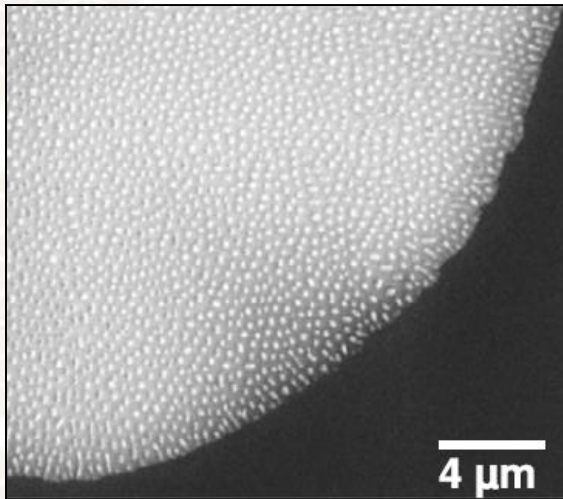
- No rim
- No edge faceting
- Matter diffusion:

$$x(t) \propto t^{0.45} \approx t^{0.5}$$

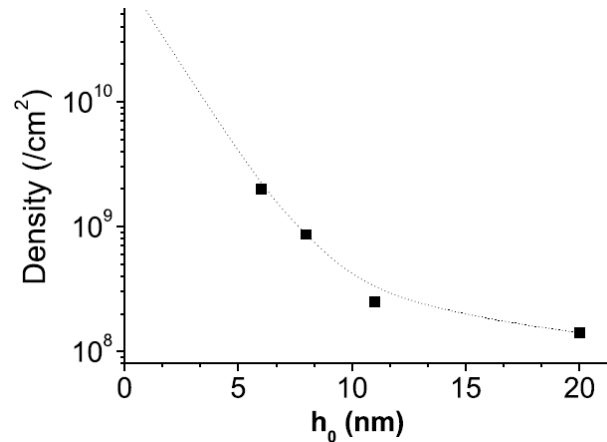
Front propagation by surface diffusion

Dewetting process organization

Dewetting process as a function of layer thickness (SOI)



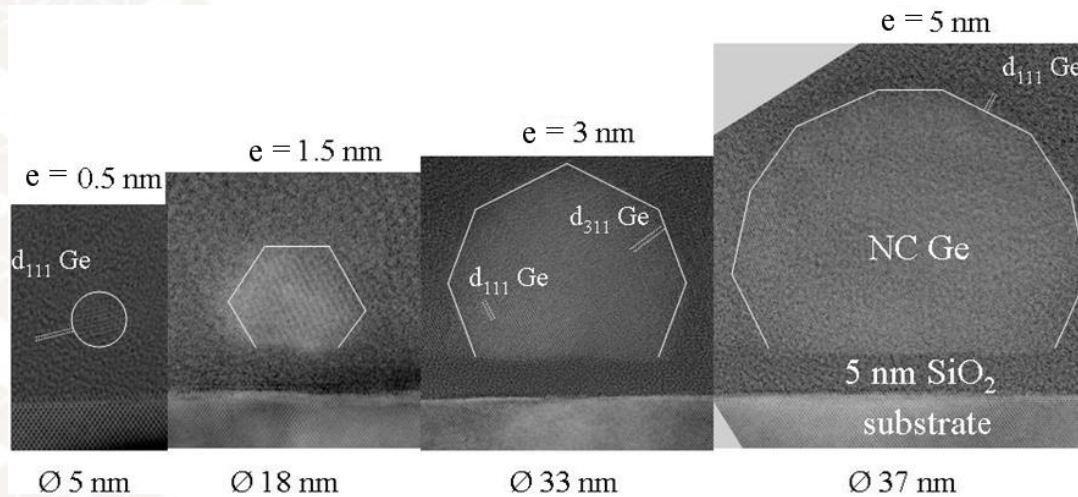
Dots diameter is almost a linear function of initial Si layer thickness h_0



