Nanotechnology and Material Science

Lecture VIII

Department of Physics, BME 2024.

Top Down & Bottom Up Approaches

Onsite course, BMETE11MF58

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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)





Wikipedia

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Top down techniques

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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 e-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, ...

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Wikipedia



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



Lithography



https://avidipta.art/a-brief-history-of-lithography/ Nanotechnology and material science Lecture I

Photolithography

Toolkit of semiconductor fabrication plant

Steps: oxidation, masking, implantation, Etching, Metallization, Lift-off in localized areas.

How do we define the areas?

Coat wafer with polymer "resist", 100-2000nm thick When exposed to light:

- Negative-type resist: light cross-links the resist → insoluble
- Positive-type resist: light induces breaks, scission of chains (e.g. PMMA) → more soluble

With an optical setup the light **is illuminated through a mask** containing the pattern and projected to the resist with ¼ reduction (exposure) Remove (un)exposed areas of pos. (neg.) resist with a solvent (development) → resist mask for any step (see top), then remove the resist with another solvent. Diffraction limit of the smallest structures:

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

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_{4/2024}



A single masking level.

Example: etching a deposited film using a positive resist https://Inf-wiki.eecs.umich.edu/wiki/Lithography

ntensıţy_{4/20} Wikipedia

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see more detail in Gabor Kiss: Micro and Nanotechnology

Photolithography

Toolkit of semiconductor fabrication plant Steps: oxidation, masking, implantation, Etching, Metallization, Lift-off **in localized areas**. Example: MOSFET (pnp and npn transistor pair) 7. Etch polysilicon and oxide



Wikipedia

Photolithography

Three technologies that determine the performance of semiconductor lithography systems



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

Resolution capability of the projection lens: For forming extremely intricate electronic circuit patterns

The projection lens consists of more than 20 lenses. Some projection lenses are more than 1 meter long.

Alignment accuracy: Ensuring that the next pattern is accurately aligned to the base pattern

When electronic circuit patterns are repeatedly formed on a silicon wafer many times, they must be positioned with accuracy to the nanometer level.



Throughput: Indicates the processing efficiency of a semiconductor lithography

Productivity during IC mass production is improved when high-speed movements of the wafer stage and other processes increase throughput.

Wikipedia

Extreme Ultraviolet 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 material is transparent for this $\lambda! \rightarrow$ no lenses
- optical system is built out of mirrors; also difficult to make, they exploit constructive
- interference in multiple layers of materials (~13 nm thick each \rightarrow expensive)
- 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)



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

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Wikipedia

Electron beam lithography

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

Typical process: metallization, see figure.

Resolution:

not limited by spot size, but by proximity effect. Secondary electrons generated by e-beam in the sample and the backscattered ones further expose the resist. E.g. 20kV secondary electrons reach a circle of ~2μm.

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

Issue: sequential, throughput is small. But for research or writing a mask for use in mass production it is the perfect tool.



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Typical process: metallization, see figure.





expositive resist wafer



800.01

800.0







b)

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μm

Wikipedia

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a)

PMMA

 SiO_2

Si

250 nm

10

Thin film technologies

Widely used techniques: evaporation, sputtering, etc. High and ultra high vacuum systems are required!

Consider: number of molecules striking the surface per area and unit time (N_s) :

$$N_{\rm s} = \frac{1}{2\sqrt{3}}\rho\sqrt{\langle v^2 \rangle}, \qquad \rho = N/V = P/k_{\rm B}T$$

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

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

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 oxygen at room temperature at $p=10^{-6}$ mbar: N_s= 1 monolayer/sec.

 \rightarrow For fine structures 10⁻⁹ mbar is a min. requirement.

Different modes of crystal growth on surface.

Typically outcome is polycrystalline.

Growth of epitaxial layers requires special conditions.



Stranski-Krastanov (SK) mode Layer-plus-island growth

Depending on interface and bulk energy balances, temperature, deposition rate, and energy of arriving atoms, different 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

Physical Vapour Deposition (PVD)

Thermal evaporation

Metal is placed in a tungsten boat and heated electrically (no image).

- Sample is clamped to a stage at a distance from the target.
- Atoms condense on all surfaces (also with e-beam)
- Quartz crystal monitor is used (lab course on piezo resonance freq.).

Electron-beam deposition (top right)

Energetic e-beam is focused on the metal target, which induces local

heating \rightarrow evaporation/sublimation

Pros: it allows evaporating refractory metals (high melting point: Ti, Nb, W, Mo...); no contamination from the boat; wide range of evap. rate. Recirculating Cooling water

Cons: risk of X-ray damage, expensive



Sputter deposition (bottom left)

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.

Electron beam deposition system

Magnetron sputtering systems use magnets to trap free e's close to the target and ionize Ar there. \rightarrow Higher sputtering rate, low e flux on substrate.

