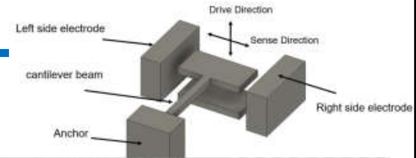
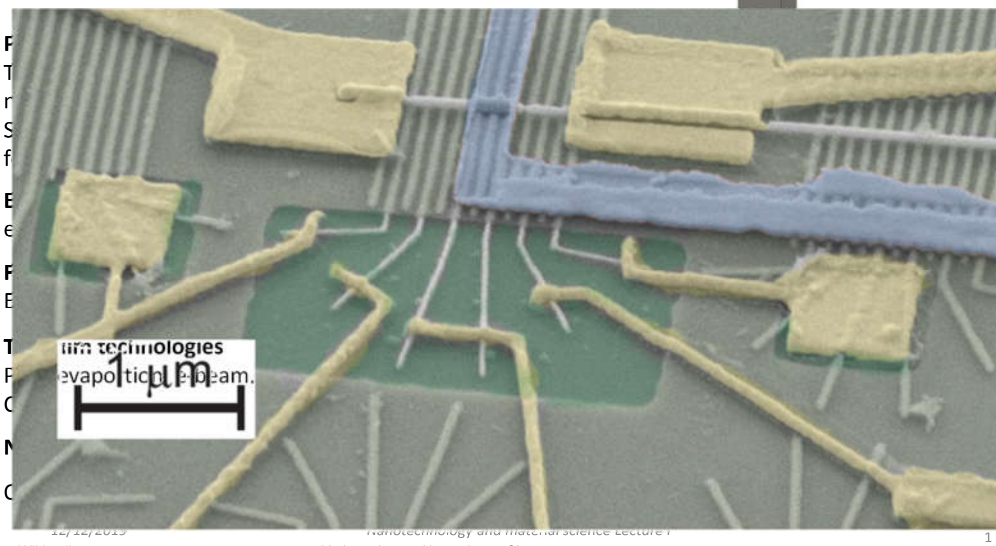
Top down techniques

„Top down means proceeding to build like a sculptor, chipping away at a block of marble to produce a statue. ”
Typical example: MEMS systems (see talk of P. Fürjes, MFA)





12/12/2019 Nanotechnology and material science Lecture I 1

Wikipedia Lindsay : Intro to Nanoscience, Chapter 5

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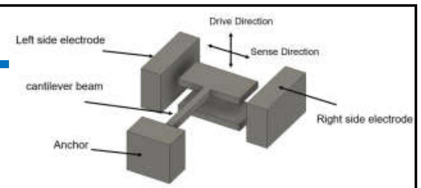


(Up)Principle structure of a MEMS gyroscope
(Down) State-of-the-art gyroscope MEMS sensor L3G4200D
http://www.memsjournal.com/2011/01/motion-sensing-in-the-iphone-4-mems-gyroscope.html
https://hobbydocbox.com/Radio/73640526-Fabrication-testing-and-characterization-of-mems-gyroscope.html

Photolitography
Typical first step to make nanostructures. Interfacing macro to micro. Diffraction limit of lenses 0.5um.
Still devices with 10nm resolution is also accessable. Only in a few semiconductor fabs. (see intro talks)

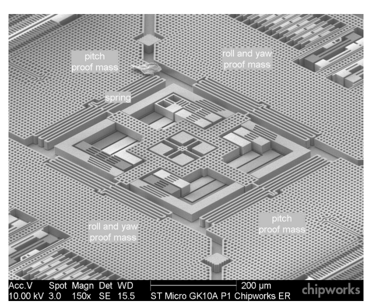
EBL (Electron beam lithography)
exposes lithography resists with very fine electron beam

FIB (Focused Ion Beam), HIB (Helium ion beam)
Etch materials directly by bombarding them with energetic ions

Thin film technologies
PVD: evaportion, e-beam, sputtering, MBE
CVD

Nanoimprint lithography

Others .. STM, AFM lithography, …



12/12/2019 Nanotechnology and material science Lecture I 2

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Photolithography

Toolkit of semiconductor fabrication plant
Steps: oxidation, masking, implantation, Etching, Metallization, Lift-off

Resist with 100-2000nm thickness is exposed to light
Negative resist cross-links the resist
Positive resist induces breaks, scission of chains (e.g. PMMA) → soluble

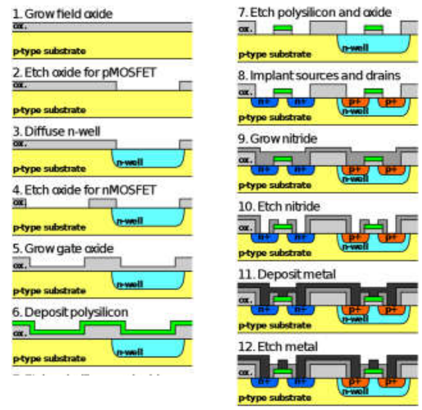
With optical setup the light is illuminated through a mask containing the pattern and projected to the resist with ¼ reduction.

Diffraction limit of the smallest structures:

$$r = \frac{k\lambda}{\mathrm{NA}}$$

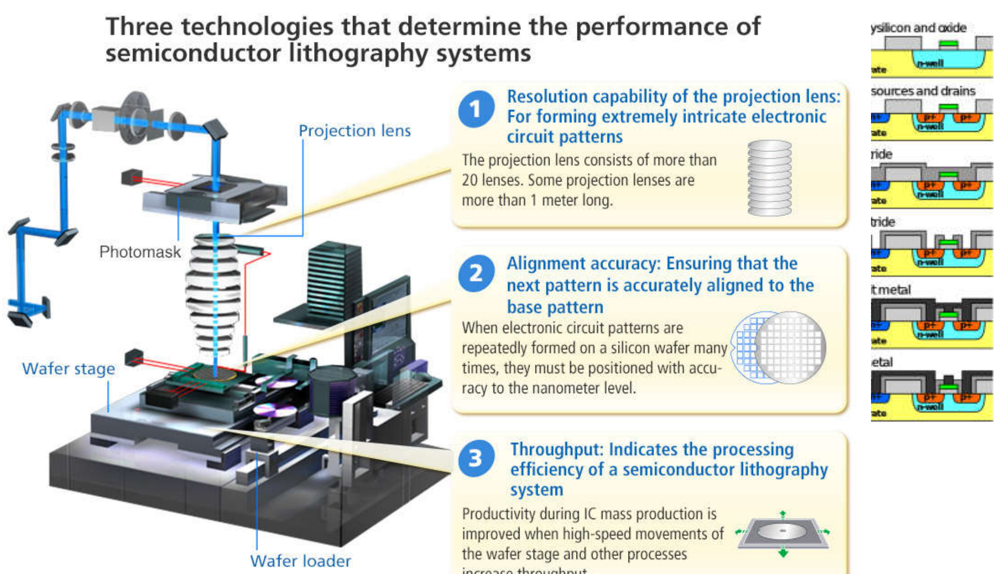
k=0.5-1, NA numerical aperture of the projector lens.
Resolution in semiconductor industry 10nm
Excimer lasers are used: deep UV λ<200nm, high light intensity.

(Up) Wikipedia



Wikipedia *Lindsay : Intro to Nanoscience, Chapter 5* *see more detail in Gabor Kiss: Micro and Nanotechnology*

Photolithography



https://www.nikon.com/products/semi/technology/story03.htm

Wikipedia *Lindsay : Intro to Nanoscience, Chapter 5* *see more detail in Gabor Kiss: Micro and Nanotechnology*

Extreme Ultraviolent Lithography

Principle:

UV light with λ=13.5nm used for exposing.
Excimer laser is used to excite tin or xenon plasma. Not a coherent source.
Challenges: make optics ,create dust free environment.
Optics: No transparent material for this light!
Optical system is built out of mirrors (instead of lenses).
Mirrors are also difficult to make, using constructive interfere of multilayers.

4x reduction system as for optical lithography

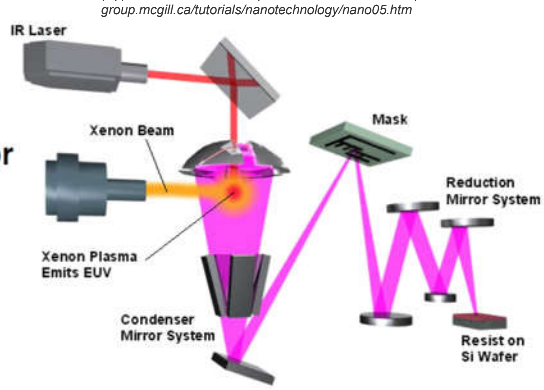
Feature | Semiconductors | Nanotechnology

5 Jan 2018 | 16:00 GMT

EUV Lithography Finally Ready for Chip Manufacturing

TSMC, Intel and Samsung will use for the next generation of 7nm node.
Resolution of 13nm with ASML Twinscan NXE3400B (2018)

(Up) Wikipedia, http://barrett-group.mcgill.ca/tutorials/nanotechnology/nano05.htm



Wikipedia *Lindsay : Intro to Nanoscience, Chapter 5*

Electron beam lithography

Spot size of SEM few nm in a large field of view
E-beam used to scission of resist like PMMA
EBL is a SEM with fast beam blanking feature

Typical process: metallization see figure.

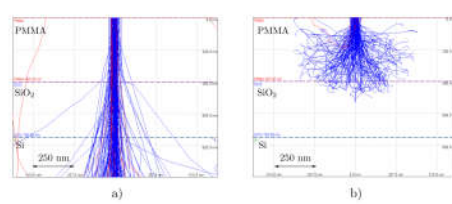
Resolution:
It is not limited by spot size, but by proximity effect.
Secondary electrons are generated by e beam in the sample and backscattered ones expose the resist.
E.g. 20kV secondary electrons reach a circle of ~2um.

Typical resolution is <100nm, 10nm is achievable.

Issue: sequential, throughput is small.
But for research or writing a mask it is a perfect tool.

(Up) principle of EBL

(Bottom) Monte Carlo simulation of electrontrajectories for a) 35kV and b) 5kV accelaration voltage G. Fülöp BSc (09)



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Thin film technologies

Widely used techniques.
High and ultra high vacuum systems are required!

Example: Number of molecules striking the surface per area and unit time (N_S):

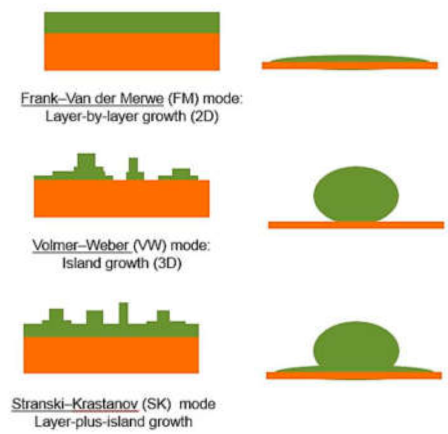
$$N_s = \frac{1}{2\sqrt{3}}\rho\sqrt{\langle v^2\rangle}, \qquad \rho = N/V = P/k_B T$$

using for kinetic energy $\frac{1}{2}mv_x^2 = \frac{1}{2}k_BT$:

$$\sqrt{\langle v^2\rangle} = 1.58 \times 10^4\sqrt{\frac{T}{M}}\ \text{cm/s}$$

where M is the mass of the molecule in atomic mass and T is the temperature in K, p is the pressure.
E.g. for oxigen at room temperature at p=10-6mbar: N_S= 1 monolayer/sec.
→ For fine structures 10-9mbar is a min. requirement.

Different modes of crystal growth on surface.
Typically outcome is polycrystalline.
Growth of epitaxial layers requires special conditions.