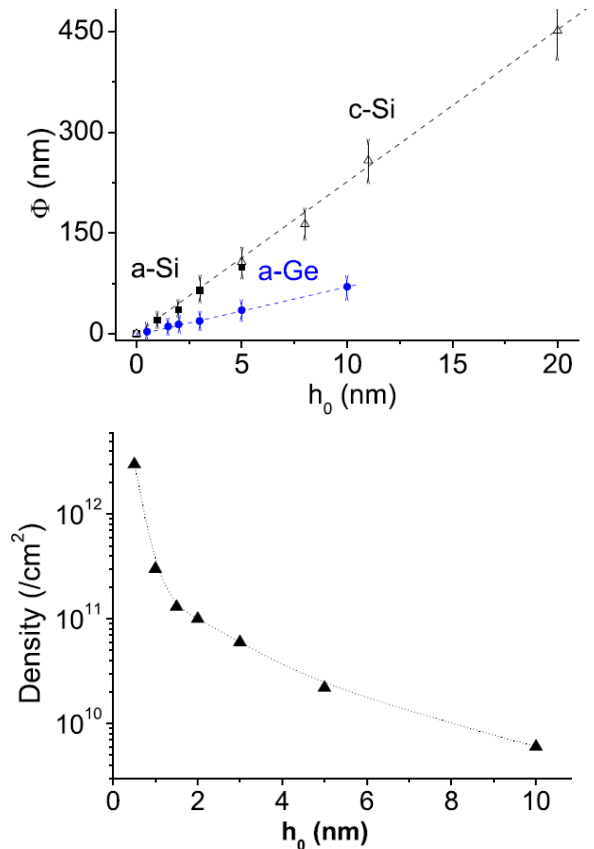
Dots density and dots diameter cannot be varied separately

Dewetting process organization

Dewetting process as a function of layer thickness (am-GeOI)



Dots diameter is a function of Ge initial layer thickness

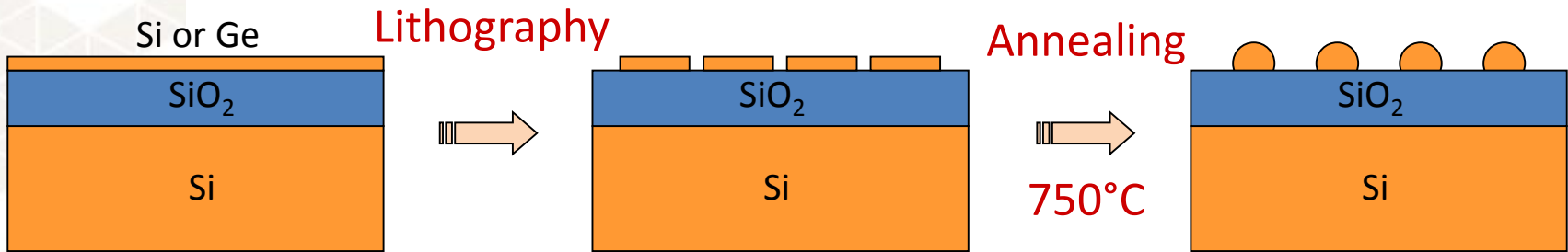


I. Berbezier, M. Aouassa, A. Ronda, L. Favre, M. Bollani, R. Sordan and A. Delobbe, *J. Appl. Phys.*, **113**, 064908 (2013)

M. Aouassa, I. Berbezier, L. Favre, A. Ronda, M. Bollani, R. Sordan, A. Delobbe, and P. Sudraud, *Appl. Phys.Lett.*, **101**, 013117 (2012)

