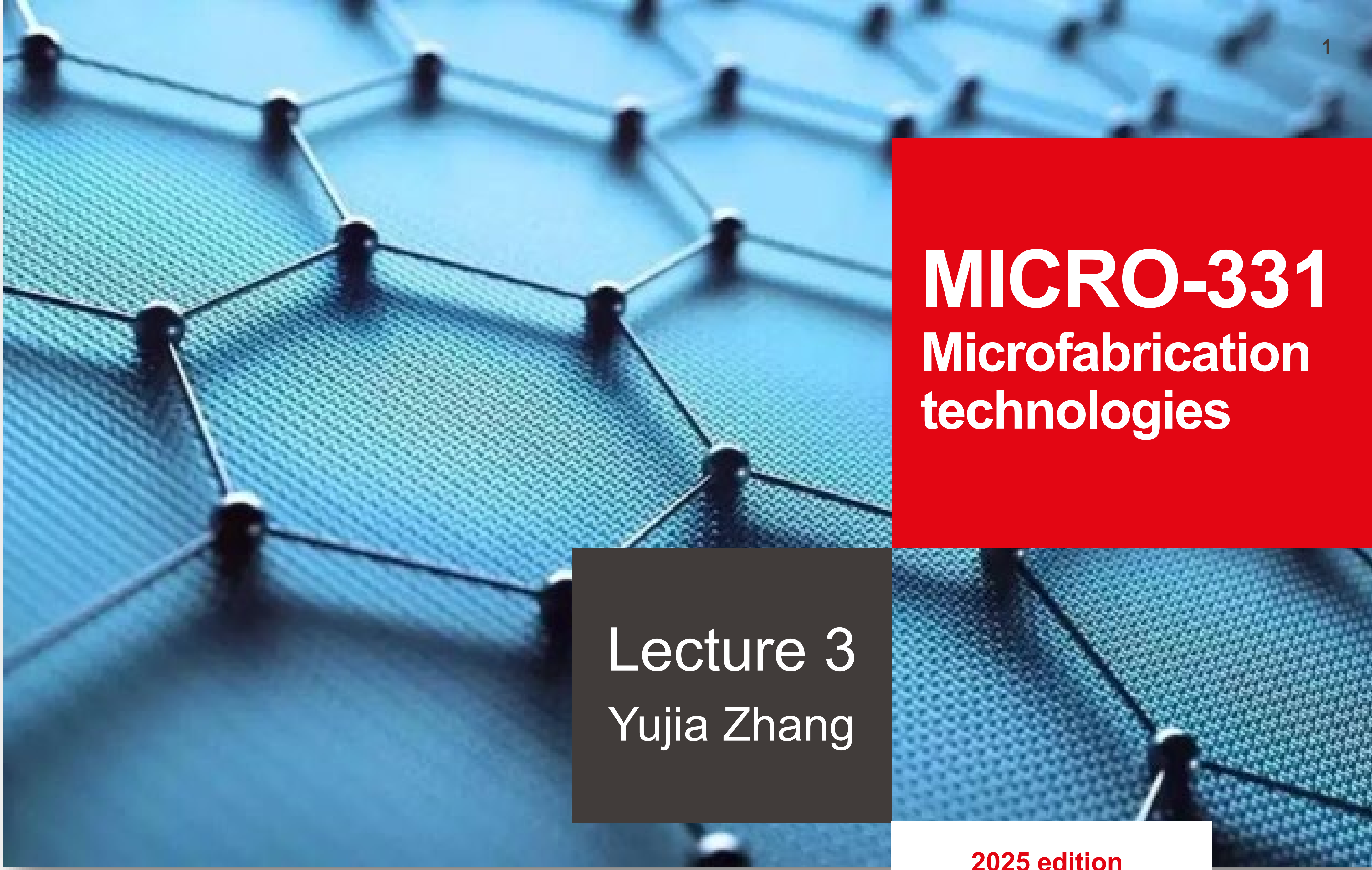
EPFL



MICRO-331
Microfabrication technologies

Lecture 3

Yujia Zhang

2025 edition

Today's program

- 1st hour:
  - Feedback of the SLT_2 (8')
  - Responses to previous questions (5')
  - Attendance at the Thursday MOOC class (2')
  - Recapitulation: CVD (15')
  - From ALD to Epitaxy (10')
  - A question on thermal oxidation (5')

- 2nd hour:
  - Electroplating: a case study of vinyl records (5')
  - Metrology (5')
  - Physical vapour deposition (PVD) (30')
  - Q&A (5')

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Speaker

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How was your experience when preparing for the second SLT by comparison with the first time?

ENT / NAME PRESENTATION

Speaker

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How was your experience during the second SLT by comparison with the first time?

ENT / NAME PRESENTATION

Speaker

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How would you like the SLT questions to be?

ENT / NAME PRESENTATION

Speaker

Answer for a previous question with the highest vote

- *Q: Be more clear on what level of details should we know for the exam?*
- Examples of the question:

Single and multiple choice questions, true/false and open questions:

(General knowledge) What is the primary function of a cleanroom in microfabrication?

(Specific related to MOOC) What role does potassium hydroxide (KOH) play in the fabrication process of the bimorph cantilever seen in the course many times?

(Basic calculation) Based on the given Arrhenius plot, what is the difference between the A and B growth rates?

(Drawings and explanations) Can you draw a schematic of the PECVD chamber and explain its working mechanism?

(Questions from SLTs with different expressions)

(Questions from Lectures and especially Guest Lecturers)

We want you to focus on critical thinking and rationale, not remembering trivial details/numbers

Attendance at the Thursday MOOC class

- It’s a good opportunity to answer questions you may have during MOOC studies.
- Prepare answers for the next SLT.
- Would you prefer switching the MOOC class and SLT session?
  - i.e., having MOOC class on Wednesday and SLT on Thursday?

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Audience Q&A

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Recapitulation: CVD

Speaker

Key information of CVD

- **Chemical reaction** is involved
- Use of **gaseous** phase results in conformal **deposition** on substrate with arbitrary texture
- Many techniques to choose from, so we should consider:
  - Quality of resulting films (electrical properties, etch selectivity, defects, residual stress, etc)
  - Conformality
  - **Deposition rate**
  - Cost
- CVD
  - Reactants are transported to the substrate, a chemical reaction occurs, and the products deposit on the substrate to form the desired film
- Physical techniques (later today)
  - Material is removed from a source, carried to the substrate, and dropped there

Different CVD approaches

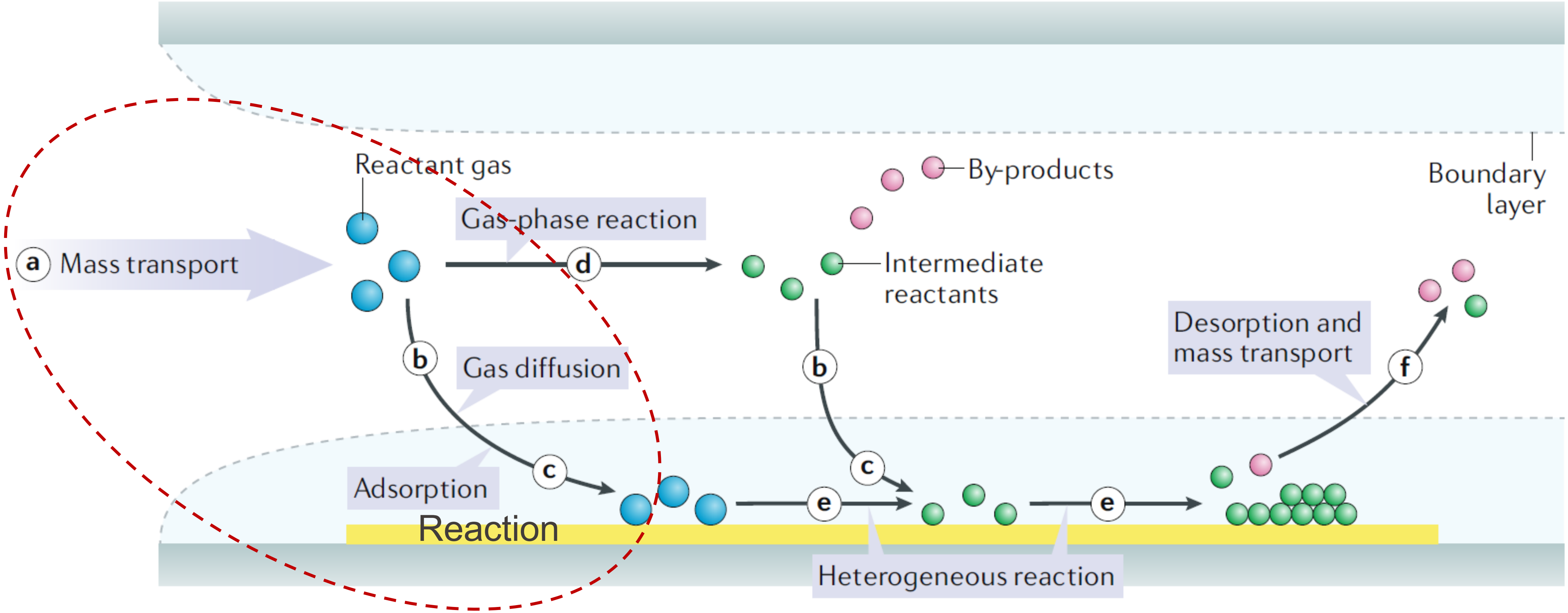
Taxonomy of deposition techniques

- ***Thermal Oxidation***
- Classification of CVD by pressure
  - Atmospheric pressure CVD (APCVD)
  - Sub-atmospheric pressure CVD (SACVD)
  - **Low-pressure CVD (LPCVD)**
  - Ultrahigh vacuum CVD (UHV/CVD)
- Classification of CVD by reactor type
- Other CVD
  - **Plasma-enhanced CVD (PECVD)**
  - Metal-organic CVD (MOCVD)
  - **Atomic layer CVD (ALCVD or ALD)**
- Epitaxy
- Electrodeposition (Electroplating)

