Chair of Experimental Solid State Physics, LMU Munich

"Introduction to Graphene and 2D Materials"

SS24 Lecture 4, 06/04/2024

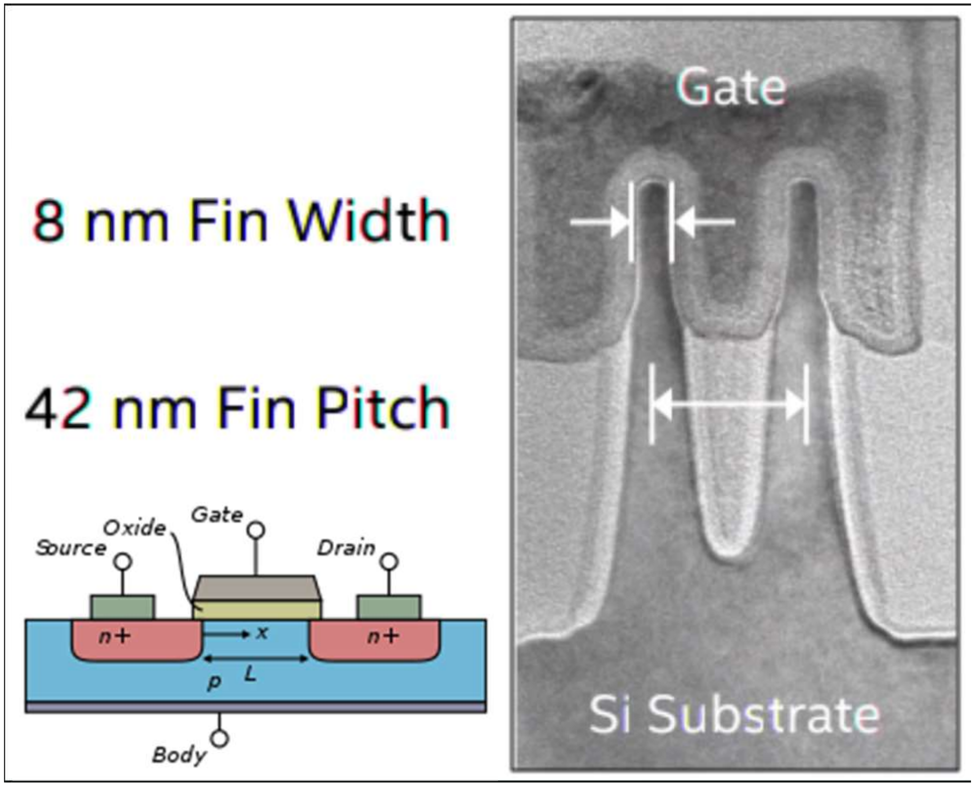
Outline - Lecture 4

- Introduction into Nanofabrication.
- Materials deposition techniques.
- Nanolithography techniques (creating nanoscale patterns).
- Etching techniques (defining nanoscale structures).
- Nano characterization techniques.
- Backend processing (wire-bonding, packaging).

Modern Electronics –semiconducting silicon ’50

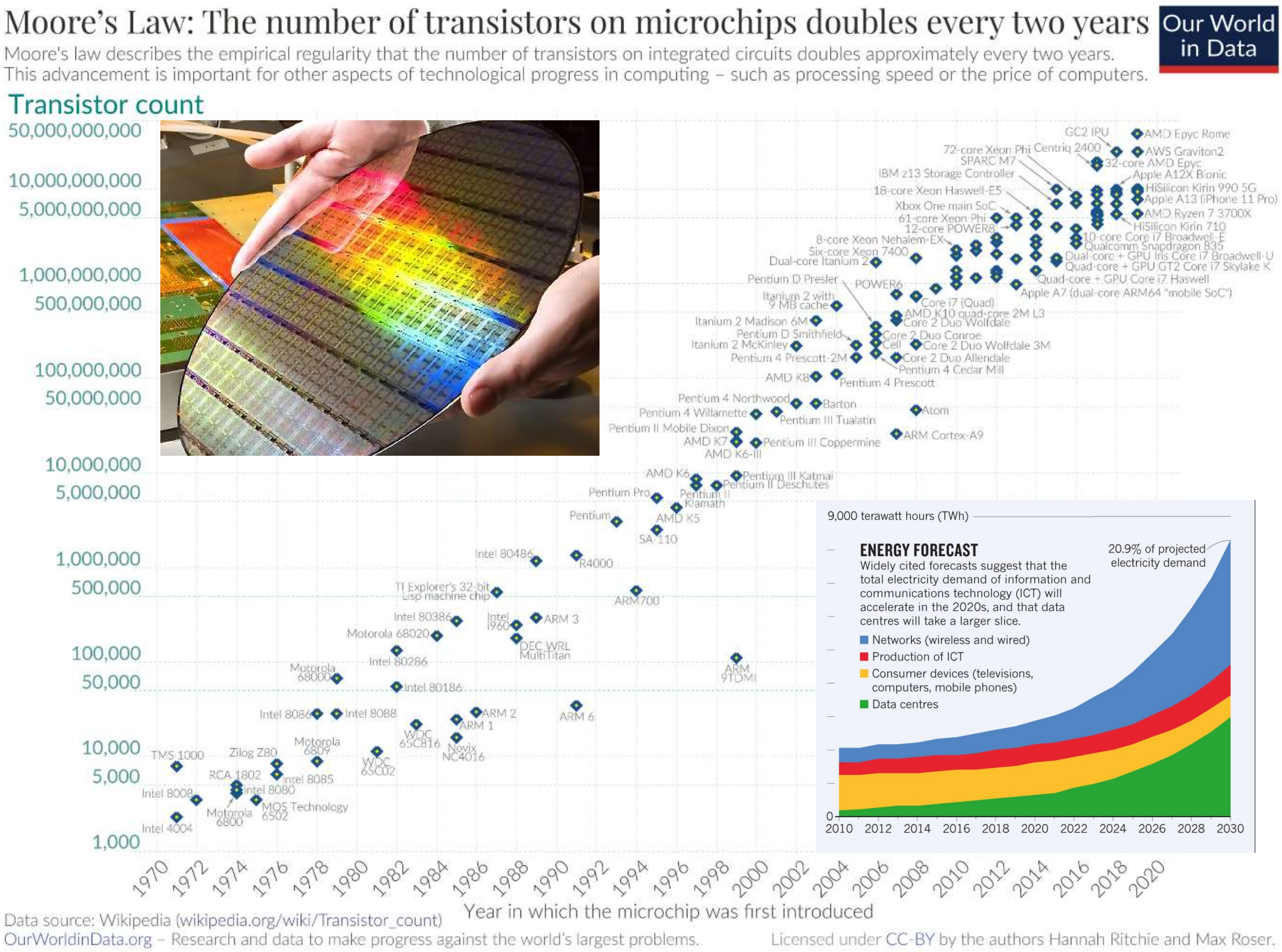
First field effect transistor:

IBM field effect transistor 2020:



→ Research and development led to miniaturization over 7 orders of magnitude from cm to nm scale.

Miniaturization – Moore’s law



Moore’s Law: The number of transistors on microchips doubles every two years

Moore's law describes the empirical regularity that the number of transistors on integrated circuits doubles approximately every two years. This advancement is important for other aspects of technological progress in computing – such as processing speed or the price of computers.

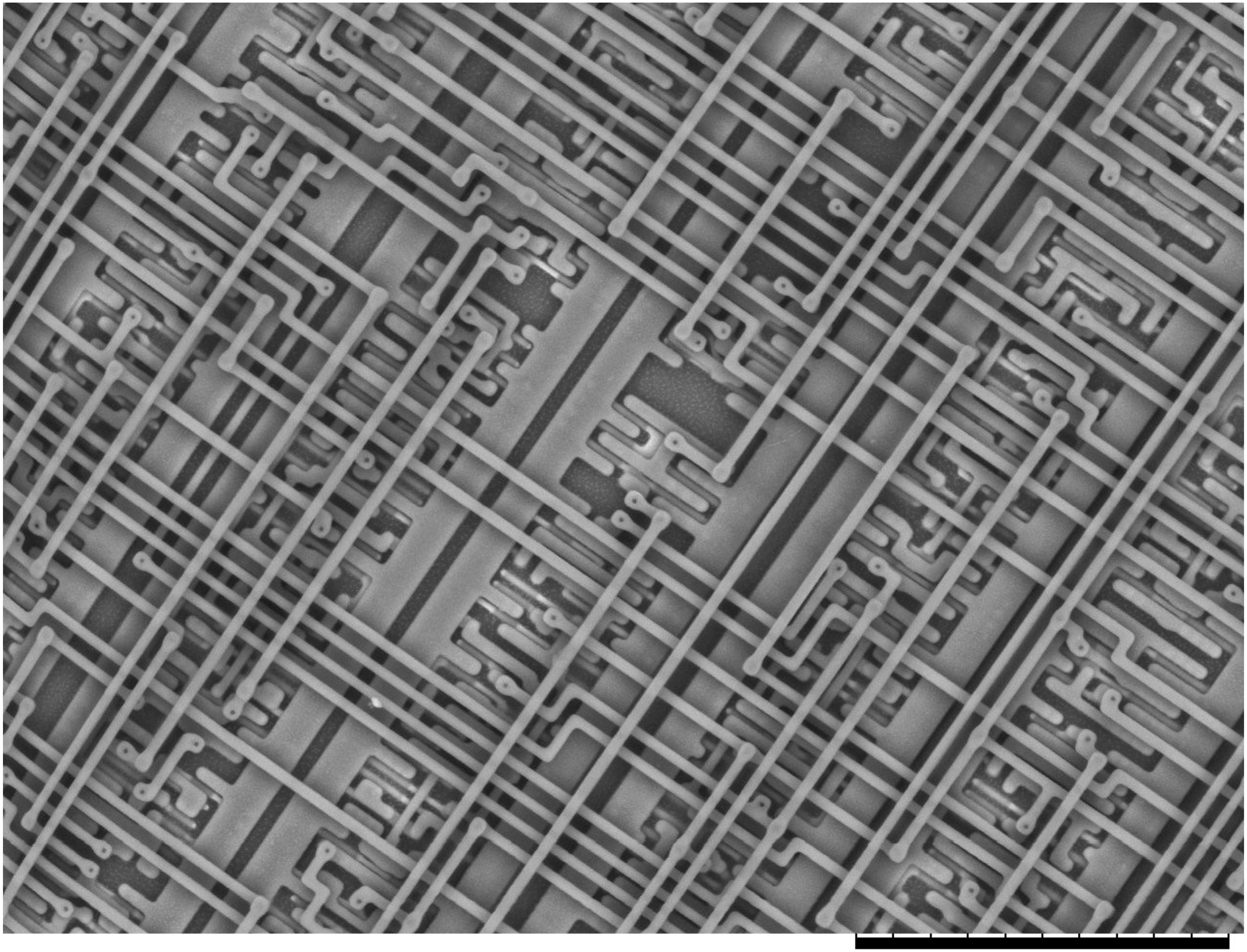
ENERGY FORECAST

Widely cited forecasts suggest that the total electricity demand of information and communications technology (ICT) will accelerate in the 2020s, and that data centres will take a larger slice.