Depending on interface and bulk energy balances, temperature, deposition rate, energy of arriving atoms differnt modes of crystal growth could take place. Three main modes are shown at atomic level (left) and bulk level (right).

https://www.mdpi.com/2073-4352/7/6/178

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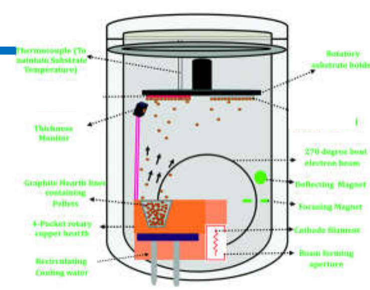
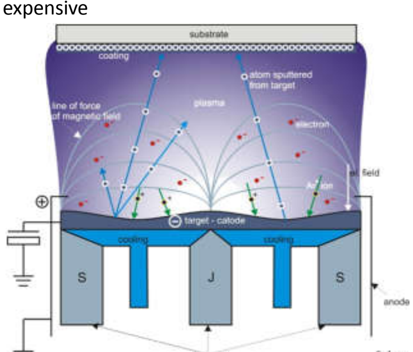
Physical Vapour Deposition (PVD)

Thermal evaporation
Metal is paced in a tungsten boat and heated electrically.
Sample is clamped to a stage at a distance from the target.
Quartz crystal monitor is used (see lab course on piezo resonance freq.).

Electron-beam deposition
Energetic e-beam is focused on the metal target, which induces evaporation. Local heating of metals. Pros/contras: + It allows to evaporate refractory metals (Ti, Nb, W, Mo…). + No contamination from the boat. +Wide range of evaporation rate – risk of X-ray damage - expensive



(Up) Electron beam deposition system https://pubs.rsc.org/en/content/articlelanding/2016/an/c6an0092k/unauth#!divAbstract



Sputter deposition
Energetic ions are used to bombard the surface of the target. Typically Ar (O, N) atoms are ionized by electrons, creating a plasma. Ar ions are accelerated by voltage towards the target, and target atoms kicked out and scattered towards the substrate. Magnetron sputtering system use magnets to trap free e-s close to the target and ionize Ar there. → Higher sputtering rate, low e flux on substrate.
Pros/contras: - High energy target atoms + easier to control composition (e.g. co-sputtering, NbN) + step coverage is better – non-directional
E-beam v.s. sputter deposition depends on particular applications.

Schematic structure of a magnetron sputtering system. http://www.umms.sav.sk/6493-en/physical-vapor-deposition/, http://www.ajaint.com/what-is-sputtering.html

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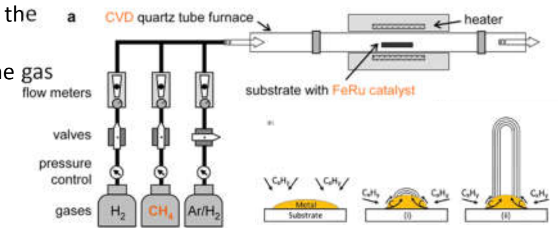
Chemical Vapour Deposition (CVD)

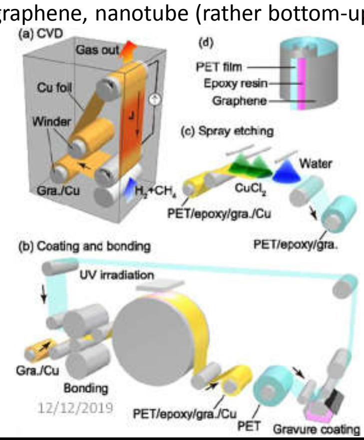
Creation of reactive chemical species close to the surface to be coated
E.g. growth of polycrystaline silicon from silene gas (600C and 1mbar)

$$SiH_4 \rightarrow Si + 2H_2$$

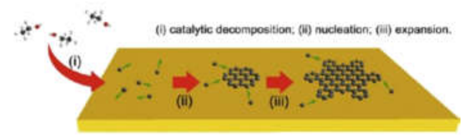
E.g.2: Growth of carbon nanostructures: graphene, nanotube (rather bottom-up)



(Up) Typical CVD system to grow carbon nanostructures. Calayst particles with e.g. CH4 gas creates carbon nanotubes. The schematic growth process is shown bellow.
https://www.nano.physik.uni-muenchen.de/nanophysics/research/rep11.html
https://www.semanticscholar.org/paper/Chemical-vapor-deposition-of-carbon-nanotubes%3A-a-on-Kumar-Ando/8cad65216fe922b14c5c947330cc791341f621fb



(Down) Basic process of CVD growth of graphene on Cu substrate from CH4.
(Left) Continous roll-to-roll CVD growth and transfer of large area graphene
https://www.sciencedirect.com/science/article/abs/pii/S0379677915300138
https://www.researchgate.net/figure/Schematic-of-continous-roll-to-roll-CVD-growth-and-transfer-of-large-area-graphene_fig5_249286739



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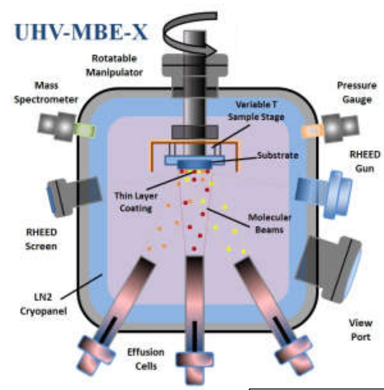
Molecular Beam Epitaxy (MBE)

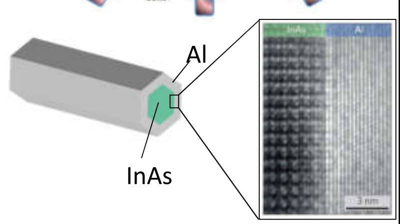
(Up) Typical MBE setup http://www.adnano-tek.com/products-ver-1.html

Epitaxialy growth: sequential deposition of crystalline layers of particular atoms.
Limited in combination of subtrate and growth material. Lattice mismatch (see intro).
E.g. GaAs/AlGaAs, Si/SiGe, InAs/AlSb, InGaN/GaN
-> Band gap engineering.

Advanced thermal evaporator system.
- UHV environment.
- Substrate: cleaned, single crytal face
- Atomic beam source placed far
- REED sensor (Reflection high-energy electron diffraction)
  Monitor thickness by diffracted beam intensity caused by reflections from the interface and surface layers
  Sub atomic layer resolution

Frank van Der Merve crystal growth competes with VW and SK growth.