SLT 2.1

See MOOC:
Overview of CVD techniques
Specific CVD processes

The scope of this course and beyond

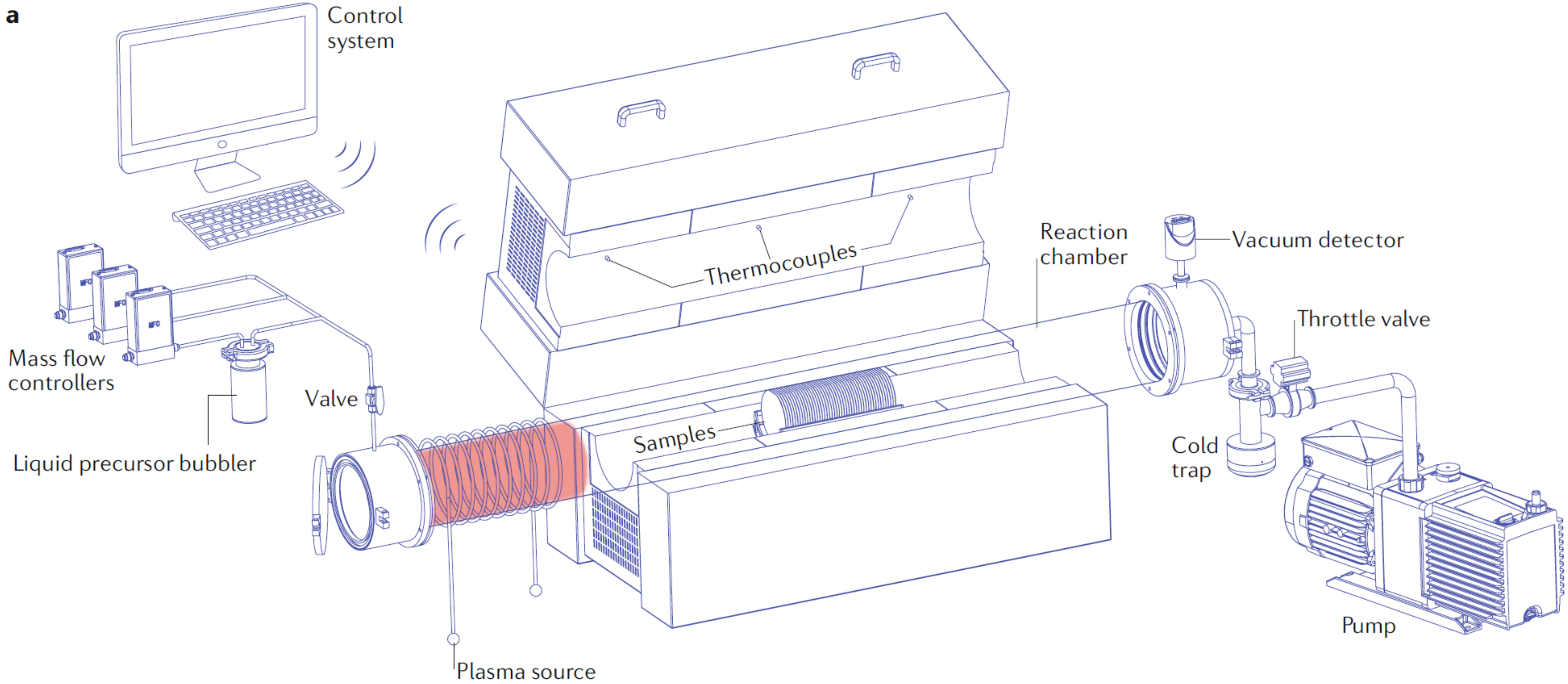


The focus of MOOC CVD

See MOOC:
Film growth (PVD)

- A very good reading material here:
  Chemical vapour deposition. *Nat Rev Methods Primers* **1**, 5 (2021).

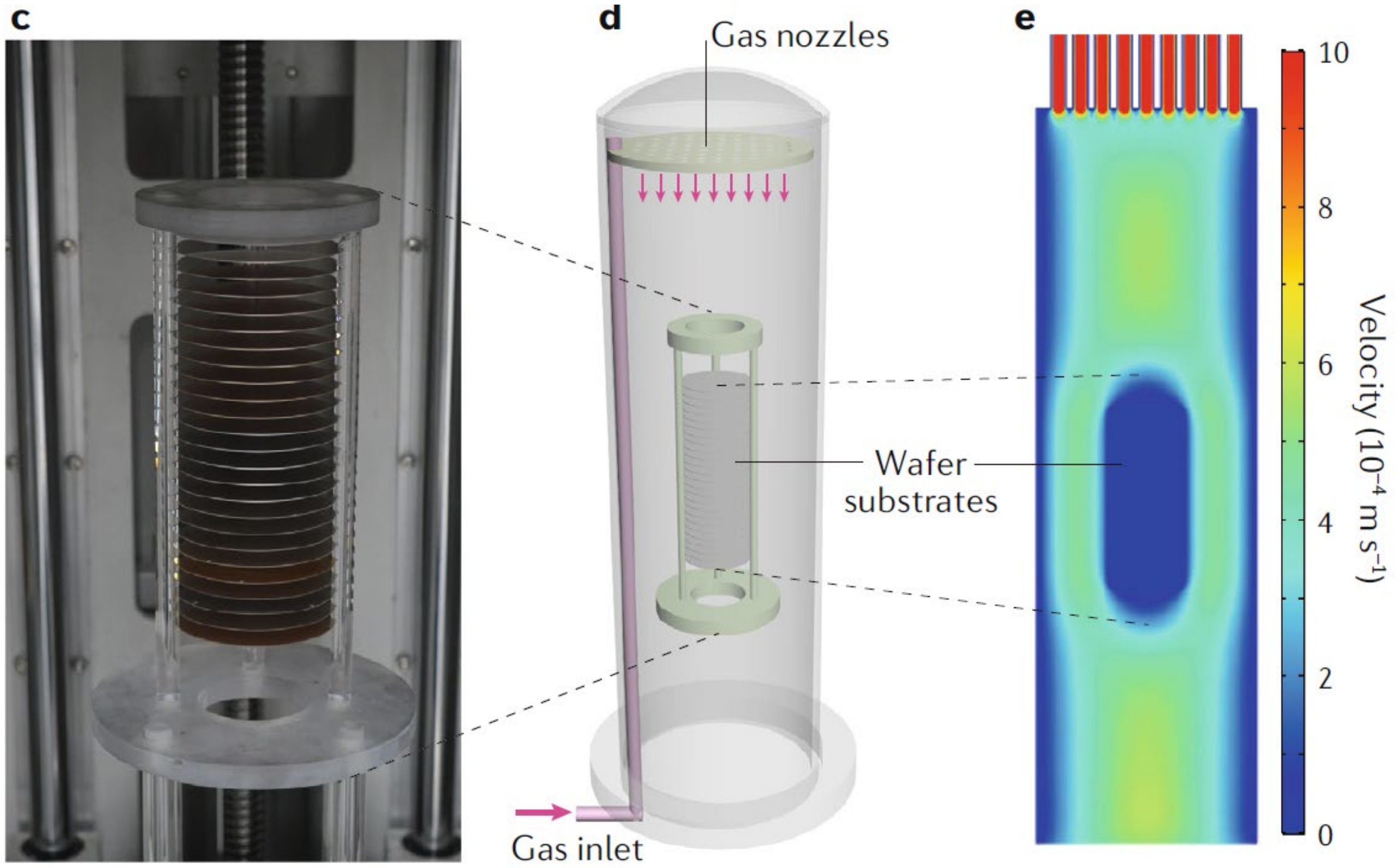
Typical CVD equipment



- Schematic diagram of a typical horizontal CVD system, which includes a gas delivery system, the quartz reaction chamber, a vacuum system, the energy system, and an auto-control system.