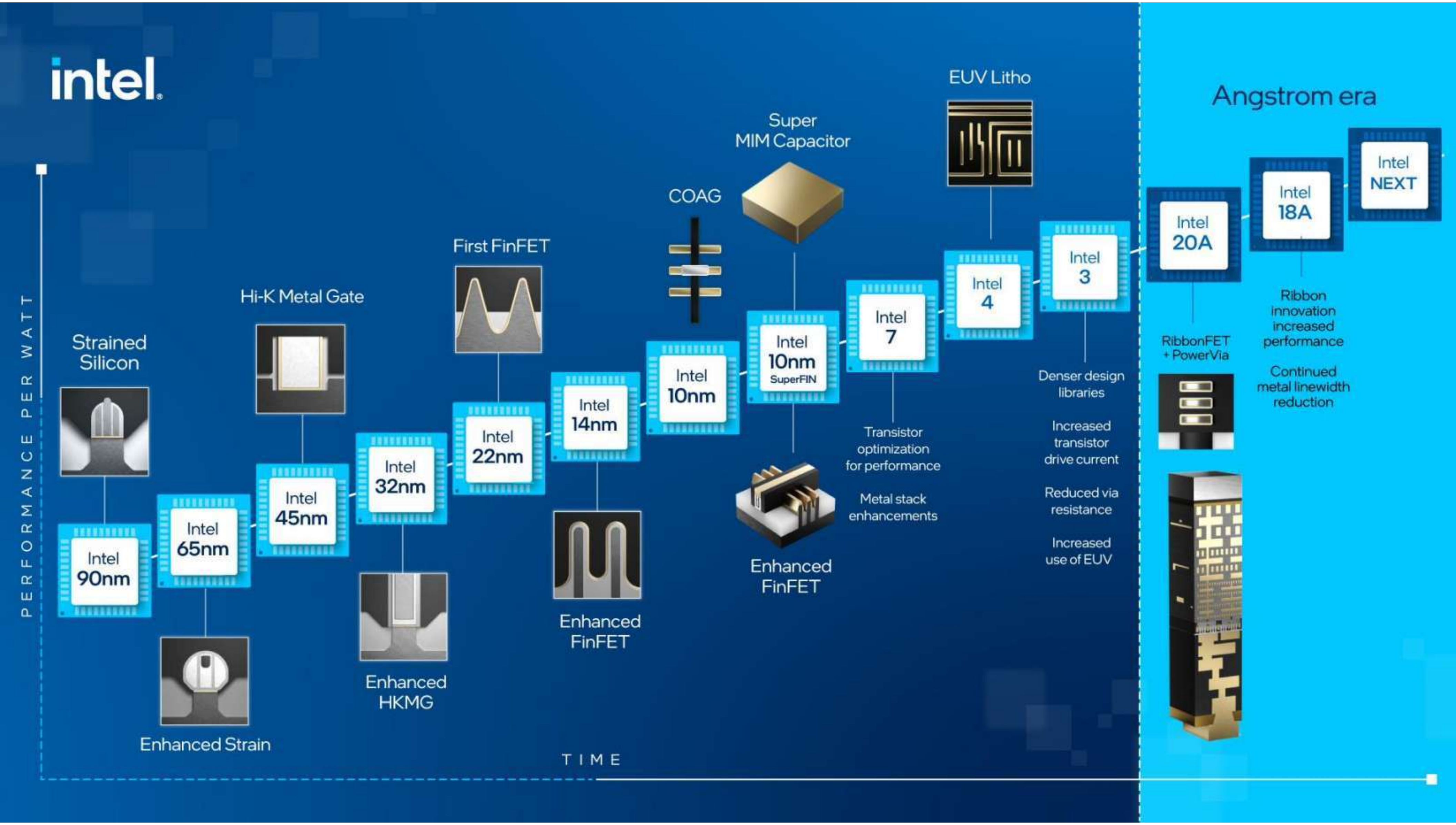
Data source: Wikipedia (wikipedia.org/wiki/Transistor_count)

OurWorldinData.org – Research and data to make progress against the world’s largest problems.