Reflection free superconductor/semiconductor interface with epitaxial growth of Al on InAs nanowires
P. Krogstrup, Nature Materials 14, 400 (2015)

Lindsay : Intro to Nanoscience, Chapter 5 see more detail in Gabor Kiss: Micro and Nanotechnology

Molecular Beam Epitaxy (MBE)

(Up) Band gap engineering, http://gorgia.no-ip.com/phd/html/thesis/phd_html/node4.html
about nano see also Nature Reviews Materials **2,** 17070 (2017)

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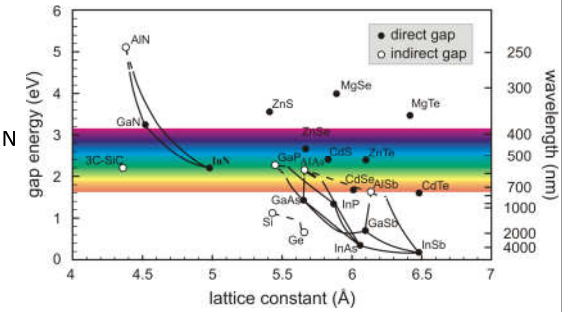

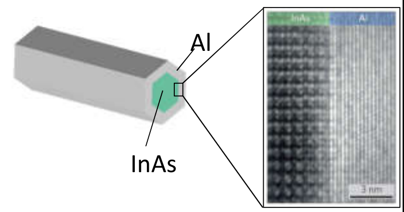

(Up) Example of MBE growth: Reflection free superconductor/ semiconductor interface with epitaxial growth of Al on InAs nanowires P. Krogstrup et al. , Nature Materials 14, 400 (15)

Lindsay : Intro to Nanoscience, Chapter 5 see more detail in Gabor Kiss: Micro and Nanotechnology

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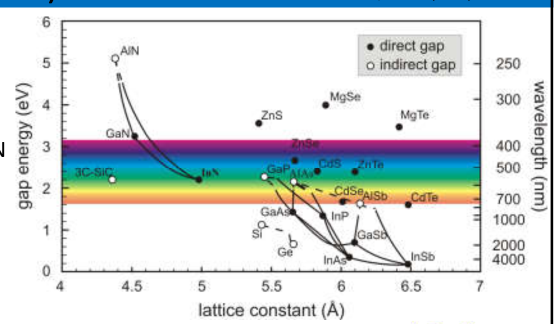

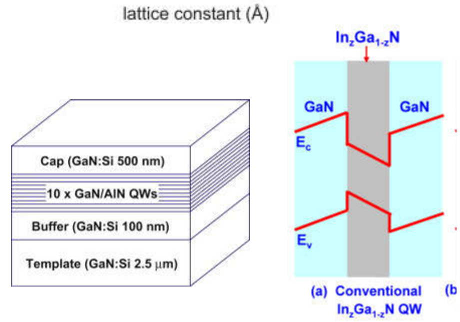

(Up) Example2 of MBE growth: optical quantum wells
https://www.osapublishing.org/oe/abstract.cfm?uri=oe-19-s4-a991
https://doi.org/10.1016/j.mejo.2008.07.065

Lindsay : Intro to Nanoscience, Chapter 5 see more detail in Gabor Kiss: Micro and Nanotechnology

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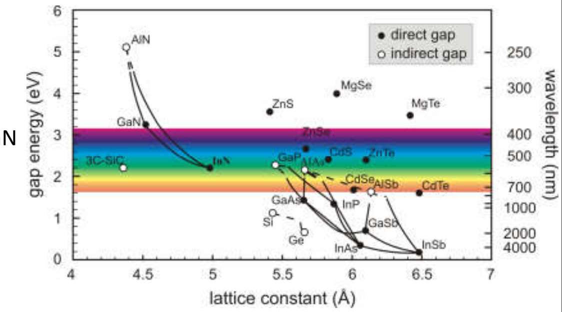

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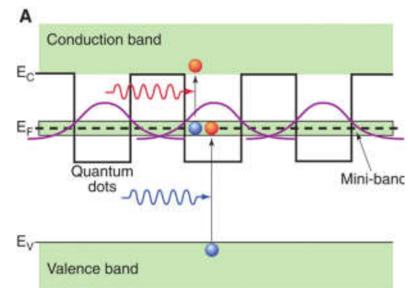


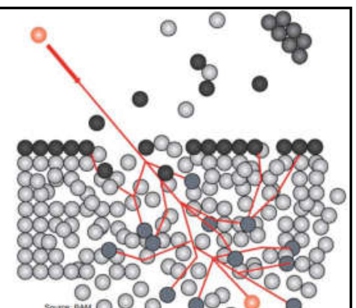
(Up) Solar cell application, Possible methods of circumventing the 31% efficiency limit for thermalized carriers in a single–band gap absorption threshold solar quantum conversion system, Lewis Science (2007) DOI: 10.1126/science.1137014

Lindsay : Intro to Nanoscience, Chapter 5 *see more detail in Gabor Kiss: Micro and Nanotechnology*

13

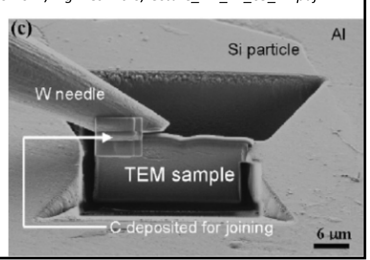
Focused Ion Beam (FIB)

Accelerate ions that chip materials away to form nanostructures. Focused sputtering.
Similar to an e-beam lithography system, but ions are accelerated instead of electrons. Typical ion energy: 5-30keV
Large mass difference between e and ion m_e-> $M_{ion} \approx 10000 x m_e$
→ $\lambda_{ion} << \lambda_e$
→ Lorenz force has weaker effect, mostly electrostatic focusing
→ Less penetration than for e-s.
→ Large momentum transfer to target
Momentum transfer kicks atoms out. → Localized ion milling with ~ few 10nm resolution limited by volume.