Chemical vapour deposition. *Nat Rev Methods Primers* **1**, 5 (2021).

The importance of uniform, laminar gas flow (vs. turbulent)



- The multiple gas inlet nozzles are designed (part **d**) to improve the uniformity of gas flow, which is simulated based on the finite element method (COMSOL).

See MOOC:
Theoretical aspects of CVD

Chemical vapour deposition. *Nat Rev Methods Primers* **1**, 5 (2021).

The Arrhenius plot

- Wait, who is Arrhenius?

Svante August Arrhenius (19 February 1859 – 2 October 1927) was a Swedish scientist. Originally a physicist, but often referred to as a chemist, Arrhenius was one of the founders of the science of physical chemistry. In 1903, he received the **Nobel Prize** in Chemistry, becoming the first Swedish Nobel laureate. In 1905, he became the director of the Nobel Institute, where he remained until his death.

The Arrhenius equation can be given in the form:

$$k = A \exp\left(\frac{-E_a}{RT}\right) = A \exp\left(\frac{-E'_a}{k_B T}\right)$$

where:

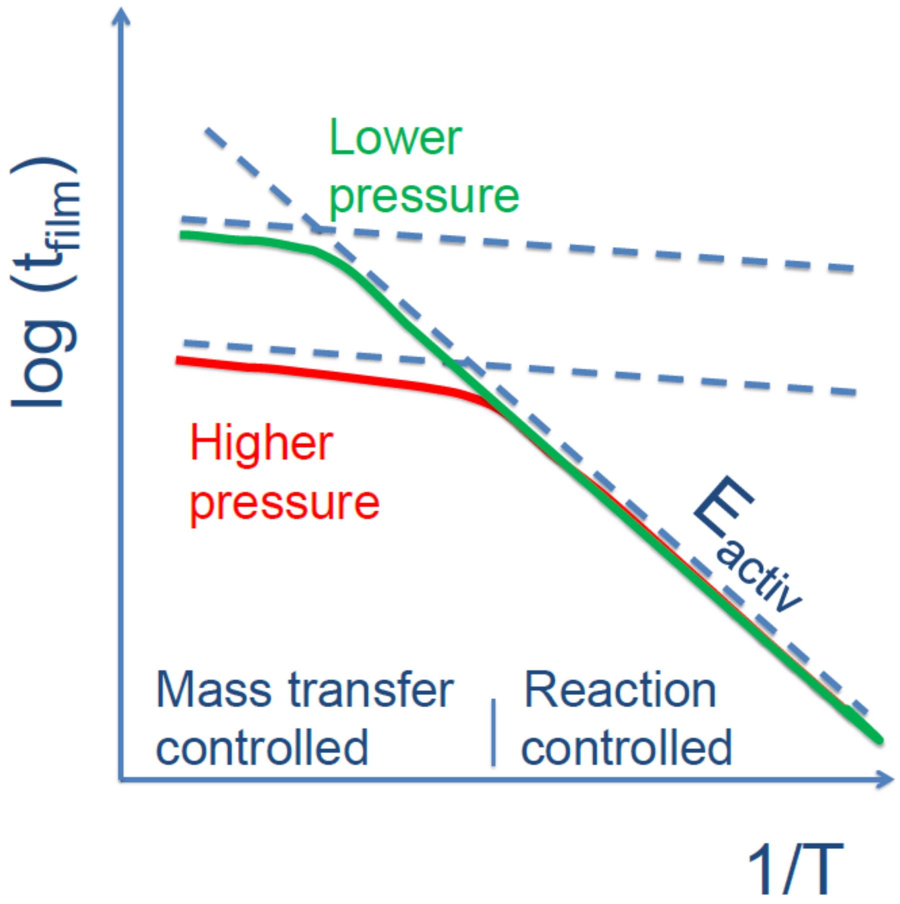
- k = rate constant
- A = pre-exponential factor
- E_a = (molar) activation energy
- R = gas constant, ($R = k_B N_A$, where N_A is the Avogadro constant).
- E'_a = activation energy (for a single reaction event)
- k_B = Boltzmann constant
- T = absolute temperature