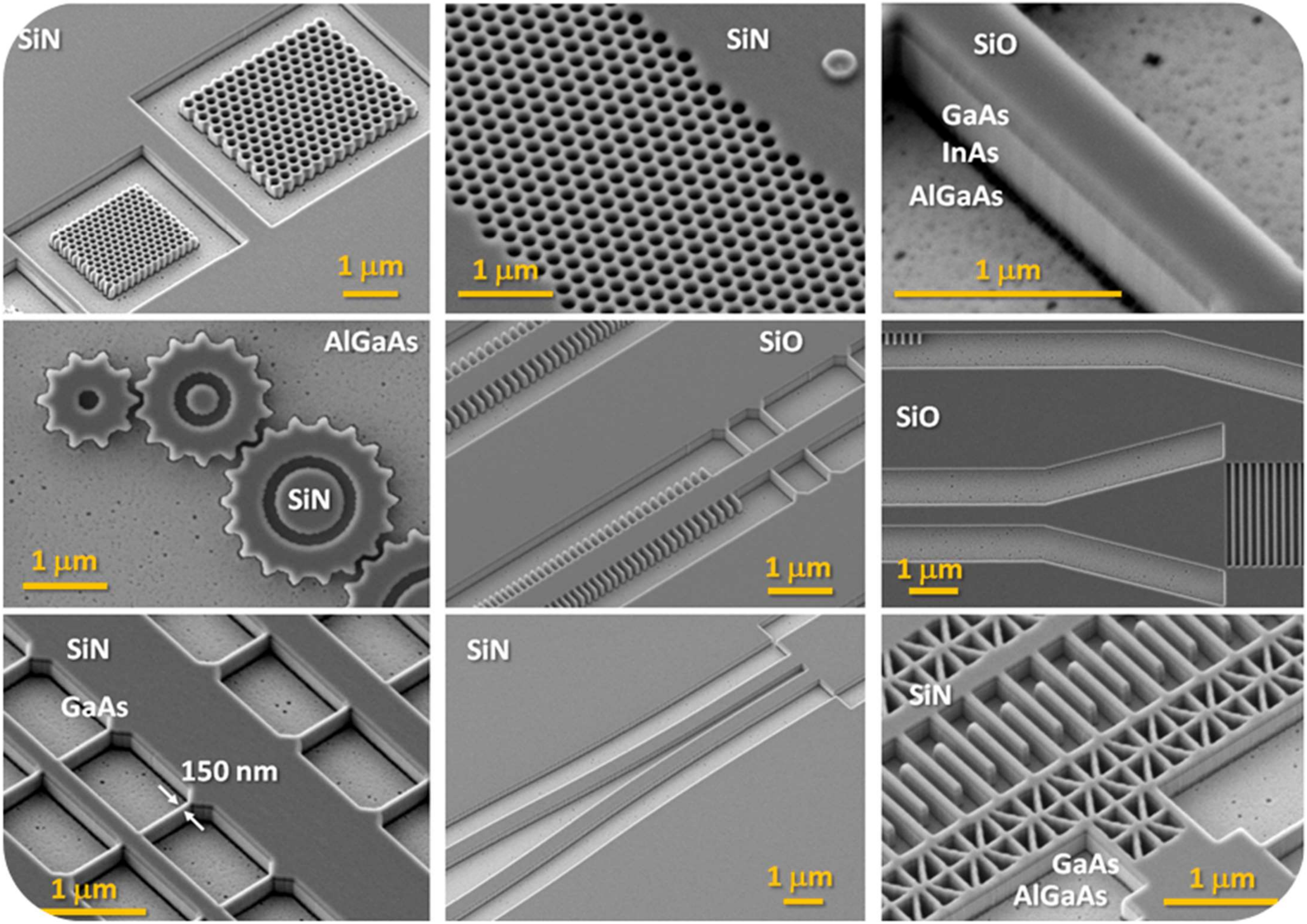
Wiring of transistors



Miniaturization – Moore's law

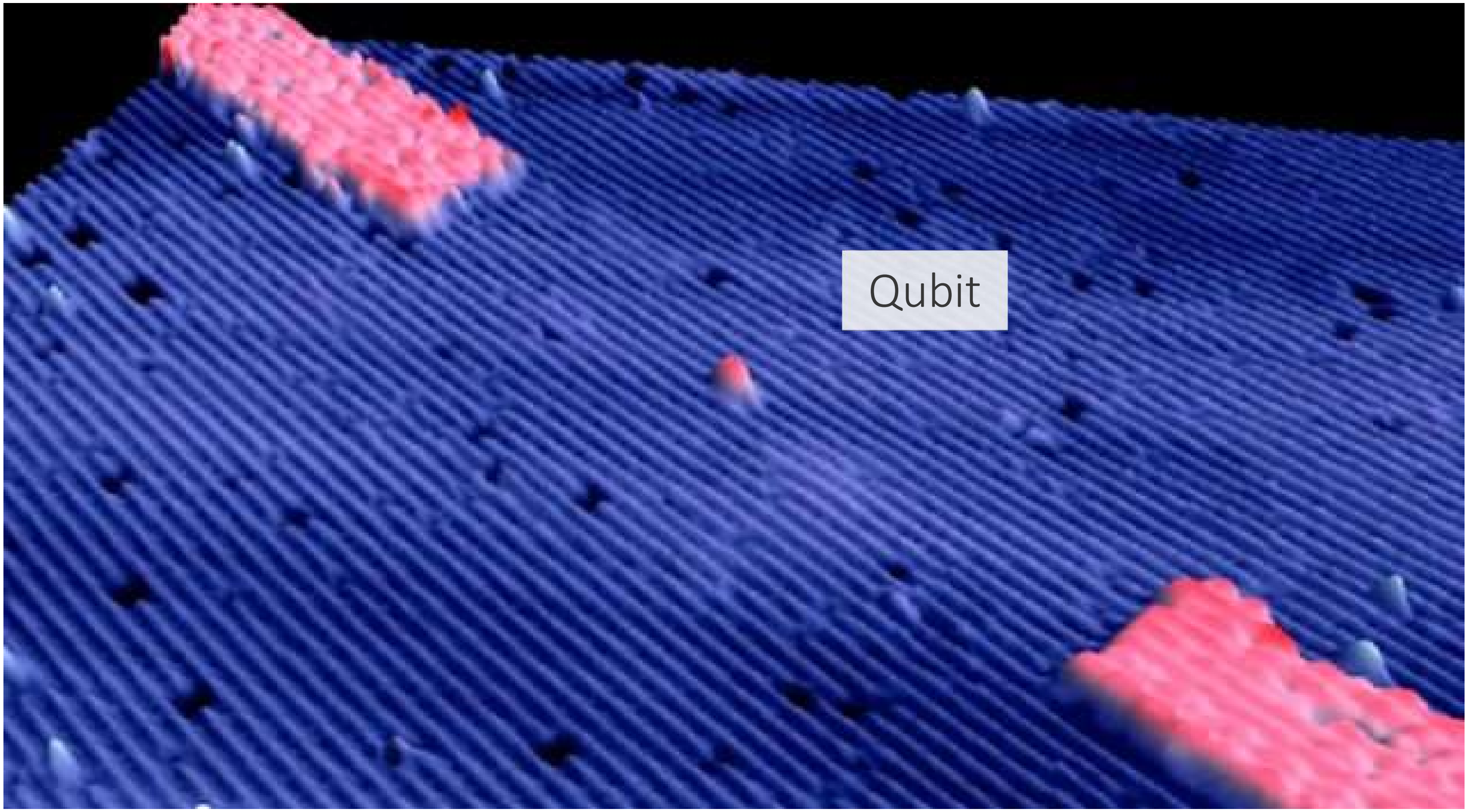


Advent of Nanotechnology



Quantum Technologies

Image of a single electron silicon transistor:



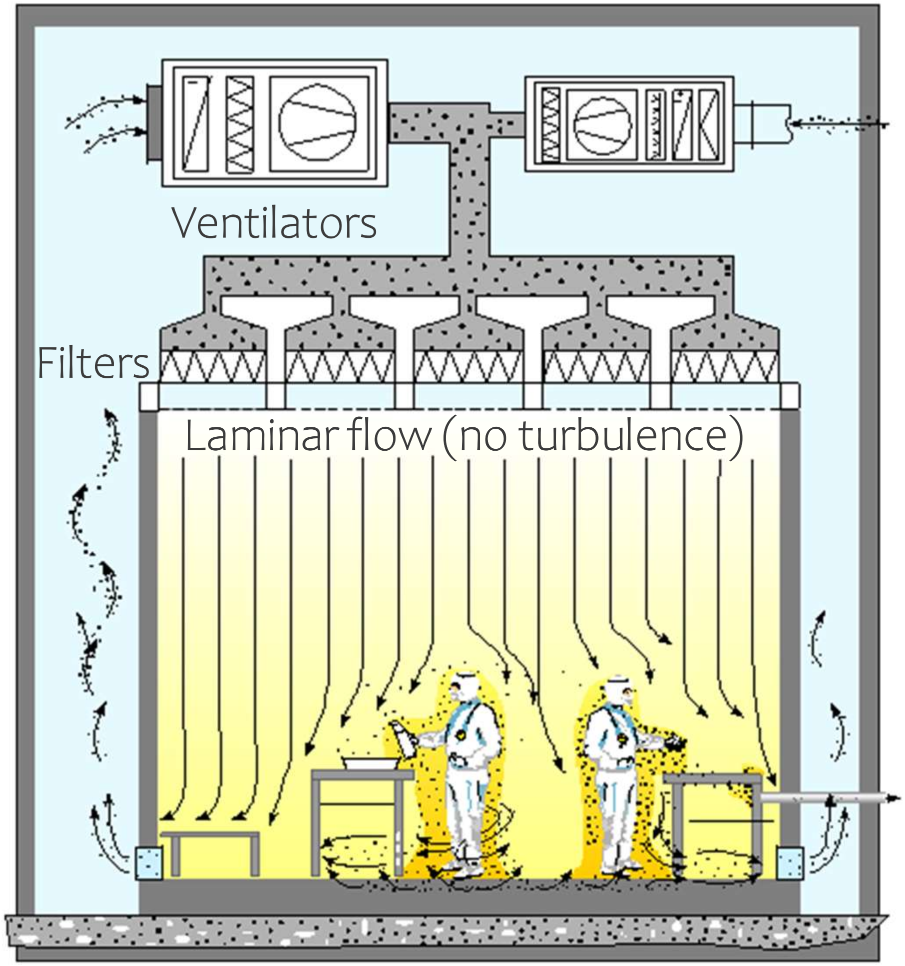
→ Transistors made of a single atom
→ Quantum mechanics plays key role
→ Qubits, can store quantum information

Cleanroom

- A **cleanroom** is an engineered space that maintains a very low concentration of airborne particulates. It is well isolated, well controlled from contamination, and actively cleansed. This are key to fabricate large scale wafers will billions of nanostructures, with the desired doping levels and free of any type of contaminants.

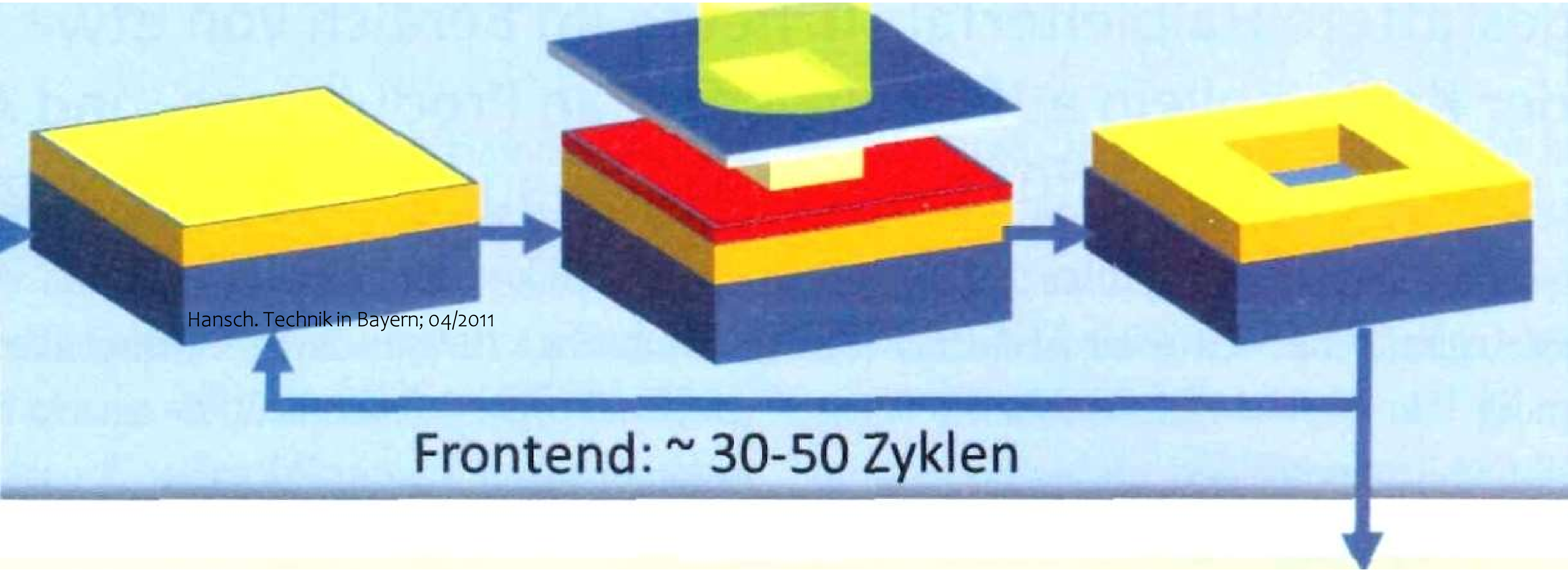
- Classification of cleanrooms: DIN EN ISO 14644 - ISO class n → less than 10n particles smaller 0,1 µm pro m^3

Cleanroom crossection:



Cleanroom impressions:

Frontend micro / nano fabrication



Deposition

Lithography

Etching

Hansch. Technik in Bayern; 04/2011

Backend

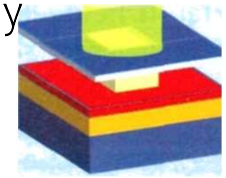
dicing, bonding, electrical tests

Fabrication processes

Deposition

- Evaporation
- Sputtering
- Molecular Beam Epitaxy
- (MBE)
- VdW Stacking
- Plasma Enhanced (CVD)
- Atomic Layer Deposition
- Electroplating
- Low Pressure CVD
- …

Lithography



- Shadow Impact Lithography
- Projection Lithography
- Laserlithography
- Electron Beam Lithography
- Nano Imprint Lithography (UV or thermal)
- 3D Printing
- …