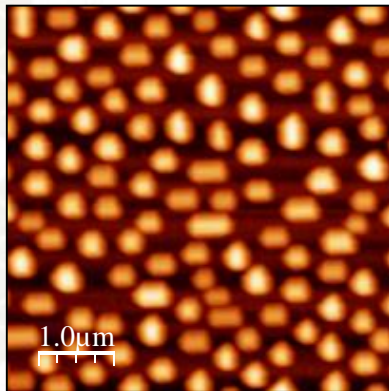
Dewetting process organization

Lithography + Annealing

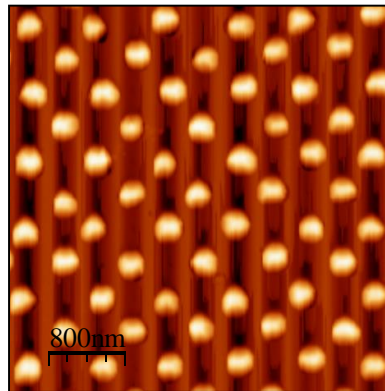


Crystalline or amorphous thin layer

Without lithography



With lithography



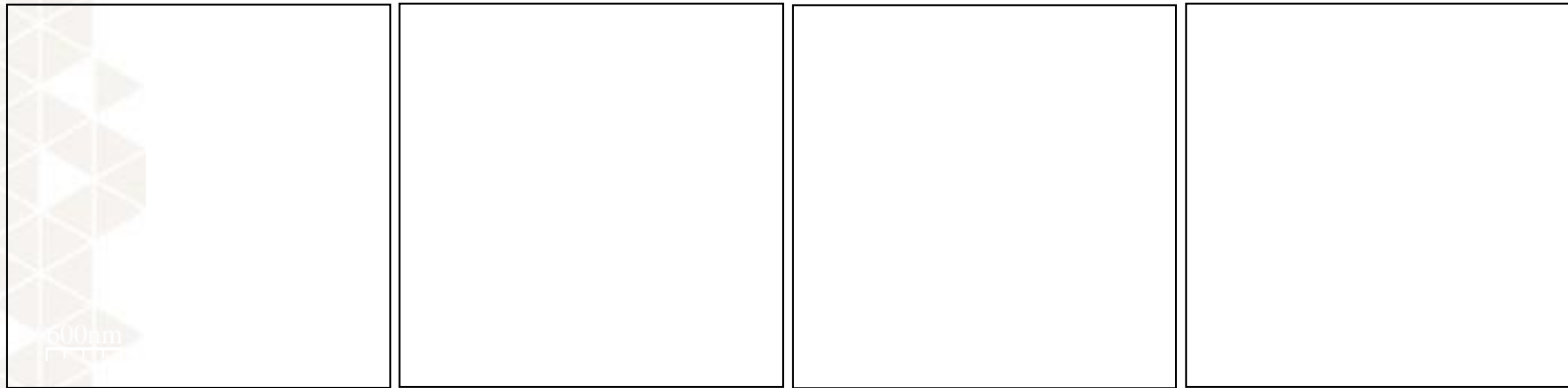
- Organization
- Narrow size distribution
- Dots shape

Dewetting process organization

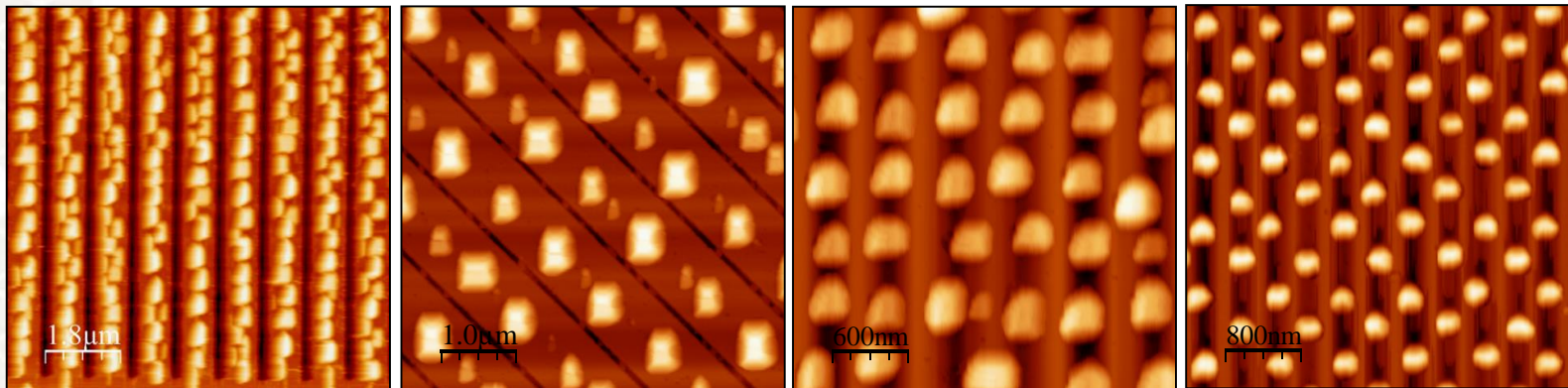
Lithography + Annealing (c-Si)