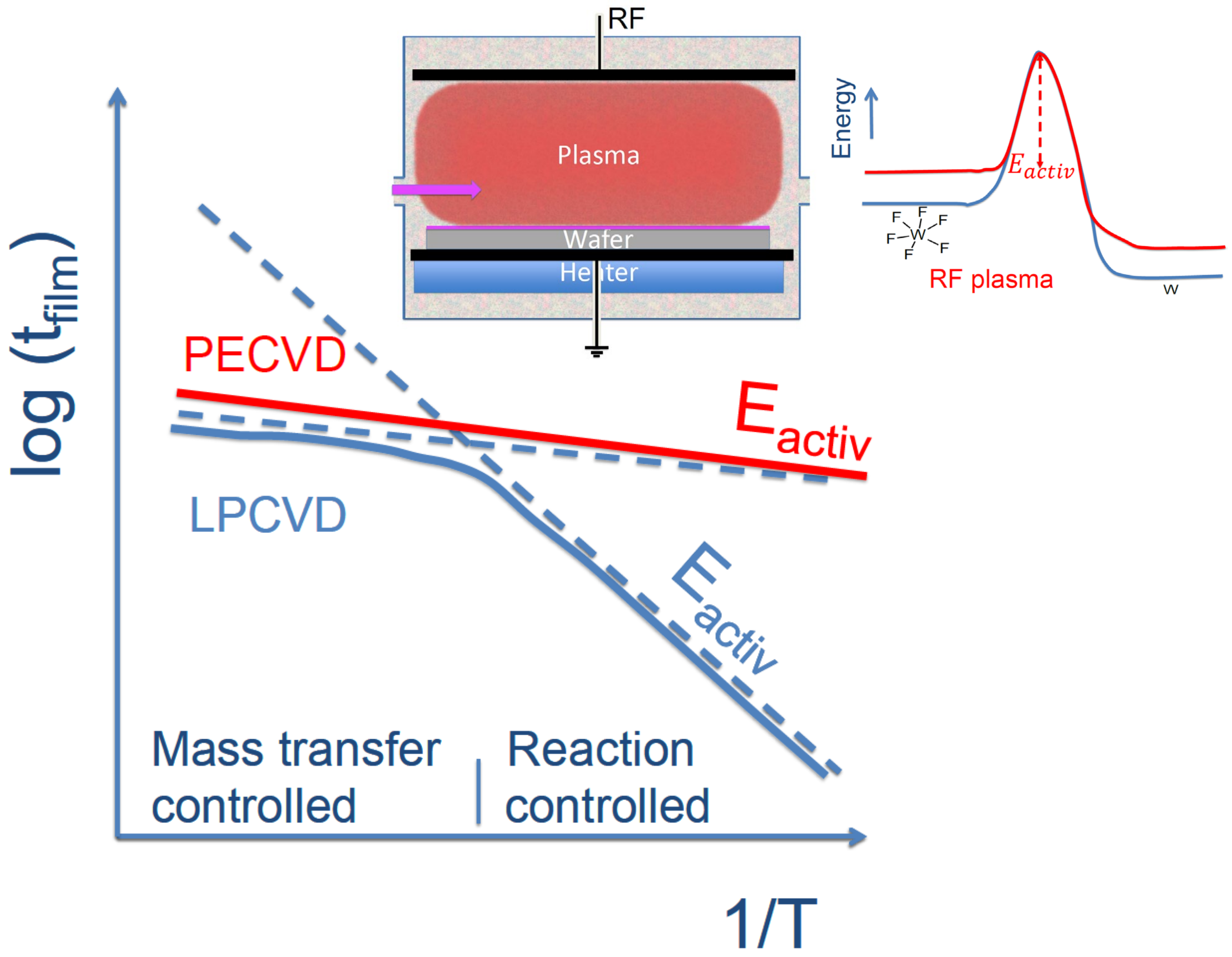
Information from Wikipedia

The Arrhenius plot

- Chemical reactions: the influence of T, P, diffusion…
- From LPCVD to PECVD

SLT 2.2



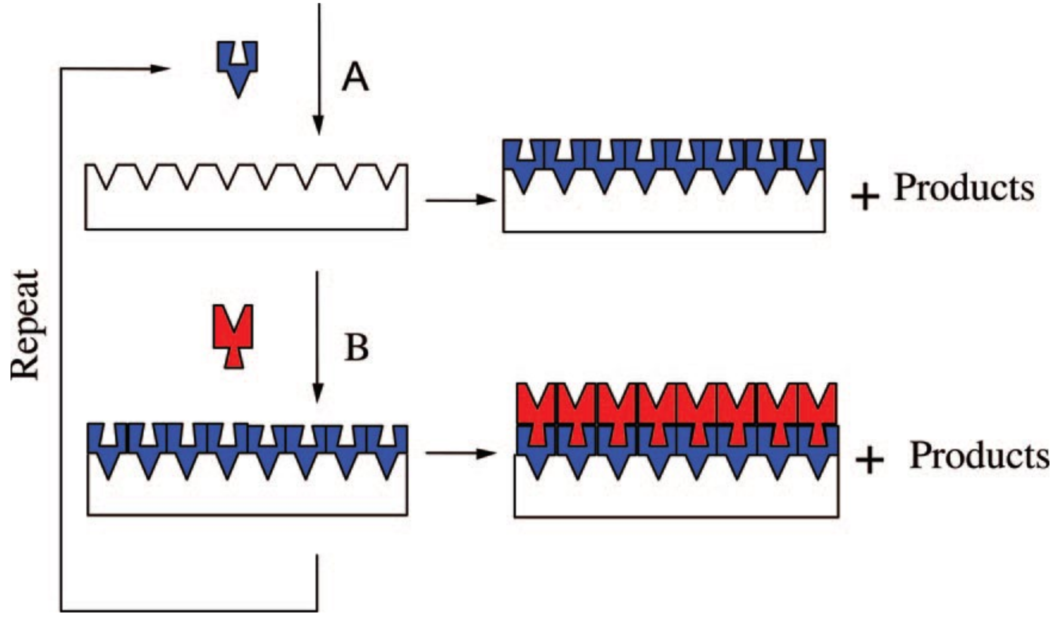


Examples of CVD processes

- LPCVD of polycrystalline and amorphous Si
- LPCVD of **Si_3N_4** and **$Si_xN_yH_z$**
- LPCVD low-temperature **oxide**
- PECVD of **diamond** and other 2D materials, e.g., graphene
- What are the **common materials** to deposit by CVD, and why? *SLT 2.3–2.5*

See MOOC:
Specific CVD processes for silicon-based materials and diamond

Atomic layer CVD (ALD)



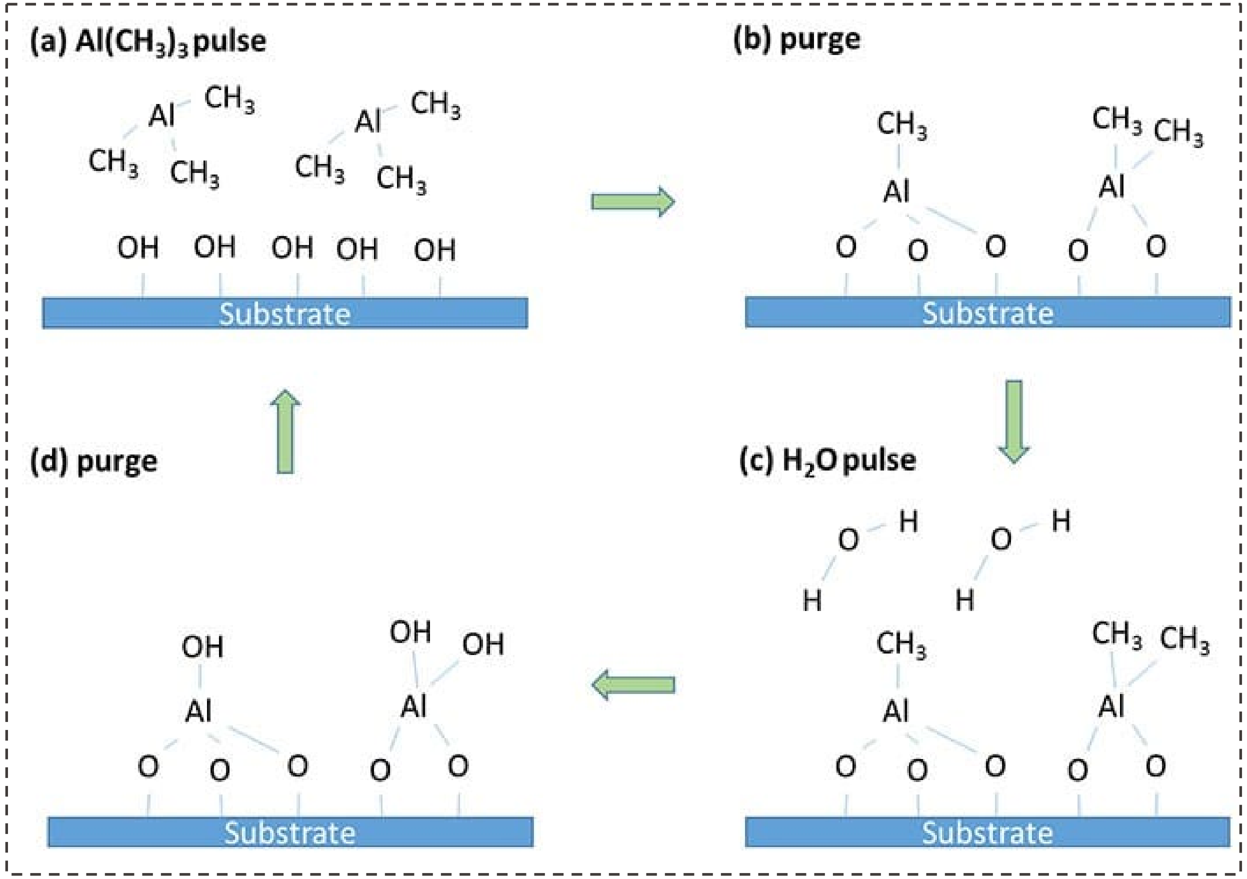
- Other materials, such as
  SiO_2, ZnO, TiO_2, polymers…
- It is still chemical reactions,

so it could be PE-ALD, etc.

SLT 2.3

ALD deposition of specific oxides and metals

- How to achieve this?
- ***Clever and elegant chemical reactions***
- Al_2O_3 ALD as a Model ALD System



- A very good reading material here:

Atomic Layer Deposition: An Overview. *Chemical Reviews* **2010** *110* (1), 111-131.

A deeper thinking in ALD

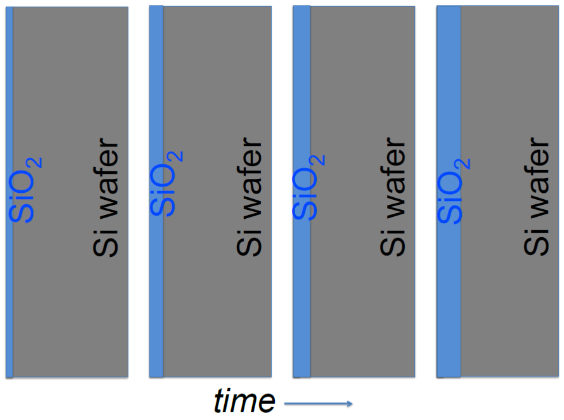
- Could you plot the ideal mass change (e.g., Si + Al_2O_3) after repetitive ALD cycles?
- What if it's not ideal, e.g., the substrate is not even?
- What if the substrate is not a 'substrate'? i.e., Can we deposit on non-conventional wafers? Or even not wafers?
- What if…?
- The more you think, the boundary between *MICRO 331* and other disciplines becomes more blurry, and you are getting closer to cutting-edge technologies.

Reading materials here:
Chemical vapour deposition. *Nat Rev Methods Primers* **1**, 5 (2021).
Atomic Layer Deposition: An Overview. *Chemical Reviews* **2010** *110* (1), 111-131.
Remote epitaxy. *Nat Rev Methods Primers* **2**, 40 (2022).
Metal–organic chemical vapour deposition for 2D chalcogenides. *Nat Rev Methods Primers* **5**, 57 (2025).