Etching

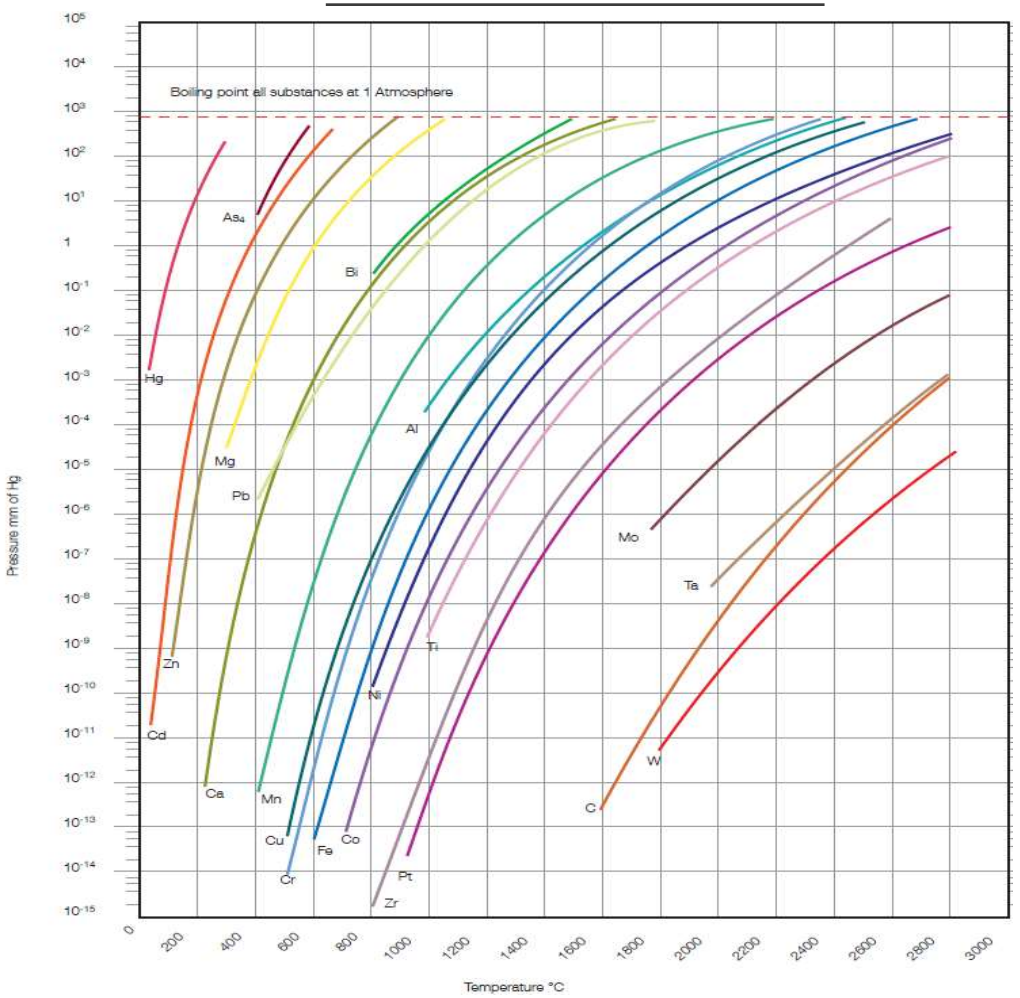
- Wet Etching
- Electrochemical Etching
- Plasma Etching
- Ion Beam Etching
- Reactive Ion Etching
- Inductively Coupled RIE
- …

Characterization

- Atomic Force Microscopy (AFM)
- Scanning Electron Microscopy (SEM)
- Ellipsometry
- Profilometry
- White Light Interf.
- Confocal Laser Microscopy
- Energy Disp. X-Ray Spectroscopy (EDX)
- Raman Spectroscopy
- …

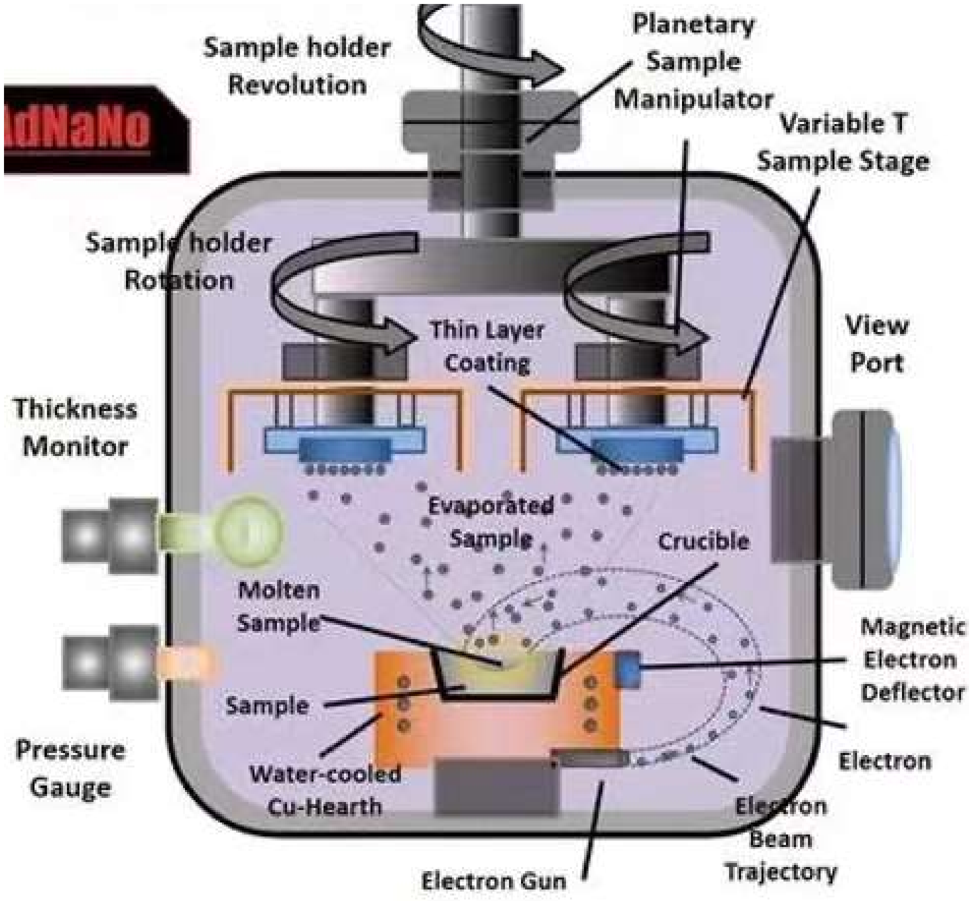
Evaporation of materials

Cleanroom crossection:



Vacuum evaporation is a form of physical vapor deposition. Such a technique consists of pumping a vacuum chamber to pressures of less than 10^{-5} torr and heating a material to produce a flux of vapor to deposit the material onto a surface.

E-beam evaporation:

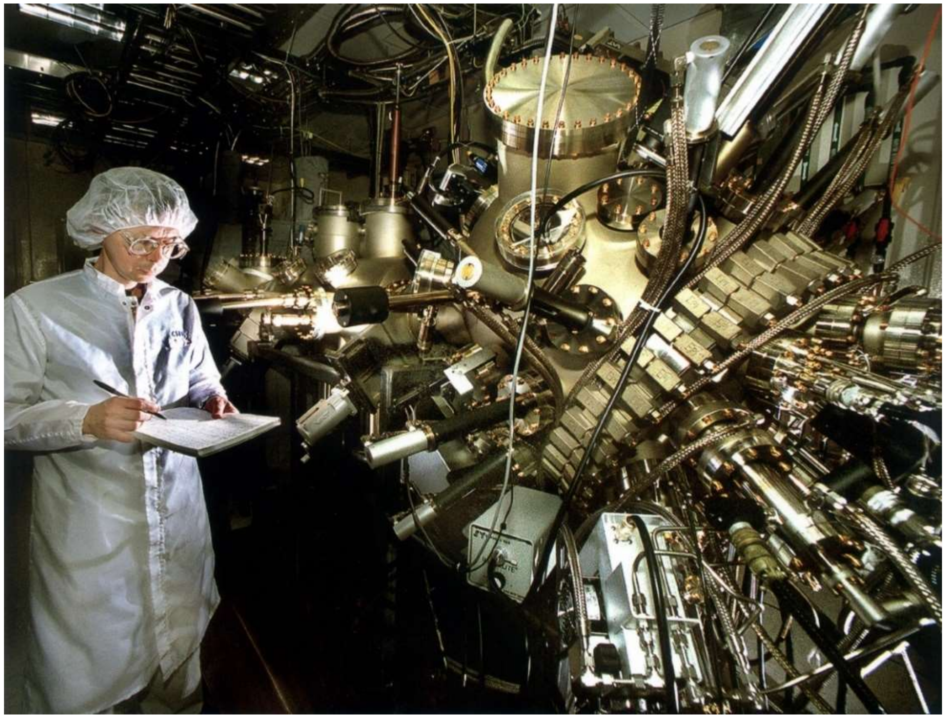


Similar:

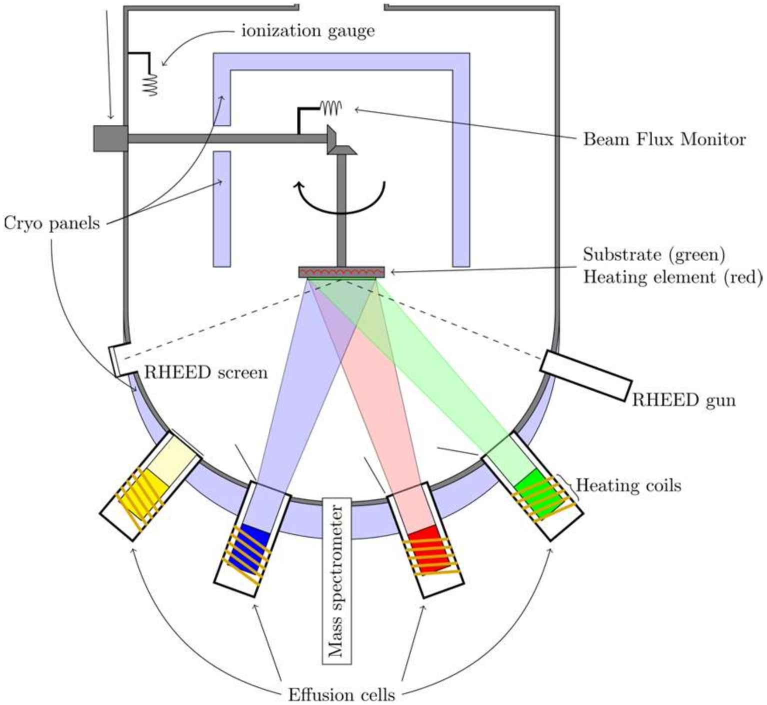
- Thermal evaporation
- Sputtering
- Etc.