Patterns geometry influence

Before dewetting



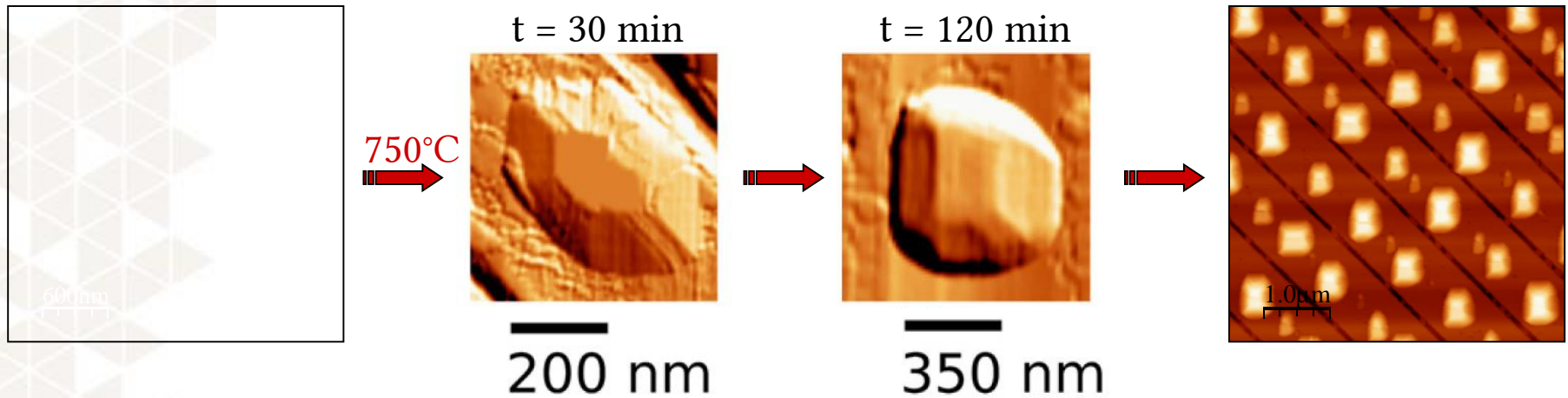
After dewetting



Dewetting process organization

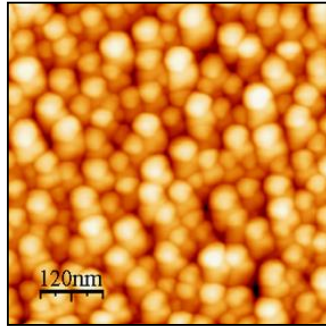
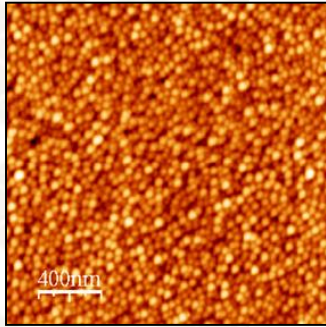
Annealing of SOI layer (c-Si)

Morphology evolution

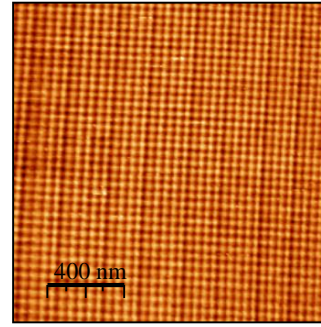


Dewetting process organization

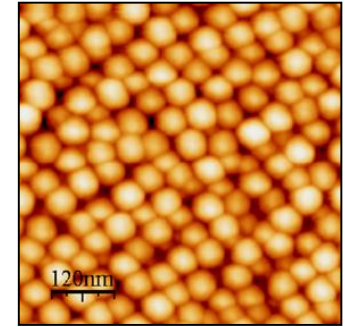
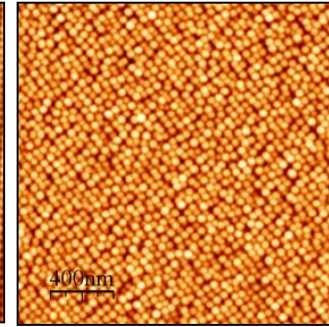
Amorphous Ge layer annealed on SiO₂ substrate : organized droplets