A question on thermal oxidation

- Someone asked me a really good question after the 2nd lecture…

Thermal oxidation mechanism

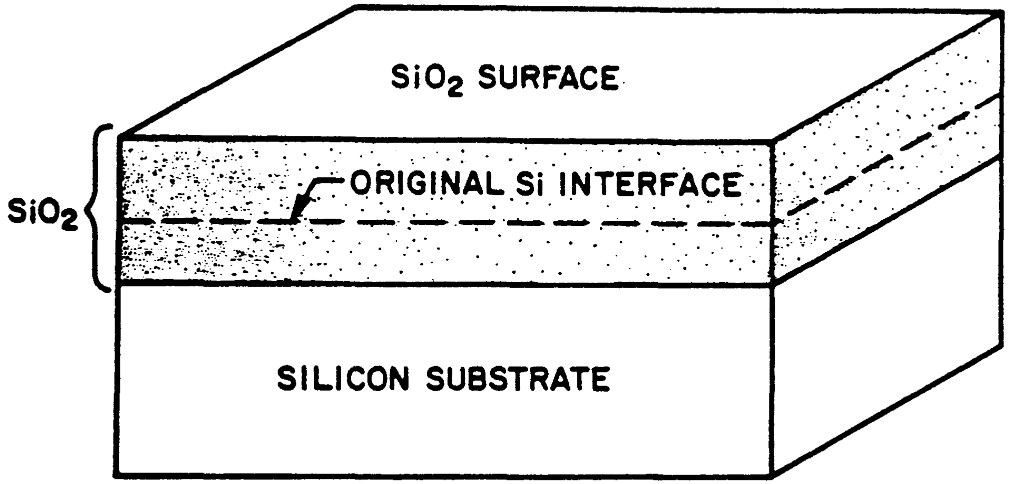


- SiO_2 is formed by diffusion of oxygen into the Si wafer
- Oxidation is at the SiO_2/Si interface
- Initially oxide thickness $t_{ox} \sim time$, later $t_{ox} \sim \sqrt{time}$ due to increased diffusion length
- In general $t_{ox}{}^2 + A\, t_{ox} = B(time + \tau)$ with A and B two constants that depend on the substrate and oxidation conditions and τ a time constant to take care of the native oxide

Micro and Nanofabrication (MEMS)

Thermal oxidation processes of silicon

But is this schematic clear?



- For every unit of silicon thickness that is consumed, approximately 2.17 units of SiO_2 thickness are formed.
  - Si → 0.46 tox
  - Why 2.17 (0.46)?
- Deal–Grove model

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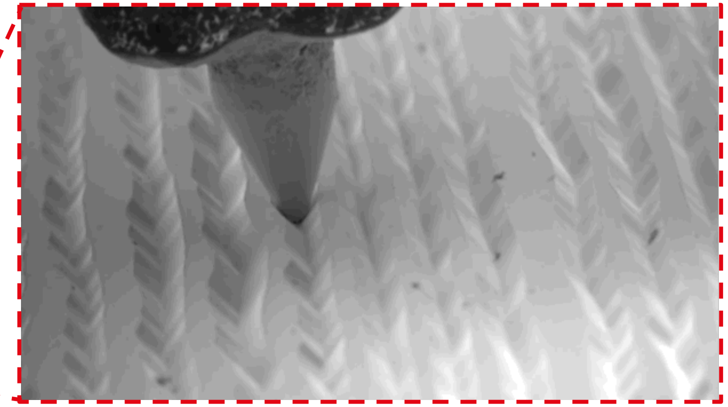
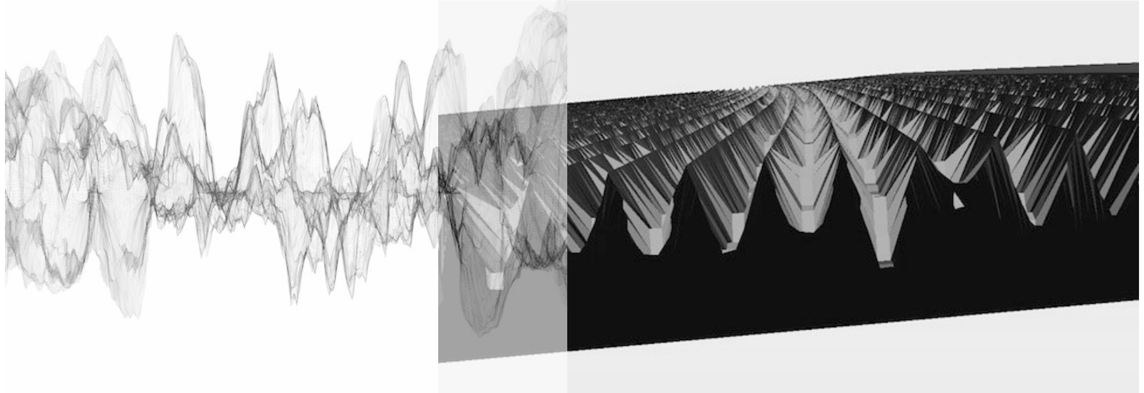
Electroplating: a case study of vinyl records

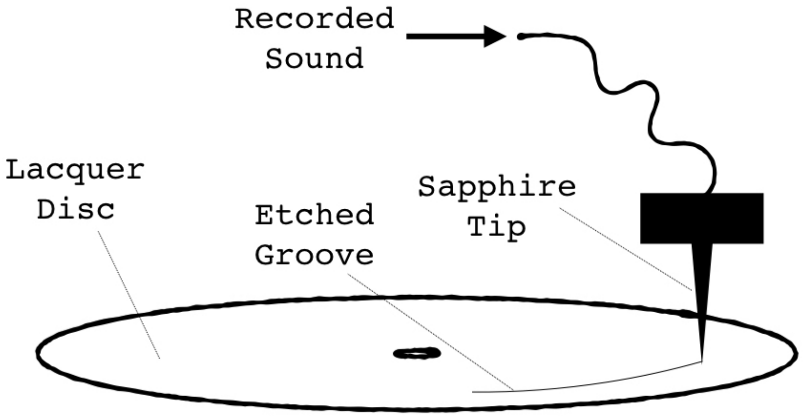




Isn't this a microtechnology?

- But how is it made?

(Thermal) Scanning Probe Lithography by Prof. Brugger



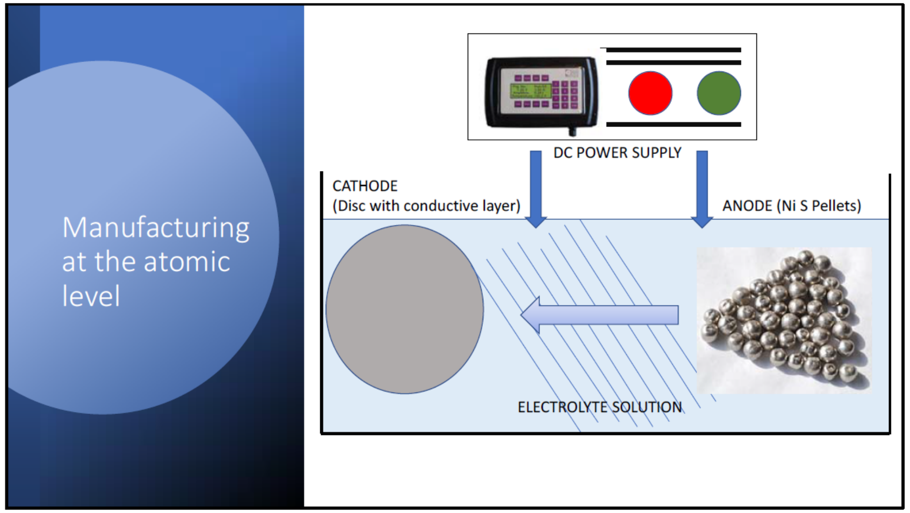
Steps to Vinyl Production

- **Lacquer Master**
- **Metal Stamper**
- **Pressing**

Steps to Plating

- **Silvering** - creates a conductive seed layer on top of the lacquer
- **Pre-Plating** - sets down a first layer of nickel at lower temperature
- **Fast Plating** – final nickel deposition to form first stamper or "father"
- **Family plating** – additional stampers from original for production scale & backup
- Finishing

Electroplating



- https://www.londonsoundacademy.com/blog/how-does-vinyl-work
https://www.youtube.com/watch?v=Yd2SW-Fys6I

Metrology

A very good question during SLT 2:

Q: How do we verify the results of ALD?

We may explain more of metrology in later lectures.