Molecular beam epitaxy

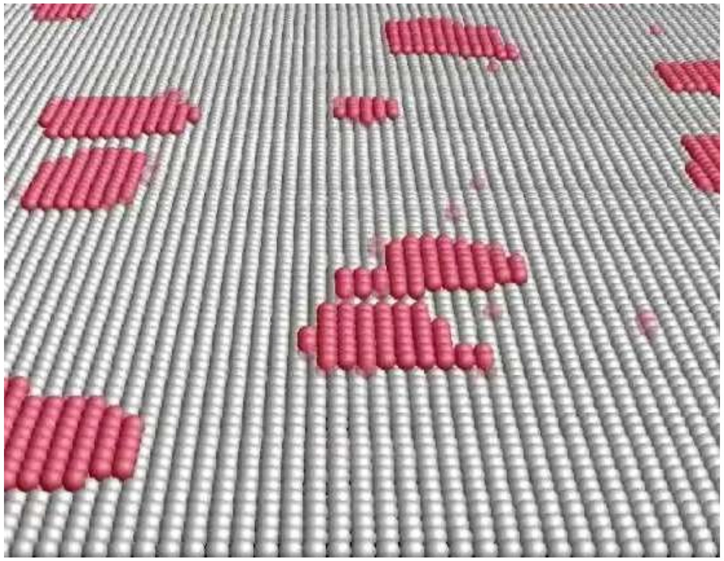
MBE machine:



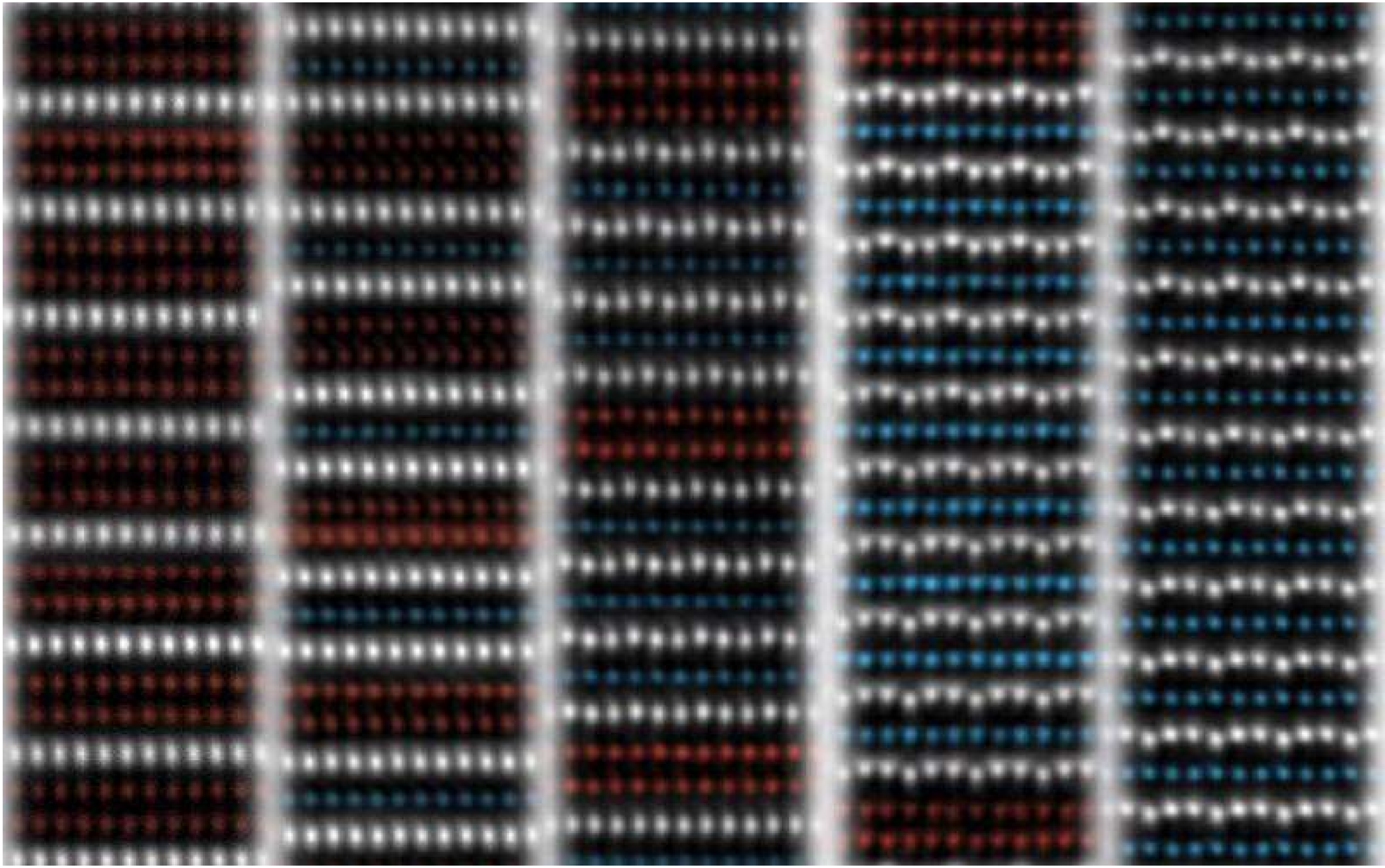
MBE crossection:



Molecular-beam epitaxy takes place in high vacuum or ultra-high vacuum (10^{-8}–10^{-12} Torr). The most important aspect of MBE is the deposition rate (typically less than 3,000 nm per hour) that allows the films to grow epitaxially. These deposition rates require proportionally better vacuum to achieve the same impurity levels as other deposition techniques. The absence of carrier gases, as well as the ultra-high vacuum environment, result in the highest achievable purity of the grown films.

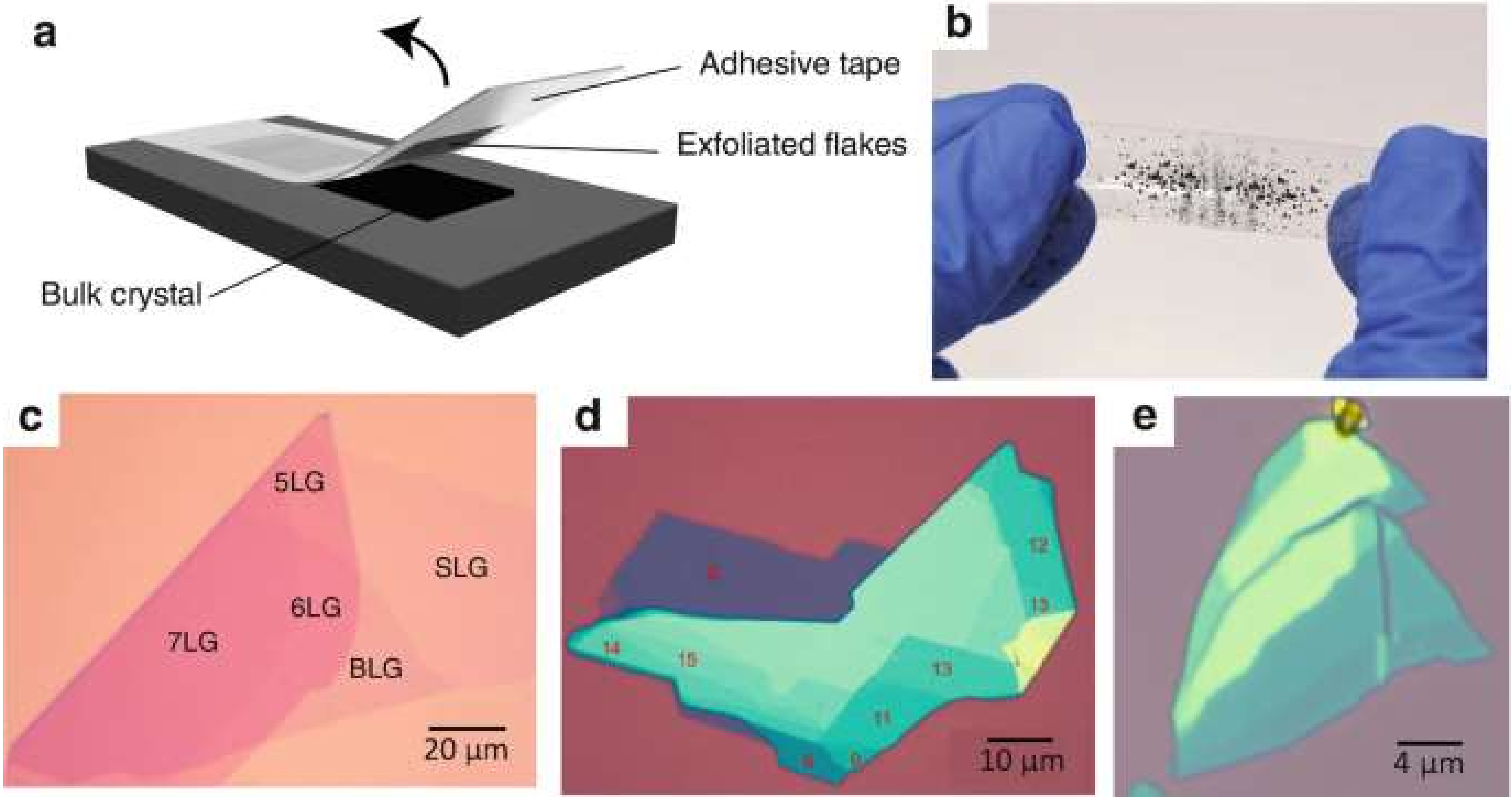


Heterostructures – Molecular LEGO



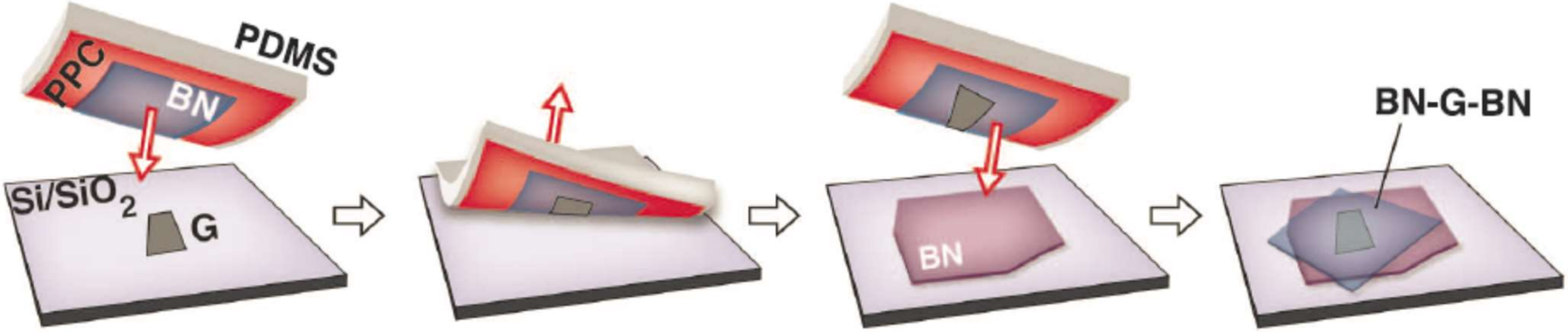
Schlom group (2016).