(Up) Ion solid interaction (Advantages/disadvantages)
- Ion implantation, - Amorphisation, - Scattering, + Creation of secondary electrons (→ imaging) + Removal of surface atoms, clusters, + Creation of secondary ions (imaging)
(Down) TEM sample fabrication by FIB
http://www.uni-stuttgart.de/mawi/aktuelles_lehrangebot/documents/LSI/SS-2017/HighResMicro/lecture_FIB_14_09_17.pdf

Mostly Ga+ ions: easy to melt, Ga is middle of periodic table → momentum transfer is optimized for most elements. (Like snooker.) Note: - Implantation of Ga ions.



Crossed beam systems SEM-FIB. Used:
- precise straight cutting of TEM samples.
- Electron beam assisted CVD e.g. C or Pt can be nanowritten on the surface.
- IC editing.

Lindsay : Intro to Nanoscience, Chapter 5 *Available at ELTE or MTA EK MFA*

Focused Ion Beam (F

milling with ~ few 10nm resolution

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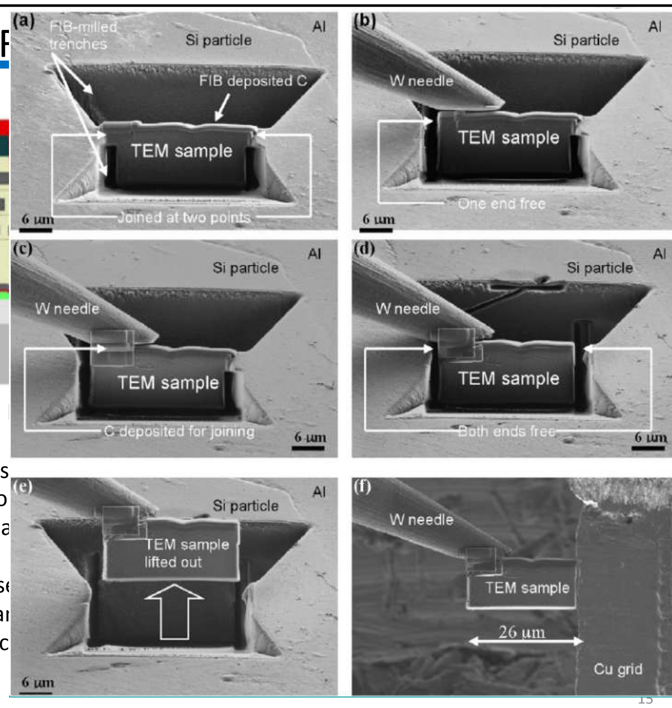
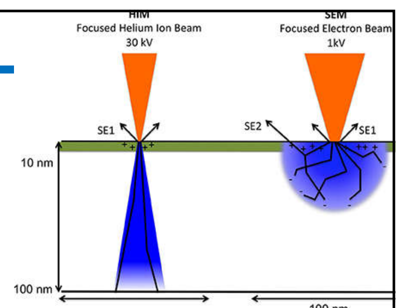
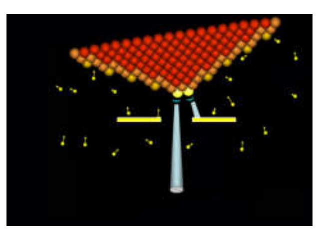


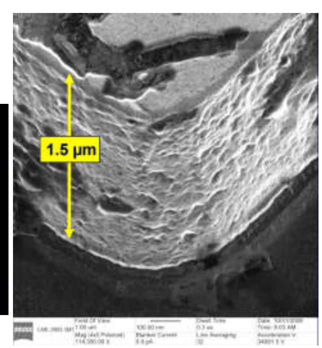
FIB - Helium ion beam (HIM)



It uses advantages of FIB in imaging.
Observation possible at sub-nanometer scale
Advantages compare to SEM:
+ wave length limit better than for SEM
+ Smaller interaction volume than for SEM, secondary electrons only generated from the surface -> sharp images
+ Due to light weight of helium, negligible damage of the sample structure compare to FIB
+ Special, well focused source: helium ions generated in a region of a single atom.

Performance:
→ Surface resolution 0.24nm (Zeiss 2009) much better than SEMs.
→ Depth of field is 5x better than for SEM (small convergence angle)





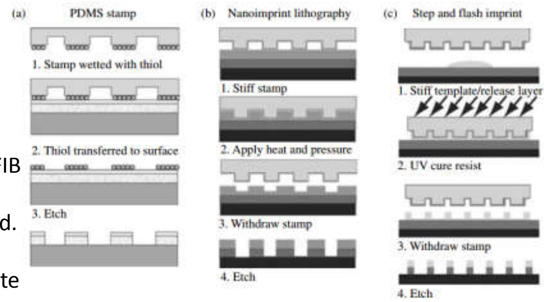
(Up) Secondary electron generation comes from a much smaller volume for HIM as for SEM. Only surface layer.
https://www.researchgate.net/figure/Schematic-comparing-between-helium-ion-beam-and-electron-beam-and-charge-distribution_fig9_267743550
(Left) Atomic sized source. Voltrage biased tip at cryo temperature. (Right) Example of field of depth Image size 1um, depth 2.5um.
DETAILS:
https://microscopy-analysis.com/editorials/editorial-listings/principles-and-applications-helium-ion-microscopy
https://www.nist.gov/sites/default/files/documents/pml/div683/conference/Postek_2009.pdf
Wikipedia

Nanoimprint lithography (NIL) -Stamping

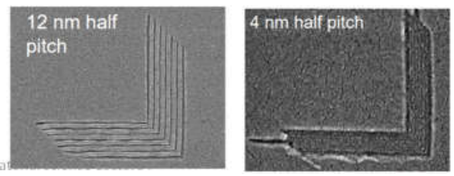
Similar to original lithography invented by Gutenberg.
Example 1 (see Panel a in Figure)
- With PDMS ~10nm resolution was achieved.
- Mold for the stamp is first cut directly into a hard substance like silicon using e-beam or FIB lithography
- PDMS is poured on the mold and cross-linked.
- Flexible silicon rubber is resulted.
- Can be used as an office ink stamp to deposite chemicals in nanopatterns.