Table 2 | **Characterization tools and their settings for assessing graphene quality and structure**

Assessment tool	Spatial resolution	Property	Advantages	Disadvantages	Considerations
Optical microscopy	Micrometres or less	Individual domain shapes; surface coverage; number of layers; grain boundaries; defects (wrinkles and folds)	Simple to operate; large-area characterization; non-destructive to the sample	Low resolution; usually needs a suitable substrate	Clearer images when choosing a suitable wavelength of light or adding an optical filter
Scanning electron microscopy	Approximately nanometres	Individual domain shapes; surface coverage; number of layers; grain boundaries; defects (wrinkles and folds)	Simple to operate; large-area characterization; high resolution; good environmental adaptability	Damages the graphene atomic lattice	Based on electron scattering; electron beam energy ranges from a few 100 eV to a few keV
Atomic force microscopy	Nanometres or less	Individual domain shapes; surface coverage; roughness; grain boundaries; number of layers	High resolution; good environmental adaptability	Scanned area is small; scanning speed is slow; unsuitable for samples with significant surface topography	Sample surface must be clean; pollutants contaminate the tip and can result in virtual and/or false images
Scanning tunnelling microscopy	Ångstroms or less	Atomic structures (point defects, grain boundaries); crystal orientation	Atomic resolution; non-destructive	Scanned area is small; substrate must be conductive and ultra-smooth; complex and expensive	Based on quantum tunnelling effect; two possible operation modes (constant current or constant height)
Transmission electron microscopy	Ångstroms or less	Atomic structures (point defects, grain boundaries); crystal orientation; purity	Atomic resolution; can obtain cross-section geometry	As-grown graphene must be transferred to a suitable substrate	High-energy electron beam (several 10 keV to few 100 keV) can induce defects
Raman spectroscopy	~100 nm (diffraction limit)	Number of layers; defects; strain; doping	Simple to operate; high sensitivity; spatially resolved distribution easily obtained by mapping the sample	Qualitative only	SiO_2 is the most frequently used substrate
X-ray photoelectron spectroscopy	100 µm	Purity	Surface-sensitive technique to analyse elemental composition and chemical state	Low spatial resolution; no accurate quantification	Detecting depth ranges from 1 to 10 nm
Angular resolution photoemission spectroscopy	Approximately millielectronvolts (relies on the resolution of the analyser, the sample and the ultraviolet source)	Electronic properties (band structure); doping	Band structure can be directly observed; as-grown graphene can be directly characterized	Band structure above the Fermi surface cannot be obtained; complex and expensive	Based on the photoelectric effect

Chemical vapour deposition. *Nat Rev Methods Primers* **1**, 5 (2021).

Physical Vapour Deposition (PVD)

Speaker

From CVD to PVD

- No chemical reaction involved
- Purely physical film formation
- Vapour condenses to a solid film
- Fundamental difference

Absorption/desorption

☞ Physisorption:

no chemical interaction involved (van der Waals).

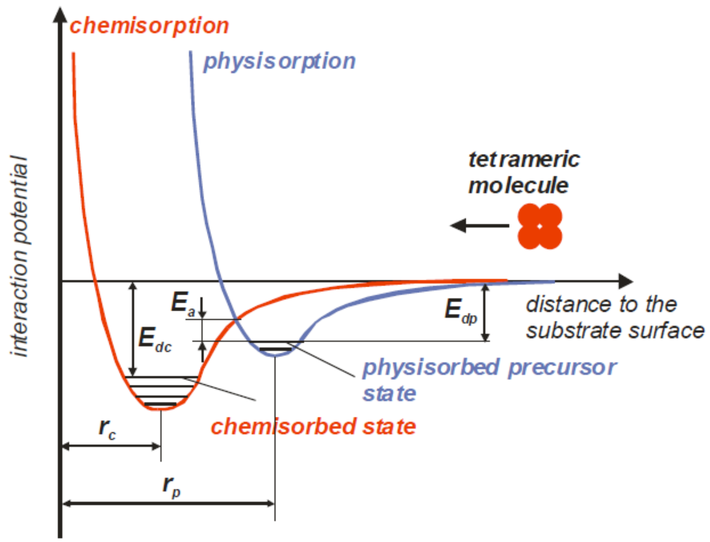
E_a ~ 100 meV/atom

☞ Chemisorption:

strong chemical bond formed.

E_a ~ a few eV/atom

(> substrate sublimation energy)



Lecture 2
See MOOC:
Film growth

Reference: Lecture note from Semiconductor Physics

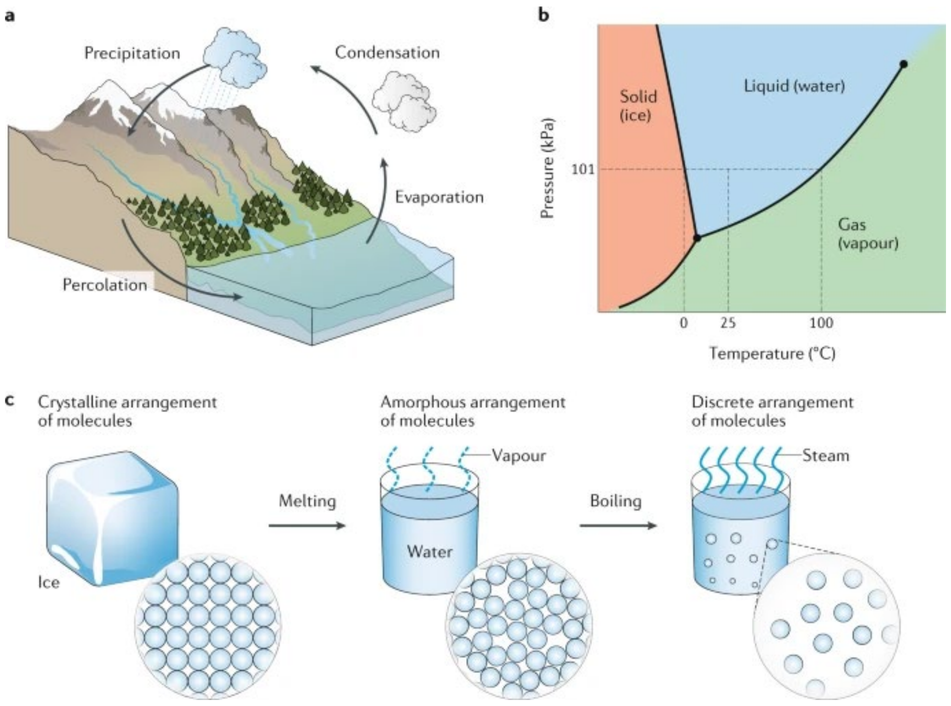
Basic principles of PVD

- Remove material from a solid source
- Transport material to substrate
- Deposit material on substrate
- Differences among PVD techniques
  - How material is removed from the source
  - Directionality when it arrives at the substrate
  - Cleanliness of deposition
  - ➢ Evaporation (Thermal, E-beam)
  - ➢ Sputtering (DC, RF, magnetron)
  - ➢ Pulsed laser deposition (PLD)
  - ➢ …

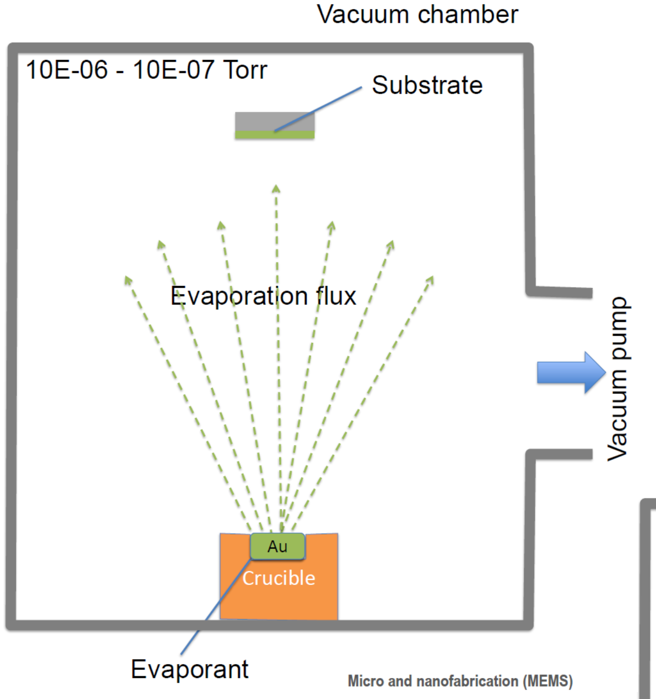
See MOOC:
Thermal evaporation, Sputtering, and Other PVD methods

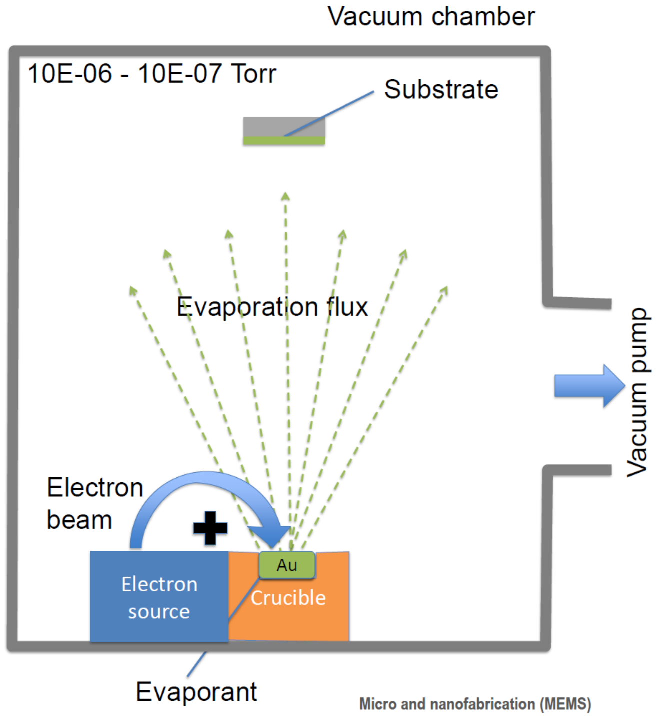
How to make vapour from solid matters?