Planar SiO₂ Substrate



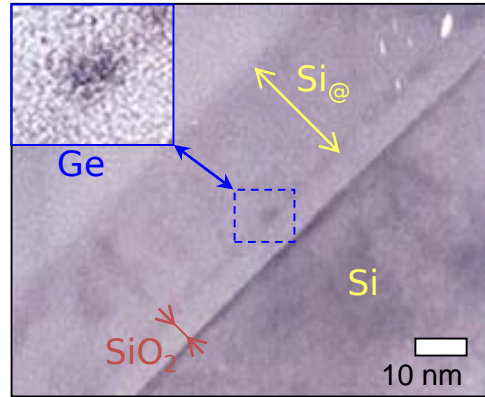
before deposition



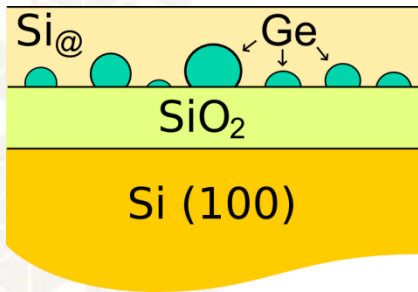
Ga ions patterned SiO₂ Substrate



TEM cross section

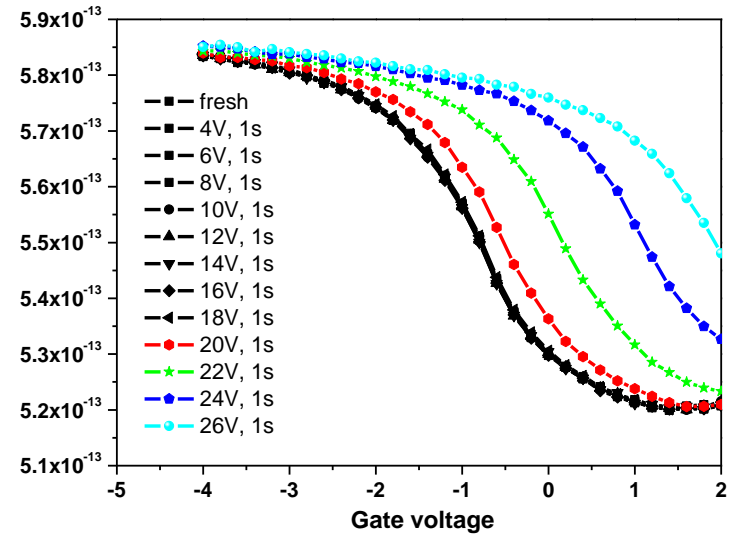


Application:
floating gate memories



Ge layer thickness	0.5 nm
Ge dots diameter (TEM)	2.5 nm

Tuned diameter

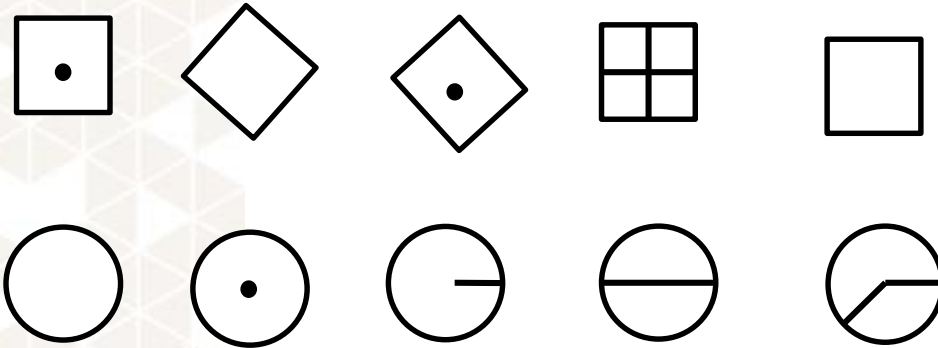


Karmous *et al.* Phys. Rev. B (2006)