Adv/Disadvantages: +No wavelength limit (High resolution) +Parallel process + Cheap – Not as reliable

It is used to produce various nanoscale structures e.g. for HD-DVD, photonic crystals, liquid crystal alignment in displays, patterning of nano resists, polimer reactors, microfluidics etc.



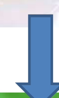
(Up) a) Imprint lithography to transfer chemicals. B) Example of nanoimprint lithography: a hard stamp is pressed to a soft resist, thinner region of the resist etched away and used as a mask. C) Similar to b) but liquid resist is used, which is crosslinked by light. Later on it is used as a mask.
(Down) HIM lithography defined mold imprinted in resist and 2nm Pt coated. Image is made by SEM which does not have the proper resolution to resolve.
http://sites.ieee.org/sfbanano/files/2016/07/NanoCON-Day-1-Wu.pdf

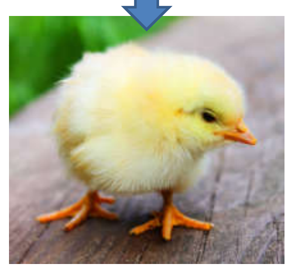


Wikipedia *Lindsay : Intro to Nanoscience, Chapter 5*

Bottom-up approach

„making complex nanostructures starting from the random collisions of the components dissolved in a solvent. „
E.g. biology: from chaotic soup of an egg → macroscopic organism





Goal/dream: Complex electronic circuitry that assembles itself from a chemical soup

Mechanism: Entropy driven processes, special balance between:
- Entropy at a temperature
- Binding energies

in order to have
- reasonable stable structures
- weak enough bonds to allow the system to explore large number of configuration to get to the desired configuration of lowest free energy (e.g. correct errors)

Carbon is a perfect starting ingredient, since it has large variety of way to bond to other atoms (with 1, 2, 3, or 4 electrons). Therefore versatile structures can be built up.

Examples: DNA origami (see Introductiory part)

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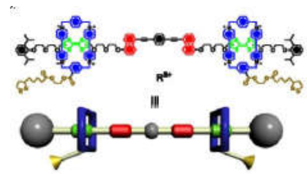
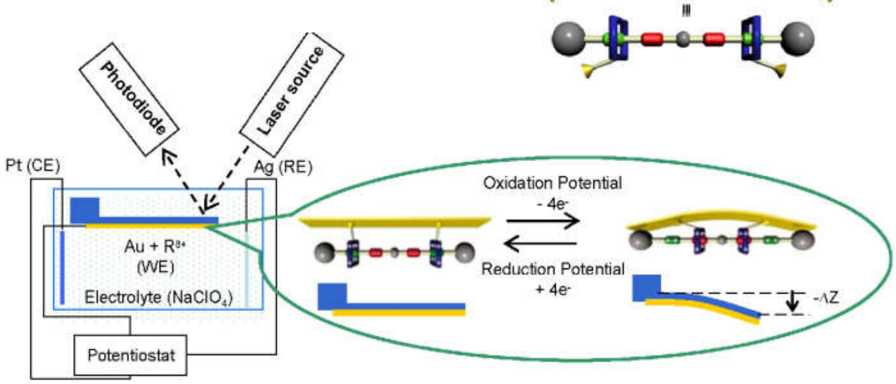
Power of organic synthesis

Example I: Nano-mascle based on rotaxane
It is based on a pair of rotaxane molecules (molecules free to slide on a shaft, blue) that switch from position green to position red as the oxidation state of the central molecule (TPR, gray middle) is changed from 8+ to 12+.
Using sulfur anchoring groups to gold surface NEMS cantilever can be bended.





Hybrid NEMS acutator based on electrocemical activation
https://www.semanticscholar.org/paper/Molecular-Muscle-based-(NEMS)-Juluri-Kumar/3cc658033253f35a740fc44148207bc6a7b71a43

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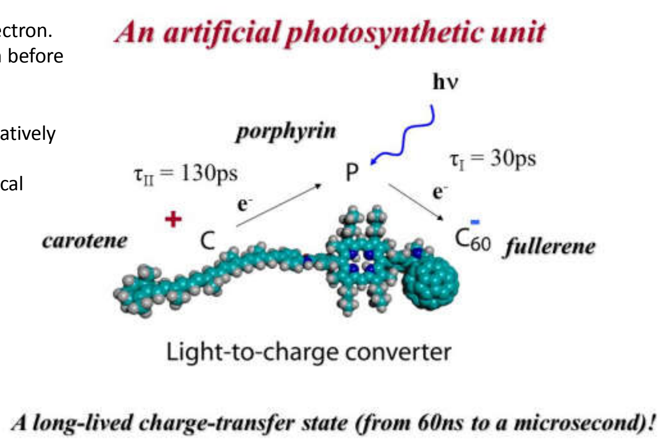
Power of organic synthesis

Example II: Light-to-charge converter

- Porphirin absorbs light, by exciting electron.
- Fullerene grabs the excited electron before decaying to ground state
- Carotene inject an electron back

→ Positively charged carotene and negatively charged fullerene. Due to light, charge separation which can be used as chemical energy.



Basics of artificial photosynthesis with CPF carotene-porphyrin-fullerene structure
https://slideplayer.com/slide/5003798/

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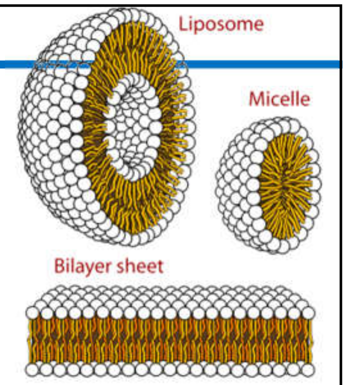
Amphiphiles in aqueous solutions



- Amphiphiles are molecules that contain both a hydrophilic and a hydrophobic component
E.g. detergents: hydrophobic component binds oily dirt while the hydrophilic part helps to pull the complex into solution.