- One way: by evaporation and thus by heat
- How to make heat?
  - By a resistive heater
    - + simple / - contamination
  - By electron beam
    - + control / - more complex



Materials for solar-powered water evaporation. *Nat Rev Mater* **5**, 388–401 (2020).





Why do we need a vacuum condition in PVD?

PVD: vapor flux towards substrate (1)

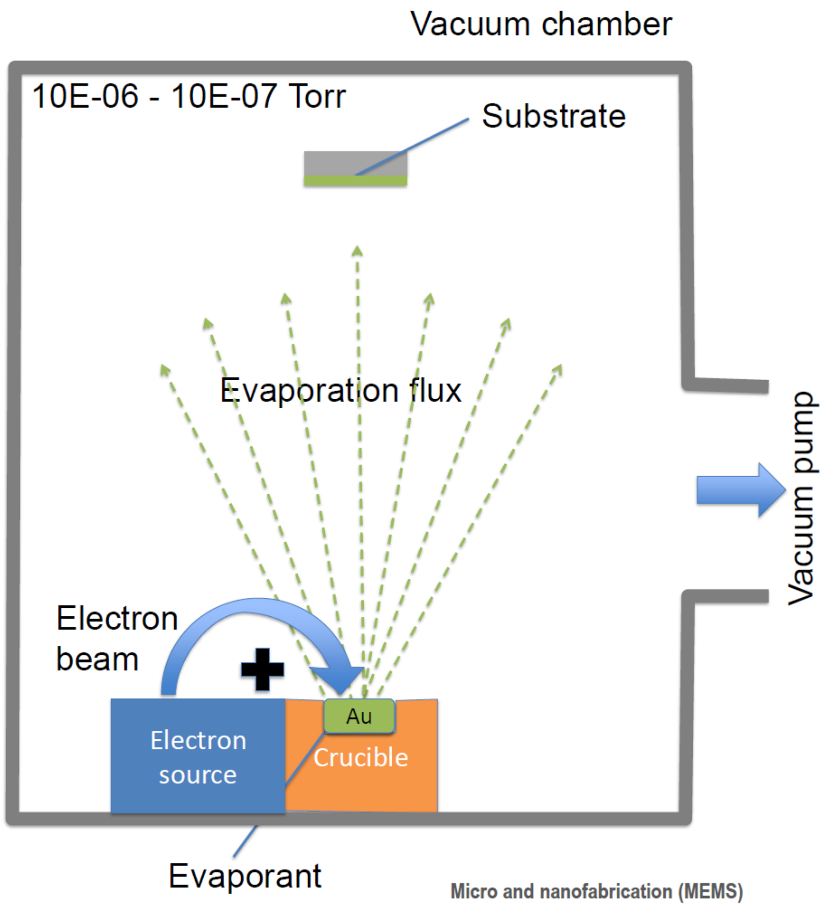
- Mean free path λ (in kinetic theory)

$$\lambda = \sqrt{\frac{\pi \cdot R \cdot T}{2M}} \cdot \frac{\eta}{P}$$

- Atoms follow straight lines until collision

λ = mean free path in [m]
R = gas constant in [J/(mol·K)]
T = temperature in [K]
M = molar mass in [kg/mol]
η = gas viscosity in [Pl]
P = reactor pressure in [Pa]

	Torr	MFP
atm	760	65 nm
LV	~1	50 µm
MV	$\sim 10^{-3}$	5 cm
HV	$\sim 10^{-7}$	500 m
UHV	$\sim 10^{-9}$	50 km



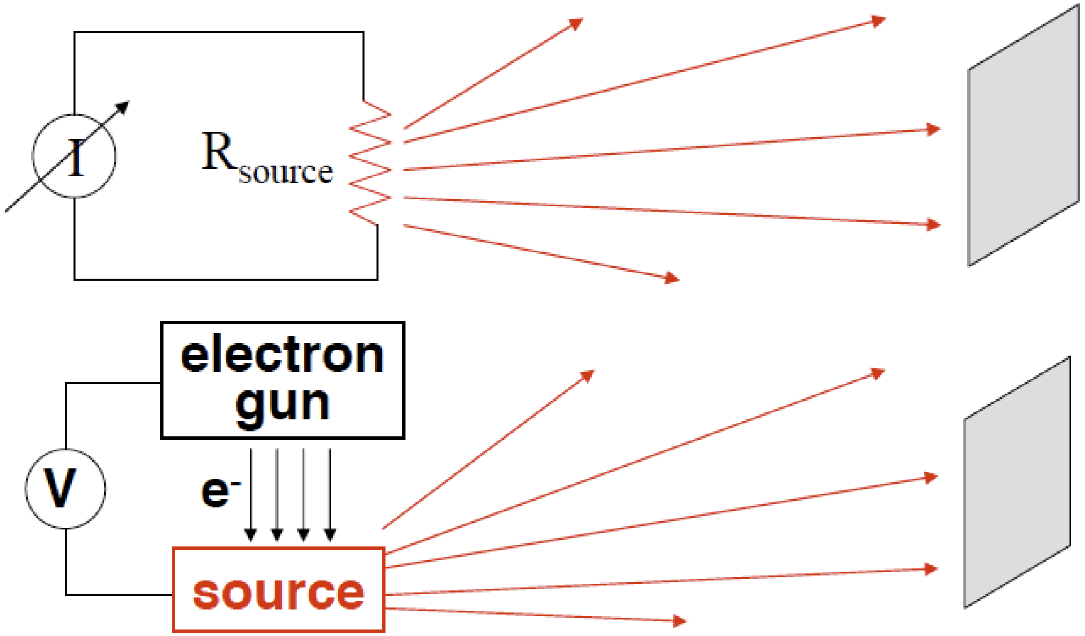
Micro and nanofabrication (MEMS)

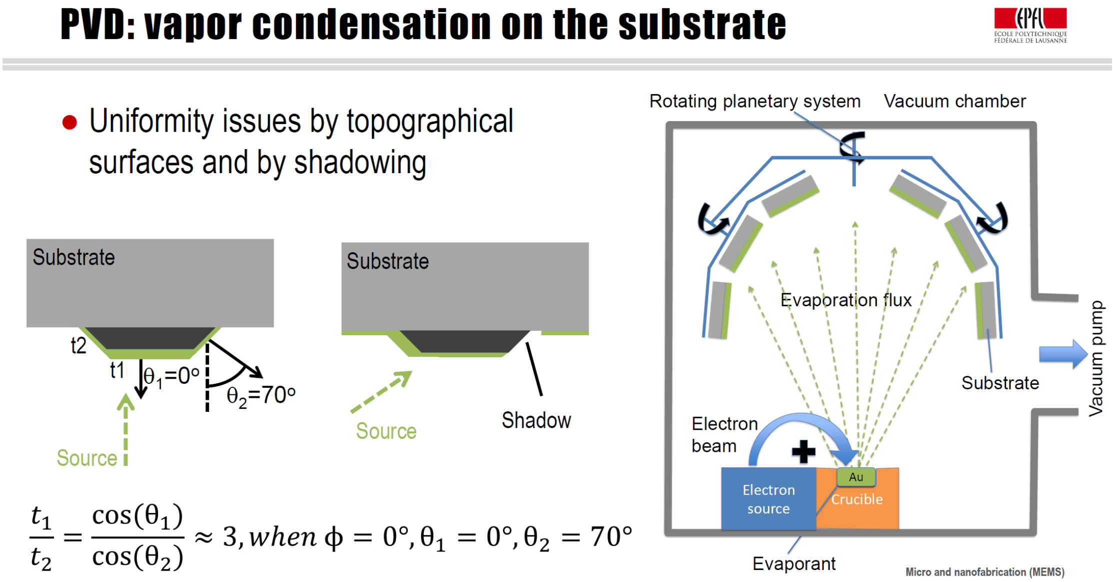
Galton board

- Video from internet

Thermal and e-beam evaporation

- Source is resistively heated
- or Electron beam heats the source in a **high vacuum**
  - Typical source: metal
- Hot source atoms are emitted in all directions and stick where they land
- Substrate receives a **directional flux** of source material (uniformity issue)
- Good for lift-off processes, otherwise **poor conformality**
  - Possible contamination from generalised heating
  - Heating is less generalised ➔ Less contamination

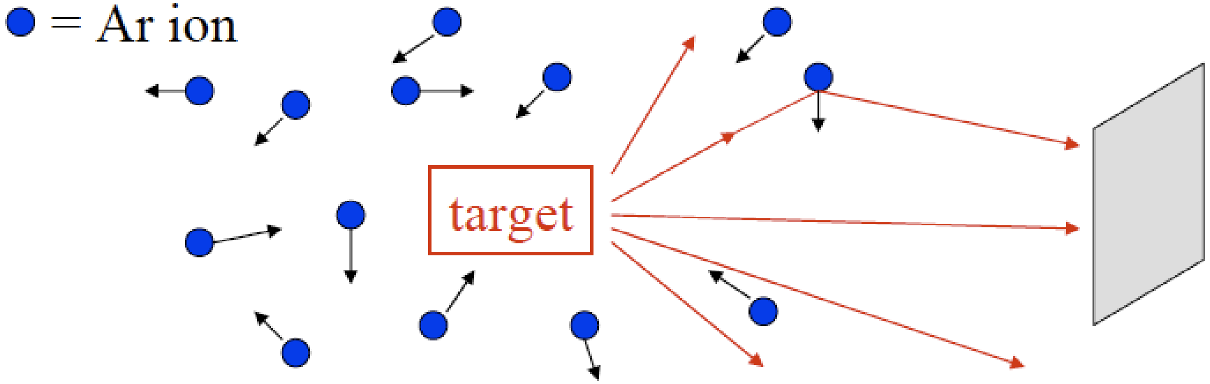




How to make vapour from solid matters?