Mechanical exfoliation of 2D van der Waals materials



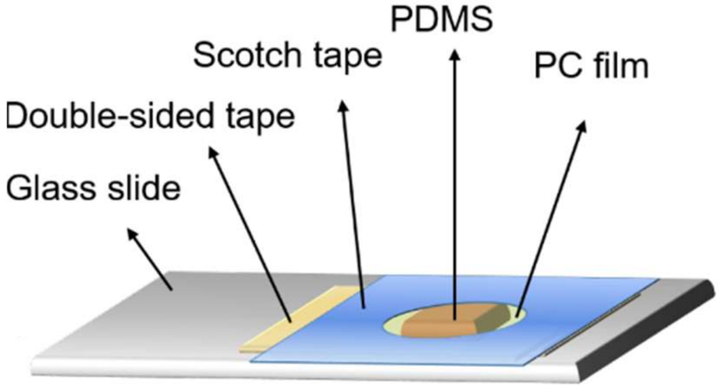
VdW heterostructures – clean coupling over < 1nm

vdW co-lamination transfer technique:

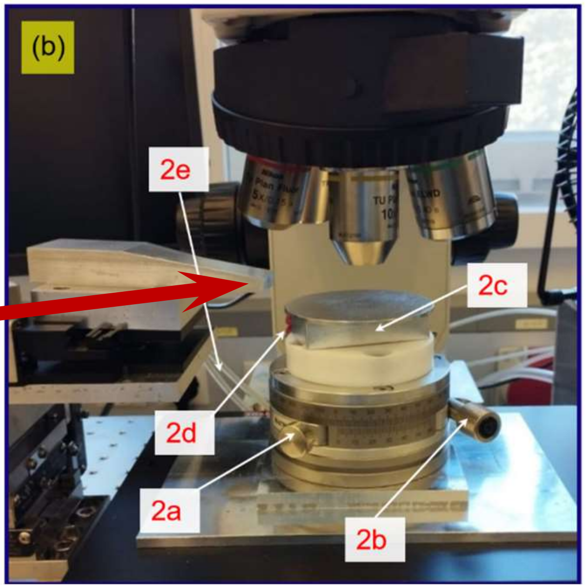


A. Geim, et. al. Nature (2013). C.Dean, P. Kim, J. Hone, et. al. Science (2013).

Transparent 2D materials stamp:

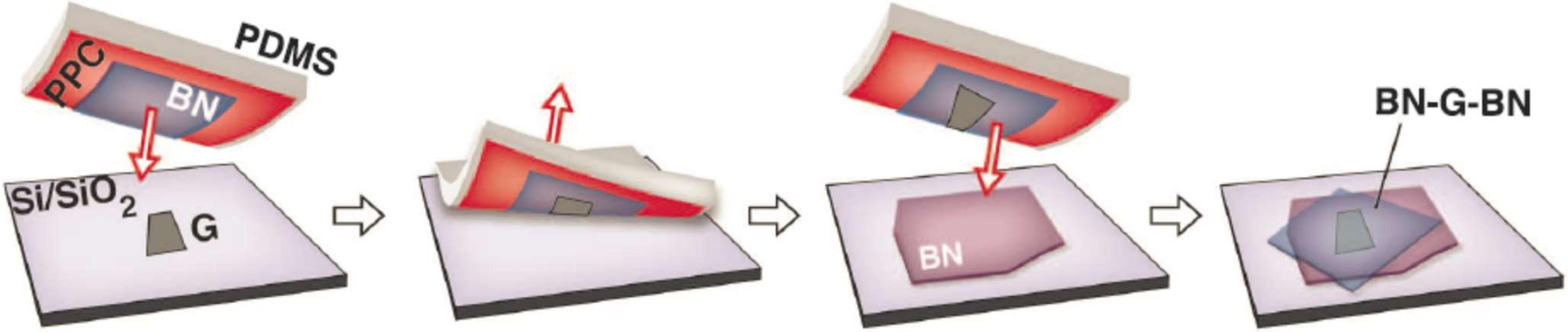


2D stamping stage:



VdW heterostructures – clean coupling over < 1nm

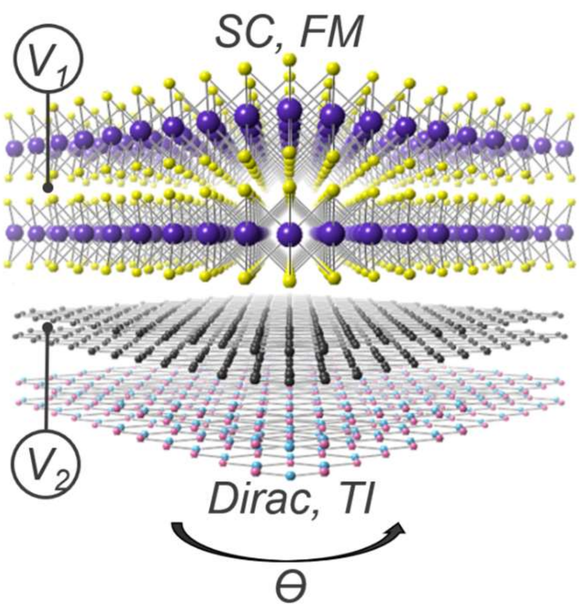
vdW co-lamination transfer technique:



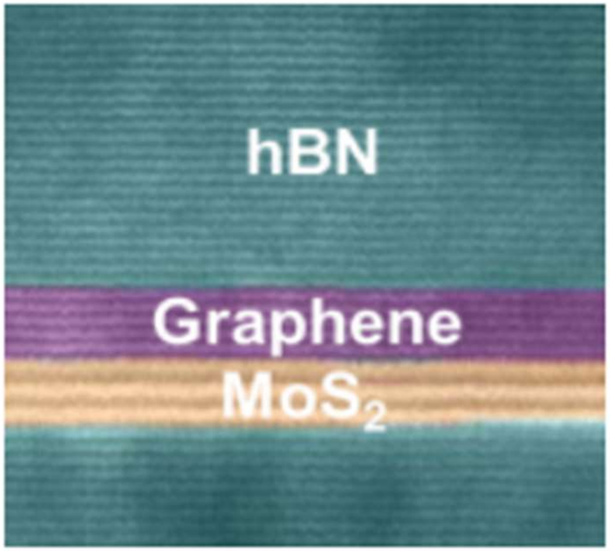
A. Geim, et. al. Nature (2013).

C.Dean, P. Kim, J. Hone, et. al. Science (2013).

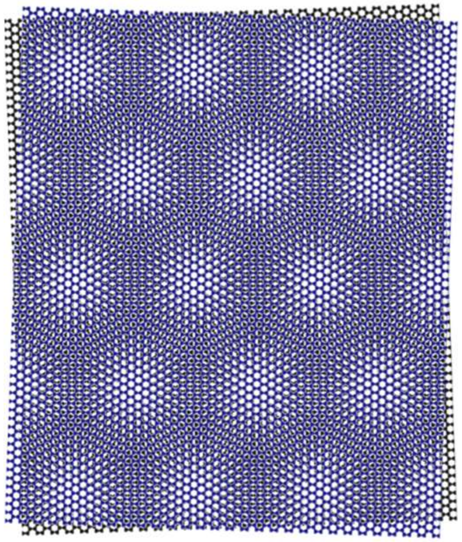
Designer vdW stack:



TEM cross-section:

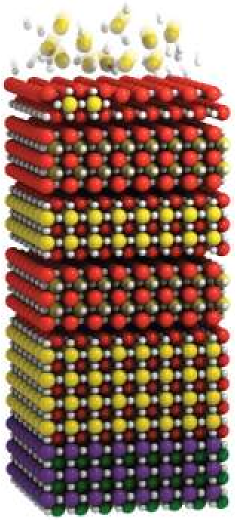


Moiré superlattice:

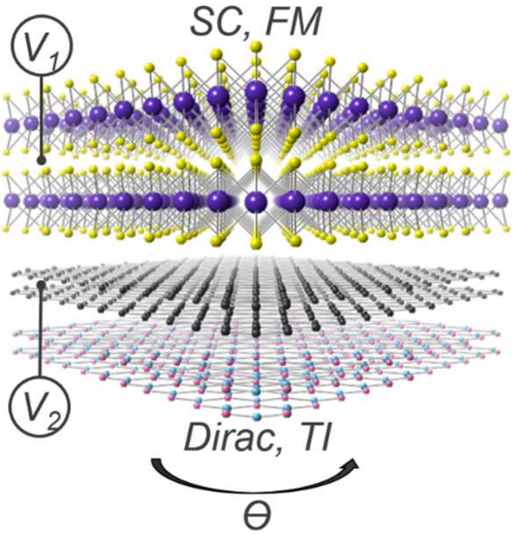


MBE (LEGO) vs. vdW (CARDS)

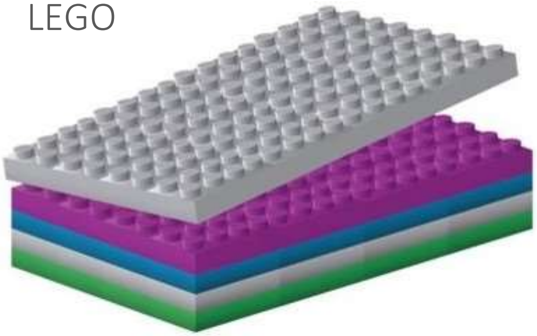
Molecular beam epitaxy:

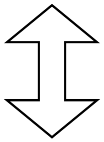


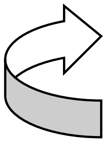
Van der Waals assembly:



LEGO







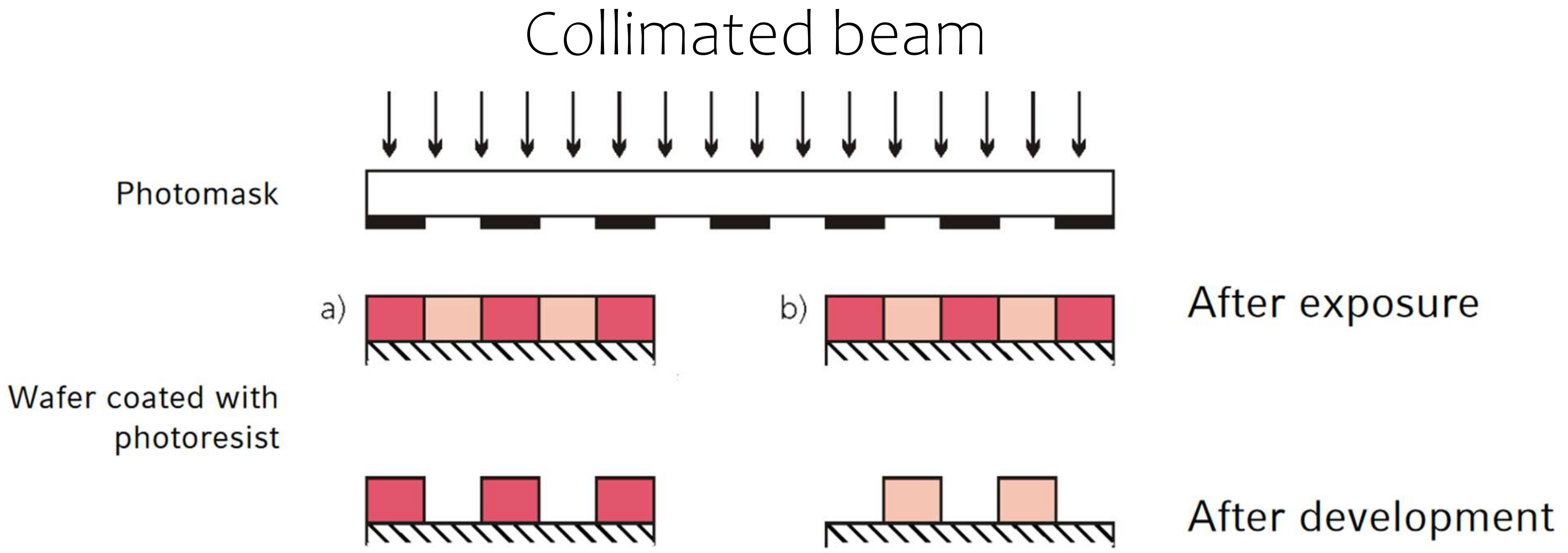
CARDS

Crystallographic orientation locked

Crystallographic orientation free

New → Free crystallographic orientation

Shadow Impact Photolithography



a) Positive tone photoresist

Typical composition: phenol resin (Novolak), photo-active compound (often DNQ), solvents

PAC acts as dissolution promotor

Exposed photoresist gets dissolved, shadowed regions remain

b) Negative tone photoresist

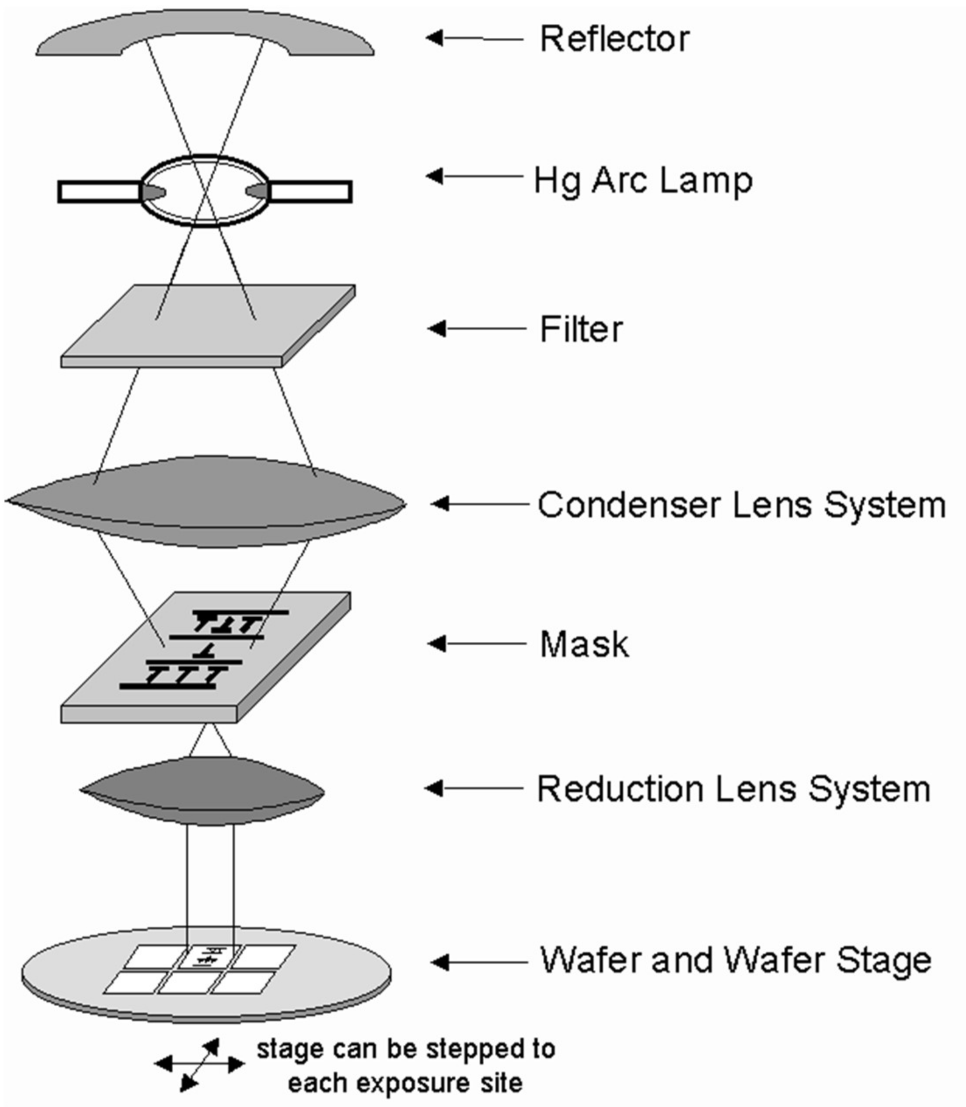
Typical composition: acrylic resin, photoinitiator, solvents

Crosslinking or polymerization (chain growth) induced by UV irradiation

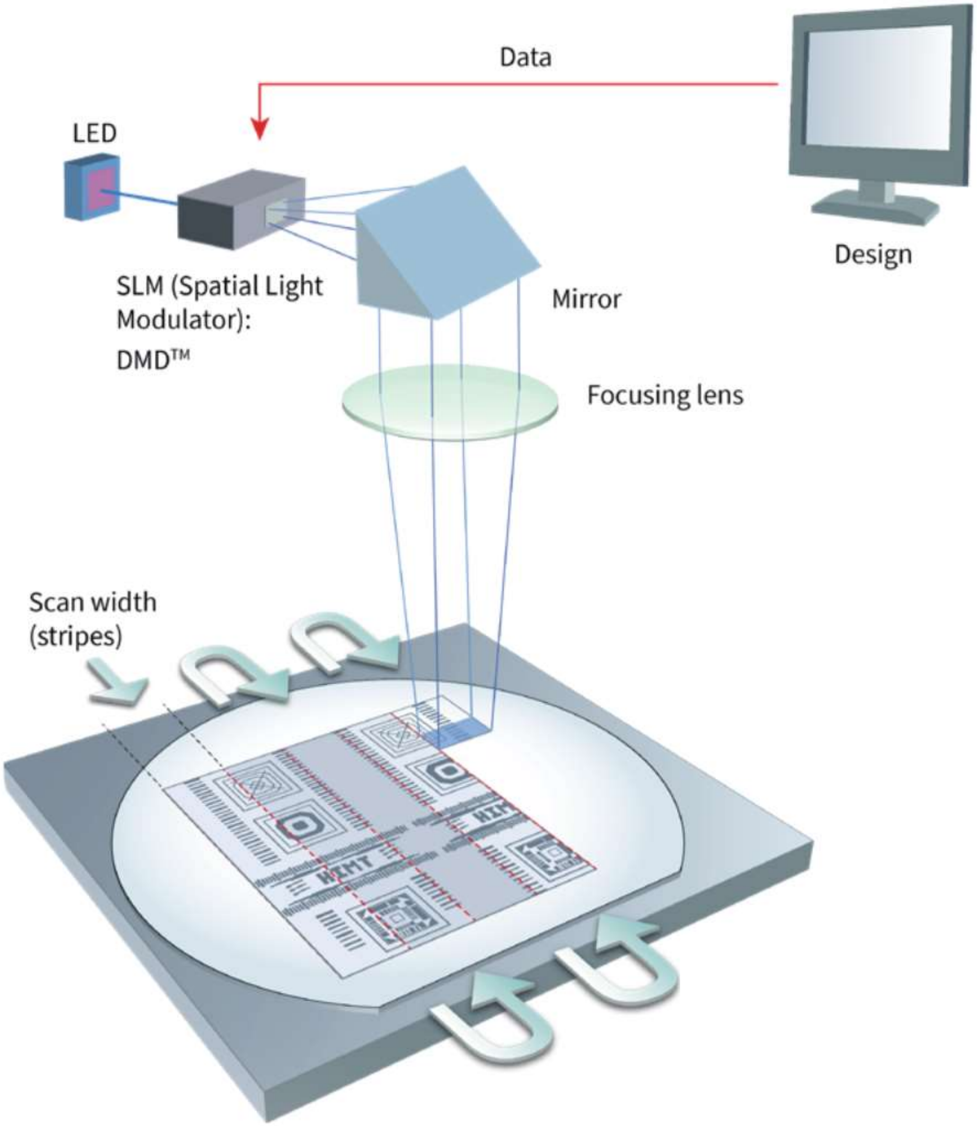
Low dissolution rate of exposed photoresist