Dewetting process organization

c-SI dewetting: optical dewetting (M. Naffouti & M. Abbarchi)

FIB patterns

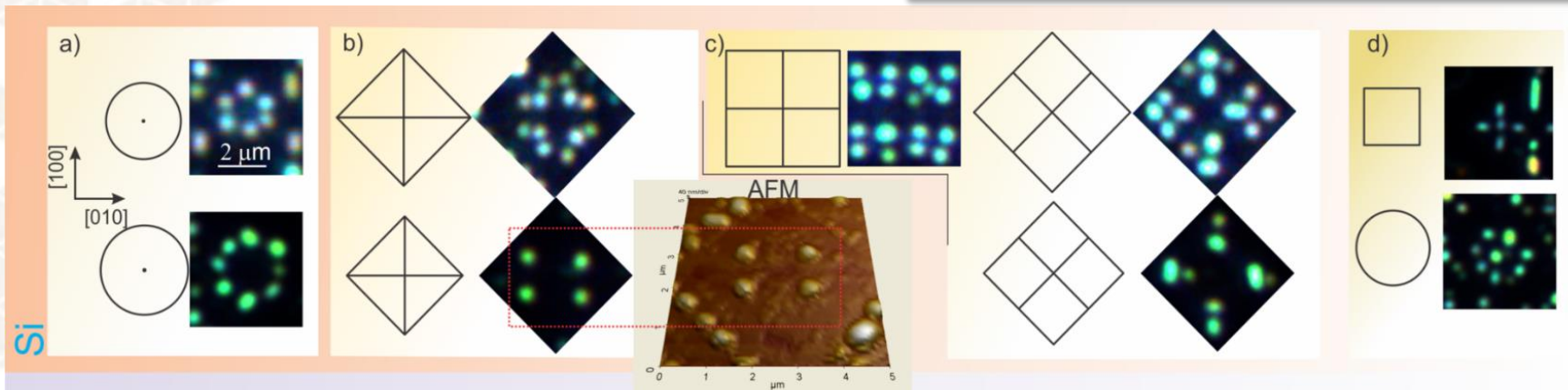


- Structures perimeter (matter conservation)
⇒ Dots size

- Dewetting front motion
⇒ Dots on pattern center

- Adding punctual defect
⇒ Dots position control

- Orientation pattern/crystallographic axes
⇒ Dots position



Dark field images of a partially dewetted SOI layer

SOI substrate (Si: 12 nm / SiO₂: 25 nm), 800°C - 60 minutes annealing)

M. Abbarchi *et al.*, ACS Nano, 8 (11), 11181 (2014)

Synopsis

Introduction

- Usual Si cleaning process
- Bottom-up is not, yet, efficient

Ion beam lithography

- Fast review of lithography technics
- Focused Ion Beam principles
- High resolution
- Limits: surface and volume defects

Using Focus Ion Beam for surface preparation

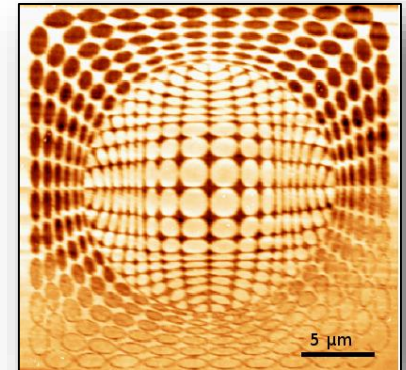
- Gold dewetting
- Au dots
- Silicon dewetting

Conclusion

Conclusion

FIB nanopatterning

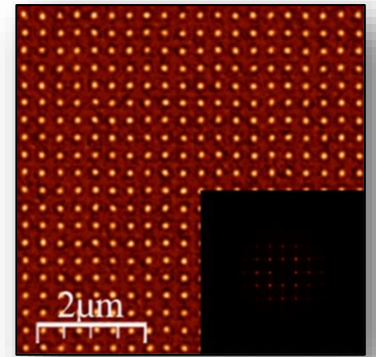
- **Critical parameters**
 - ❖ Minimum dose: swelling vs milling
 - ❖ Ion focusing
 - ❖ Excavated volume: linear with current and time
 - **Sacrificial layer**
 - ❖ High resolution: 15 nm diameter
 - ❖ **Compatible with Au salt galvanic displacement**
-
- **VLS Nanowires growth ready**