Pros: easier to control composition (e.g. co-sputtering, NbN); step coverage is better (more diffusive gas; e-beam is nearly ballistic) Cons: High energy target atoms; non-directional (diffusive) and E-beam v.s. sputter deposition depends on particular applications.

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



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. <u>https://www.nano.physik.uni-muenchen.de/nanophysics/research/rep11.html</u> <u>https://www.semanticscholar.org/paper/Chemical-vapor-deposition-of-carbon-nanotubes%3A-a-on-Kumar-Ando/8cad65216fe922b14c5c947330cc791341f621fb</u>

(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 <u>https://www.sciencedirect.com/science/article/abs/pii/S0379677915300138</u> https://www.researchgate.net/figure/Schematic-of-continous-roll-to-roll-CVD-growth-and-transfer-of-large-area-graphene_fig5_249286739



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

Molecular Beam Epitaxy (MBE)

Epitaxialy growth: sequential deposition of crystalline (NOT polycrystalline) layers of specific elements/compounds. Limited in combination of substrate 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 crystal face
- Atomic beam sources are placed far
- RHEED sensor (Reflection high-energy electron diffraction) Monitor thickness by diffracted beam intensity caused by reflections from the interface and surface layers Sub-atomic layer-thickness resolution→ follow growth from layer to layer
- Frank van Der Merve crystal growth competes with VW and SK growth.
- Expensive technique with perfect outcome.
- Semiconductor heterostructures are widely used in electronics

(GaAs/AlGaAs) and optoelectronics. (GaN – see blue LEDs, InGaN) Huge impact on applications and also on quantum electronics and quantum optics.



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

Molecular Beam Epitaxy (MBE)

Band gap engineering,

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



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)



(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

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

Focused Ion Beam (FIB)

Accelerate ions that locally remove material 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} ≈ 10000 x m_e

- $\rightarrow \lambda_{ion} \ll \lambda_{e}$
- \rightarrow Lorenz force has weaker effect, mostly electrostatic focusing
- \rightarrow Less penetration than for e's.
- \rightarrow Large momentum transfer: momentum transfer kicks atoms out (Like snooker.)
- \rightarrow Localized ion milling with ~ few 10nm resolution limited by volume.

Mostly Ga⁺ ions: easy to melt, Ga is middle of periodic table \rightarrow momentum transfer is optimized for most elements. Cons: 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.



(Up) Ion-solid interaction.

Pros: Creation of secondary electrons (\rightarrow *imaging*); Removal of surface atoms, clusters; Creation of secondary ions (imaging)

Cons: ion implantation, Amorphisation, Scattering. (Down) TEM sample fabrication by FIB http://www.uni-

stuttgart.de/mawi/aktuelles_lehrangebot/documents/L SII/SS-2017/HighResMicro/lecture FIB 14 09 17.pdf



Focused Ion Beam (F



Sample preparation for TEM

FIB assisted IC editing

(a) AI **FIB-milled** Si particle trenches FIB deposited C TEM sample Joined at two points : 6 um (c) AI Si particle AND DESCRIPTION OF THE OWNER Wneedle TEM sample - C deposited for joining 6 um









FIB - Helium ion microscopy (HIM)

It uses advantages of FIB in imaging. Detect secondary electrons. Observation possible at sub-nanometer scale Advantages compare to SEM:

- + wave length limit smaller than for SEM (no adverse diffraction)
- + 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 compared to FIB
- + Special, well-focused source: helium ions generated in a region of a single atom.

Performance:

- \rightarrow Surface resolution 0.24nm (Zeiss 2009) much better than SEMs.
- → Depth of field is 5x better than for SEM (more collimated / small convergence angle)

(Up) Secondary electron generation comes from a much smaller volume for HIM as for SEM. Only surface layer. <u>https://www.researchgate.net/figure/Schematic-</u> comparing-between-helium-ion-beam-and-electron-beamand-charge-distribution fig9 267743550 (Left) Atomic sized source: voltage-biased needle at cryo temperature in He gas \rightarrow field ionization. (Right) Example of field of depth. Image size 1um, depth 2.5um. DETAILS:

https://microscopy-analysis.com/editorials/editoriallistings/principles-and-applications-helium-ion-microscopy https://www.nist.gov/sites/default/files/documents/pml/di v683/conference/Postek_2009.pdf







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Wikipedia

Nanoimprint lithography (NIL) - Stamping

Similar to original lithography invented by Gutenberg.

Example 1

- 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 rubber is resulted.
- Can be used as an office ink stamp to deposit chemicals in nanopatterns (see Panel (a) in Figure)
- With PDMS ~10nm resolution was achieved.