- The second way:

- But in a more gentle way…
- **Sputtering**

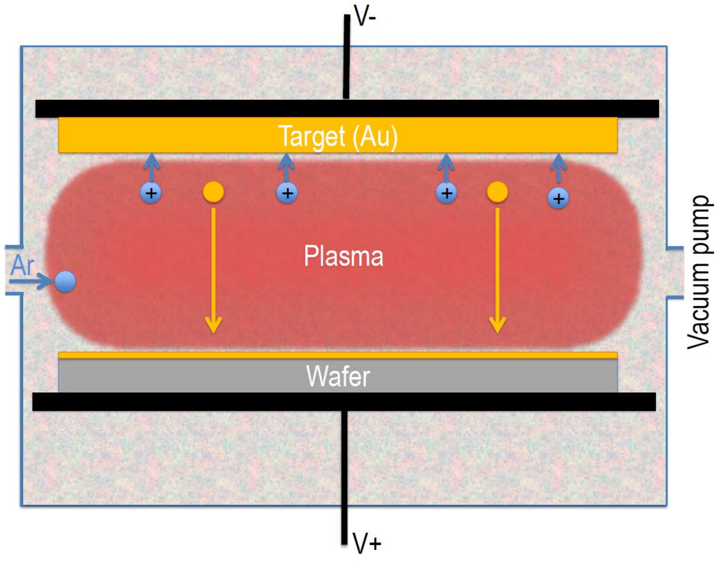


Sputtering

- Unreactive ions (i.e., **Ar**) knock material off a target by momentum transfer
  - Targets: metals, dielectrics, piezoelectrics, etc.
- Different methods of obtaining energetic ions
  - DC, RF, Magnetron …
- Low pressure, but not high vacuum
- Less directional and faster than evaporation

Sputtering principle

- Working principle
  1. Target made of material to deposit
  2. Plasma ions collide on target
  3. Atoms from target are ejected
  4. Atoms deposit onto the wafer



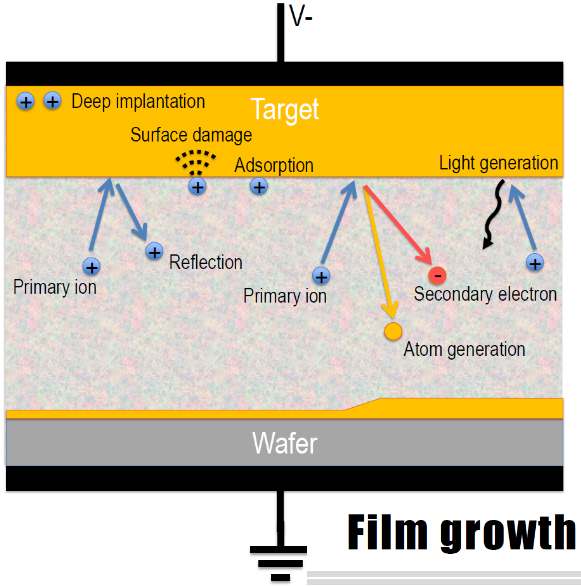
- Deposition of compounds
- Deposition of refractory materials
- Good adhesion
- Good step coverage
- Deposition of relatively thick layers

Micro and Nanofabrication (MEMS)

A closer look of sputtering

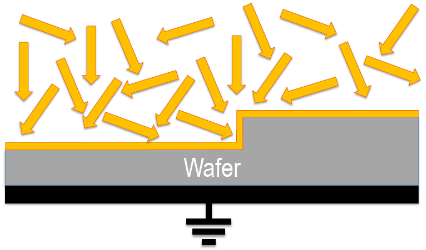
Ions-target interactions

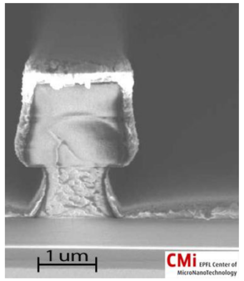
- Ions-target interactions
  - Reflection, adsorption, surface damage, gas desorption
  - Secondary electrons, ions and atoms generation
  - Deep implantation
  - Photons and x-rays generation
- 95% of ions energy heats up the target
  - Target cooling is required
- Mechanical energy ejects atoms
  - Compounds and alloys deposition is possible



Film growth & control parameters

- Plasma requires atoms between electrodes
  - Pressure in sputtering is higher than in evaporation
  - Atoms undergo many collision from target to substrate
  - Atoms deposit on substrate with random incidence angles
  - Good step coverage: 20-50%
- Reactive and co-sputtering is possible
- Collimated sputtering is possible





Cross section of sputtered Al on double layer lift-off resist.
Large metal sidewall coverage under the T shaped resist.
No real shadow effect.

Evaporation versus sputtering

Evaporation

- Simple & Fast
- Pure materials (elements)
- Does not allow depositing: composite and refractory materials
- High vacuum: long mean free path, micro-shadowing, grainy films
- But good for lift-off

Sputtering

- Deposition of compounds and refractory materials
- Good adhesion and step coverage
- Deposition of a large amount of material

Type of Material: Examples
Metals: Al, Cu, Zn, Au, Ni, Cr, W, Mo, Ti
Alloys: Ag-Cu, Pb-Sn, Al-Zn, Ni-Cr
Nonmetals: graphite, MoS_2, Ws_2, PTFE
Refractory oxides: Al_2O_3, Cr_2O_3, SiO_2, …
Refractory carbides: TiC, ZrC, HfC, NbC
Refractory nitrides: TiN, Ti_2N, ZrN, HfN, …
Refractory borides: TiB_2, ZrB_2, HfB_2, CrB_2, …
Refractory silicides: $MoSi_2$, WSi_2, Cr_3Si_2

PVD at CMi

- Equipment
- https://www.epfl.ch/research/facilities/cmi/equipment/thin-films/

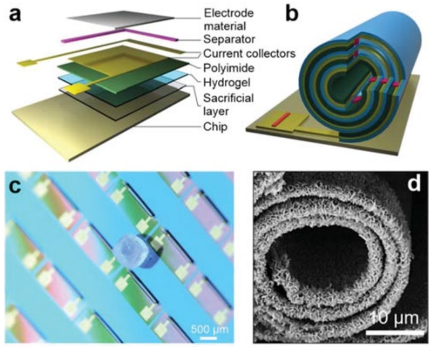
- Available PVD targets in Cmi
- https://www.epfl.ch/research/facilities/cmi/process/thin-films/available-pvd-targets-in-cmi/#Targets_Sputtering

Take home messages

- The rationale behind different microtechniques
- CVD: Why are there many different CVD approaches? What are the influencing factors behind?
- ALD and Thermal oxidation: what are the mechanisms behind?
- Electroplating
- **Metrology**
- PVD: Why are there many different PVD approaches? What are the influencing factors behind?
- Comparison of various approaches.

Course plan

- Tomorrow (Thu / 16 Oct):
  - MOOC self-study, lecture room is available, MOOC: **PVD**
- Next week: Autumn break!
- Wed / 29 Oct
  - SLT_#3 in groups, Topic: **PVD**
  - **My Inaugural lecture: 'Swiss Army Knife' Droplets for Iontronic Biointerfaces**
  - https://memento.epfl.ch/event/inaugural-lecture-swiss-army-knife-droplets-for-io/
- Thu / 30 Oct
  - MOOC self-study, lecture room is available, MOOC: **Lithography**
- Wed / 5 Nov
- **Lecture**, Topic: PVD recap, other deposition techniques, and Lithography
- **Guest lecturer**: Prof Minshen Zhu
  - How to fabricate batteries smaller than a grain of rice
  - How to fabricate '*Swiss Rolls*' in the clean room



See you next time!