Conclusion

FIB assisted Au catalyst dewetting

- **Critical parameters**
 - ❖ Initial Au layer thickness
 - ❖ Pitch pattern
 - ❖ Dewetting temperature
 - **AuSi droplets**
 - ❖ Perfect ordering
 - ❖ Narrow size distribution
 - ❖ VLS Nanowires growth ready
-
- Extendable to many metal catalyst systems



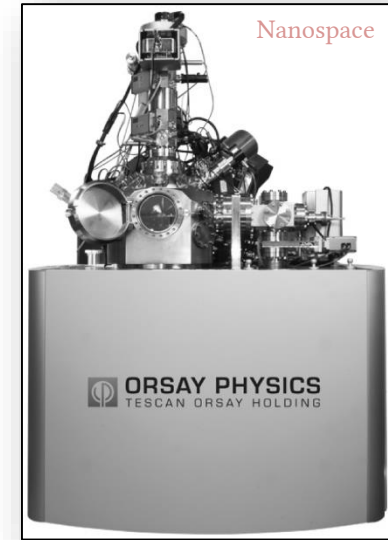
Conclusion

Next FIB generation

- **Ion beam column**
 - ❖ Enhancing optics resolution
 - ❖ Variable working voltage: limit induced defects

- **UHV compatible**
 - ❖ Milling and MBE growth successive steps
 - ❖ No oxidation
 - ❖ No contamination

- **Ultinatool project** (IM2NP, LAAS, Orsay Physics, Riber)



Acknowledgements

M. Abbarchi



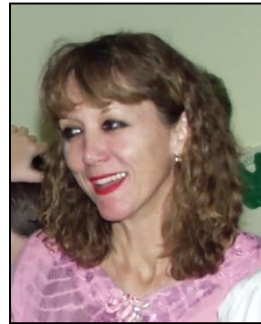
(Assistant professor)

J.-N. Aqua



(Assistant professor)

I. Berbezier



(Professor)

T. David



(Post Doc.)

A. Ronda



(Engineer)

A. Benkouider



(PhD)

J.-B. Claude



(PhD)

K. Liu



(PhD)

M. Naffouti

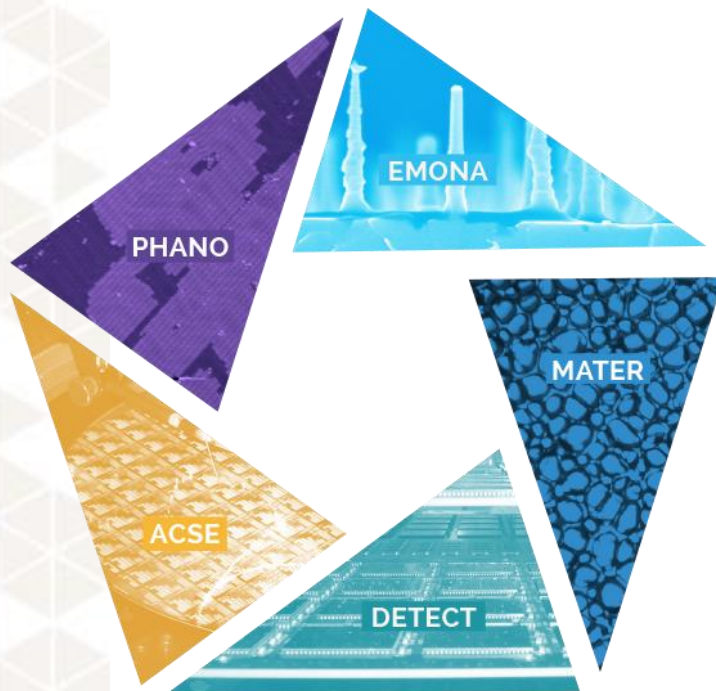


(PhD)



Institut **M**atériaux **M**icroélectronique **N**anosciences **P**rovence

Thank you for your attention



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