Walls of cells: self-assembled from amphiphilic molecules.
Building blocks are phospholipids, which consist of a pair of long alkane chains (repeated units of methylenes, –CH2–) connected together by a head group that contains a number of charged atoms on the phosphate group

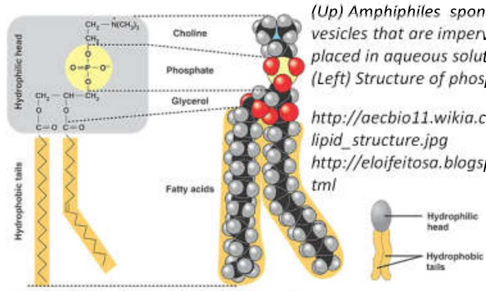
Different structures depending on the geometry of building molecules
Micelles are clusters that consist of a single layer of amphiphiles in which all the hydrophobic tails point inwards to exclude water completely while the hydrophobic heads point outwards
Liposome are bilayer spheres that enclose water
They self- assemble. With molecular dynamics the final form can be tested

$\frac{v}{a_0\ell_c} < \frac{1}{3}$ → sperical micelle

$\frac{1}{3} < \frac{v}{a_0\ell_c} < \frac{1}{2}$ → non-sperical micelle

$\frac{1}{2} < \frac{v}{a_0\ell_c} < 1$ → liposome/bilayers

where v volume of molecule, a_0 area of the head group, l_c is the length of hydrocarbon chain.



(Up) Amphiphiles spontaneously form small vesicles that are impervious to water when placed in aqueous solutions
(Left) Structure of phospholipid molecule

http://aecbio11.wikia.com/wiki/File:Phospholipid_structure.jpg
http://eloifeitosa.blogspot.com/p/research.html

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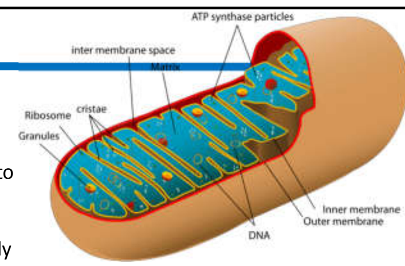
Mitochondrion

- Example 1: Mitochondrion, a **self-assembled nanochemistry machine**
Inside our cell outside of the nucleus. Largely assembled from phospholipids. Machines that allow us to use atmospheric oxygen to complete the oxidation of sugars to produce biological energy.

It has two sets of lipid bilayer membranes. Outer one is a filter, only allowing small molecules into the intermembrane space. Protons are pumped into the intermediate space, the gradiant of protons between intermembrane space and inner part drives synthesis of ATP molecules. ATP is transported out of mitochondrium and serve as source of energy.



(Up) Structure and TEM image of mitochondrion
https://www.thoughtco.com/mitochondria-defined-373367

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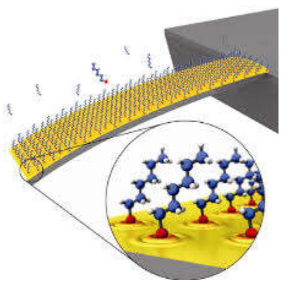
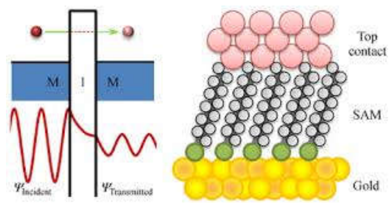
Molecular monolayers

Non interacting molecules chemically attracted by a surface
E.g. alkane chains (-CH_2-) with thiol groups (-SH) on gold surface From ethanol solution few hours a monolayer forms on the surface. Good to modify chemical property of the surface. Depending on the end group e.g. extremely hydrophobic / hydrophilic. Such molecular monolayers also used in molecular electronics for ensemble measurement.

(Down left) Alkane chain on Au surface on cantilever as biochemical sensor based on surface stress. (Down right) Way to test molecular electron transport https://www.researchgate.net/figure/Artistic-view-of-an-alkanethiol-self-assembled-monolayer-SAM-on-a-gold-coated_fig1_230938526





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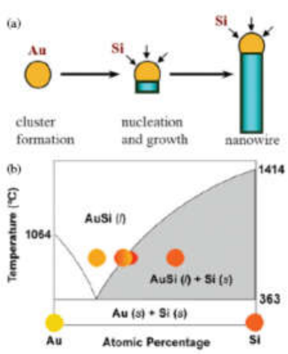
Synthesis of Nanowires



Vapor-liquid-solid (VLS) growth:

- Catalyst metal nanopartice
- Heated above the eutectic T of metal-semicond.
- Presence of vapor source of semiconductor
- Supersaturates -> nucleation of semiconductor
- Growth the NW with droplet on the top

Reactants generated:
CVD (Chemical vapour depos), MBE, MOVPE, CBE

Competing interfaces: (p, flow, T, reactant …)

- Liquid-solid: elongation of the NW, metal particle determinates the diameter
eg.: possible 3nm diameter Si
well defined crystallographic orientation

- gas-solid: thickening in the radial direction

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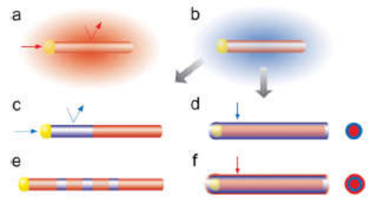
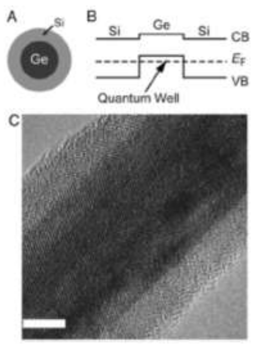
Synthesis of Nanowires





Crystalline heteroepitaxy can be archived

Depending on preferred reaction interface:

- **Radial heterostructures**, core/shell (b→d)

eg.: Si/Ge core/shell structure -> hole gas
GaN ->light emitting diodes, SiO/Si

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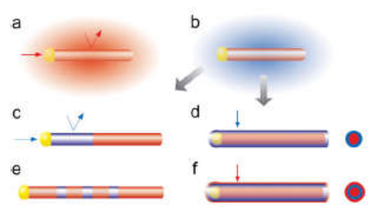
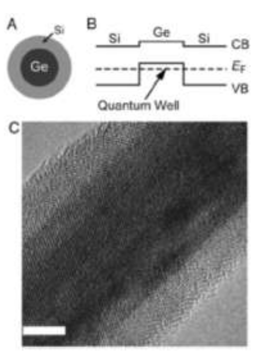
Synthesis of Nanowires





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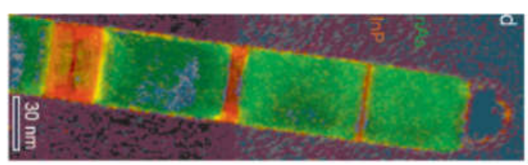
- **Axial heterostructures** (b→c)

with Au nanoparticle wide range III-V, IV
eg.: InP/InAs interface of few atomic layers
0.6eV conduction band offset



Nano Lett 2, 87 (2001)

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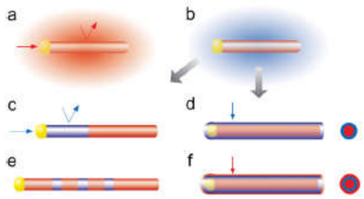
Synthesis of Nanowires



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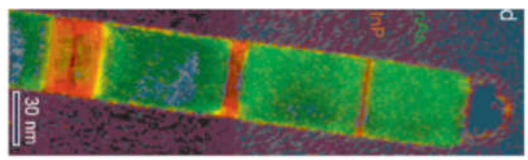

- High level of control on dimension, chemical composition
- Band structure engineering
- Combine heavily mismatched materials up to >11% mismatch
- Monolithic integration of III-V into Si
- Epitaxial growth with defined crystal symmetries

Nano Lett 2, 87 (2001)

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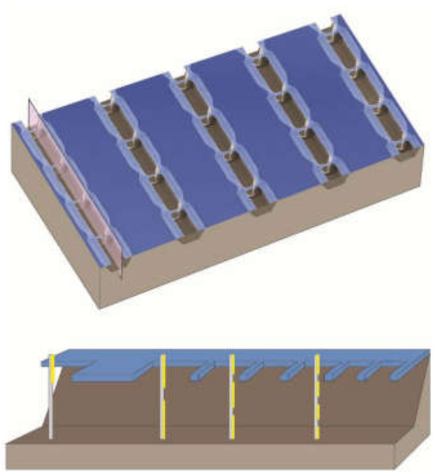
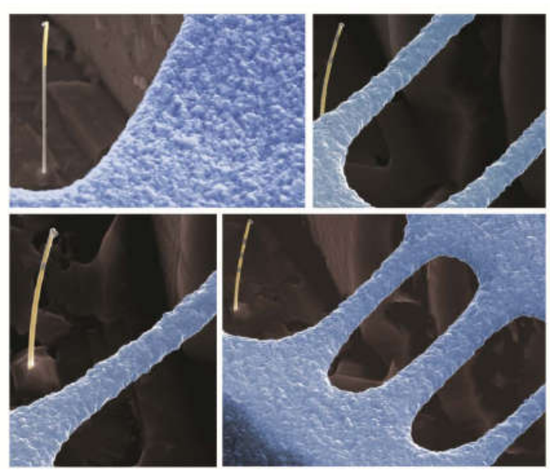
Synthesis of Nanowires

InAs nanowires with epitaxial Al shell in certain sections. Also InP barriers are possible.





Unpublished from J. Nygard

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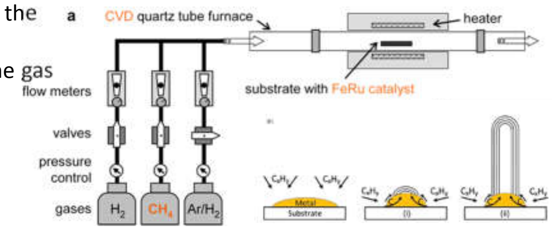
Chemical Vapour Deposition (CVD)

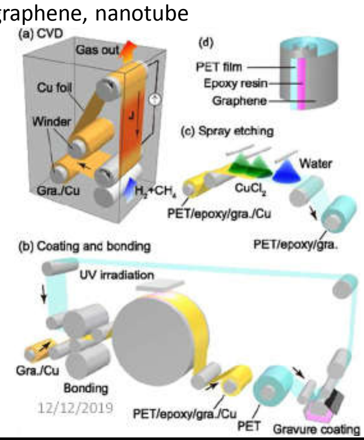
Creation of reactive chemical species close to the surface to be coated
E.g. growth of polycrystaline silicon from silene gas (600C and 1mbar)

$$SiH_4 \rightarrow Si + 2H_2$$

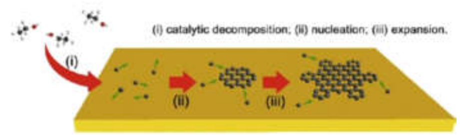
E.g.2: Growth of carbon nanostructures: graphene, nanotube



(Up) Typical CVD system to grow carbon nanostructures. Calayst particles with e.g. CH4 gas creates carbon nanotubes. The schematic growth process is shown bellow.
https://www.nano.physik.uni-muenchen.de/nanophysics/research/rep11.html
https://www.semanticscholar.org/paper/Chemical-vapor-deposition-of-carbon-nanotubes%3A-a-on-Kumar-Ando/8cad65216fe922b14c5c947330cc791341f621fb



(Down) Basic process of CVD growth of graphene on Cu substrate from CH4.
(Left) Continous roll-to-roll CVD growth and transfer of large area graphene
https://www.sciencedirect.com/science/article/abs/pii/S0379677915300138
https://www.researchgate.net/figure/Schematic-of-continous-roll-to-roll-CVD-growth-and-transfer-of-large-area-graphene_fig5_249286739



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Lindsay : Intro to Nanoscience, Chapter 5 see more detail in Gabor Kiss: Micro and Nanotechnology