Advantages: No wavelength limit (High resolution), Parallel process, Cheap Disadvantages: 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. 11/14/2024 Nanotechnology and mat

(a)



3. Withdraw stamp 4. Etch

(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

4. Etch



Wikipedia

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 at room temperature
- 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 ways to bond to other atoms (with 1, 2, 3, or 4 electrons). Therefore versatile structures can be built up.

Examples: DNA origami (see Introduction)





Nanotechnology and material science Lecture I Lindsay : Intro to Nanoscience, Chapter 6

Power of organic synthesis

Example I: Nano-muscle based on rotaxane It is based on a pair of rotaxane molecules (blue) free to slide on a shaft: switch from position green to position red as the oxidation state of the central molecule (TPR, gray middle) is changed from 8+ to 12+.



Power of organic synthesis

Example II: Light-to-charge converter

- Porphirin (P) absorbs light by exciting electron. An artificial photosynthetic unit
 A coupled fullerene grabs the excited electron
- A coupled fullerene grabs the excited elect before e in P decays to ground state
- A coupled carotene (C) injects an electron back to P
- P is now in its ground state, but:
- Carotene is now positively charged and fullerene is negatively charged.
- Charge separation can be used as chemical energy.



Light-to-charge converter

A long-lived charge-transfer state (from 60ns to a microsecond)!

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 hydrophobi component.

E.g. detergents: hydrophobic component binds oil/grease 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 methylene, -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 (Right) Amphiphiles

Liposome are bilayer spheres that enclose water They self-assemble. With molecular dynamics the final form can be tested and simulated.

$$\frac{v}{a_0 \ell_c} < \frac{1}{3}$$
 → spherical micelle
 $\frac{1}{3} < \frac{v}{a_0 \ell_c} < \frac{1}{2}$ → non-spherical 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, I_c is the length of hydrocarbon chain.





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solutions

research-

eaates/

Mitochondrion

A self-assembled nanochemistry machine

Inside eukaryotic cells (like ours), outside of the nucleus. (They carry their own DNA. "Presumably, they became incorporated into anaerobic organisms as a symbiotic partner that helped these cells survive the increase in atmospheric oxygen that occurred a few hundred million years ago.")

Machines that allow us to use atmospheric oxygen to complete the oxidation of sugars to produce biological energy.

Largely assembled from phospholipids: two sets of lipid bilayer membranes.

Outer one is a filter, only allowing small molecules into the intermembrane space.

Protons are pumped from the matrix into the intermediate space. The gradient 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

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) A way to test molecular electron transport https://www.researchgate.net/figure/Artistic-view-of-analkanethiol-self-assembled-monolayer-SAM-on-a-goldcoated_fig1_230938526





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J. Phys. D: Appl. Phys. 39, 387

Vapor-liquid-solid (VLS) growth:

- Catalyst metal nanoparticle
- Presence of vapor source of semiconductor
- eutectic mixture (metal + semicond. here): a homogeneous mixture with a melting point lower than those of the constituents.
- eutectic temperature: the lowest possible melting point over all of the mixing ratios
- Heated above the eutectic T
- Supersaturates -> nucleation of semiconductor
- Growth the NW with droplet on the top

³ Reactants: same precursors as in other processes,

CVD (Chemical vapour depos.), MBE, MOVPE, CBE

Competing interfaces:

- Liquid-solid (at metal particle): elongation of the NW, metal particle determinates the diameter
- eg.: possible 3nm diameter Si

well-defined crystallographic orientation

• gas-solid (NW surface): thickening in the radial direction

Which dominates? p, flow, T, reactant Nanotechnology and material science Lecture I



Crystalline heteroepitaxy can be achieved Depending on preferred reaction interface (axial or radial growth):

• Radial heterostructures, core/shell (b→d)

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



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- Radial heterostructures, core/shell (b→d)
- eg.: Si/Ge core/shell structure -> hole gas GaN ->light emitting diodes, SiO/Si
- 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



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Nano Lett 2, 87 (2001)



- 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

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Nano Lett 2, 87 (2001)

InAs nanowires with epitaxial Al shell in certain sections

(define shadow mask - blue - on chip before growing NW; then evaporate Al at an angle). Also InP barriers are possible.







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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. <u>https://www.nano.physik.uni-muenchen.de/nanophysics/research/rep11.html</u> <u>https://www.semanticscholar.org/paper/Chemical-vapor-deposition-of-carbon-nanotubes%3A-a-on-Kumar-Ando/8cad65216fe922b14c5c947330cc791341f621fb</u>

(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 <u>https://www.sciencedirect.com/science/article/abs/pii/S0379677915300138</u> https://www.researchgate.net/figure/Schematic-of-continous-roll-to-roll-CVD-growth-and-transfer-of-large-area-graphene_fig5_249286739