Photolithography

Shadow mask:



Laser writer:

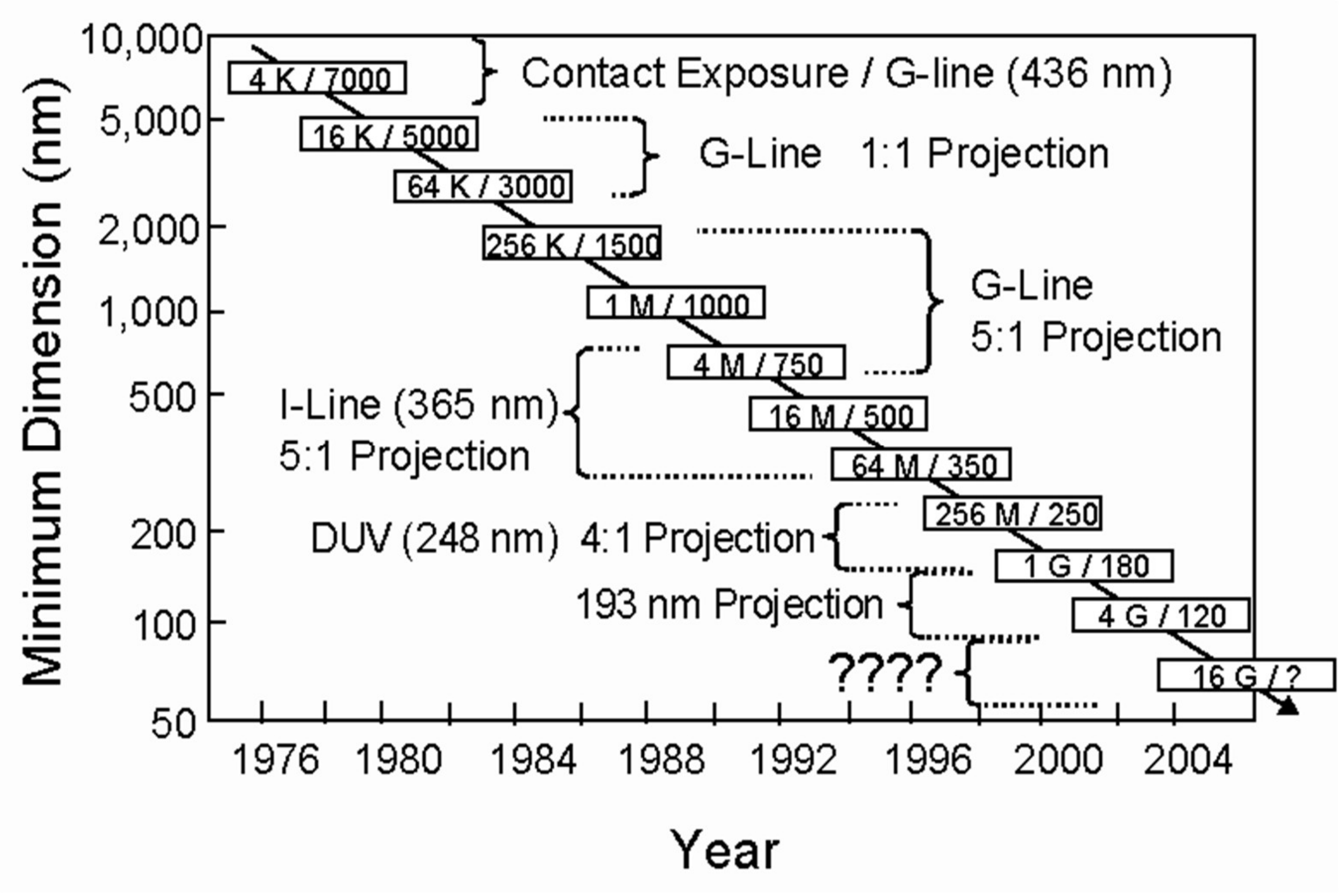


Evolution of Photolithography

- **The Abbe diffraction limit for a microscope states:** Light with wavelength λ, traveling in a medium with refractive index n and converging to a spot with half-angle θ will have a minimum resolvable distance of d:

$$d = \frac{\lambda}{2n \sin\theta}$$

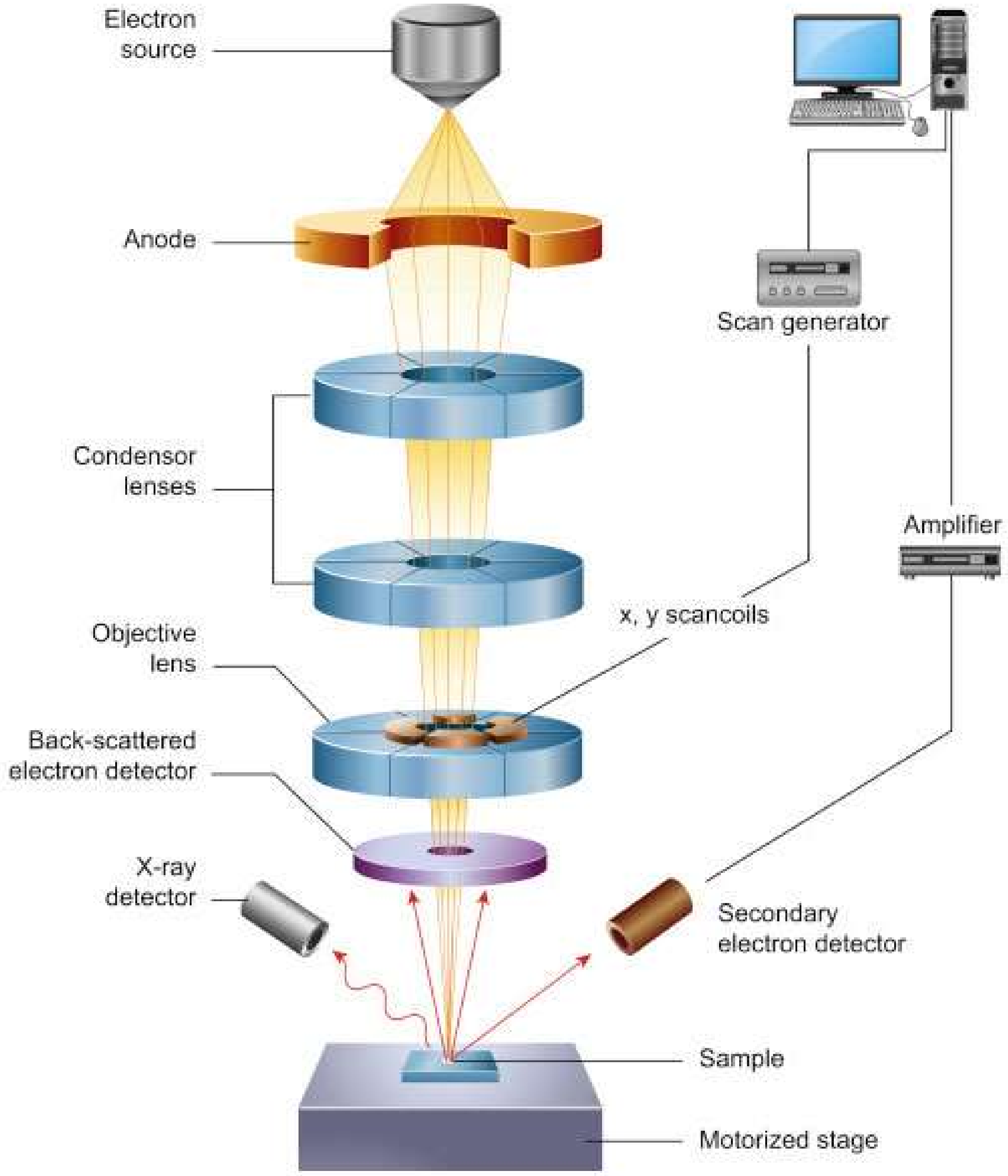
- To increase the resolution, shorter wavelengths can be used such as UV and X-ray microscopes. These techniques offer better resolution, but ultimately also hit a natural wavelength limit at several nm.



Dmitri K. Efetov

Chair of Experimental Solid State Physics

E-beam lithography (vector scan)

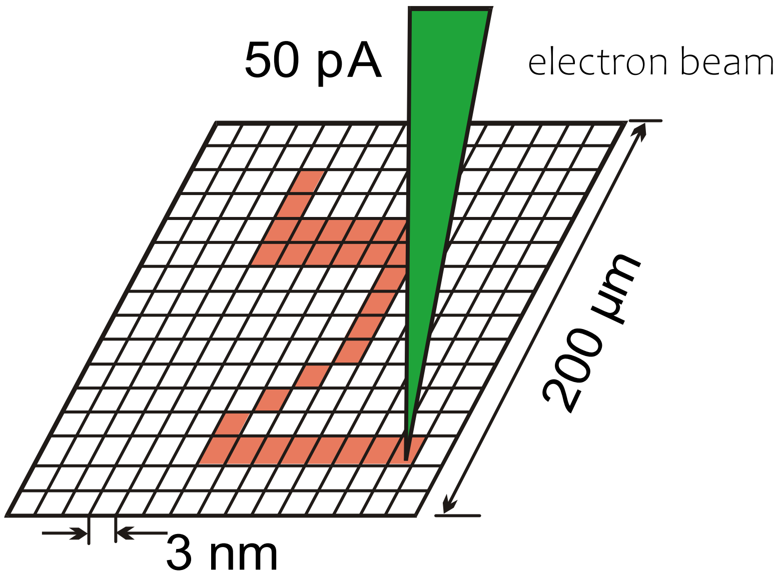


- The acceleration of electrons in an electron beam gun with the acceleration voltage Va results in the corresponding de Broglie wavelength

$$\lambda_{\text{de Broglie}} = \frac{h}{p_e} = \frac{h}{m_e \cdot v_e}$$

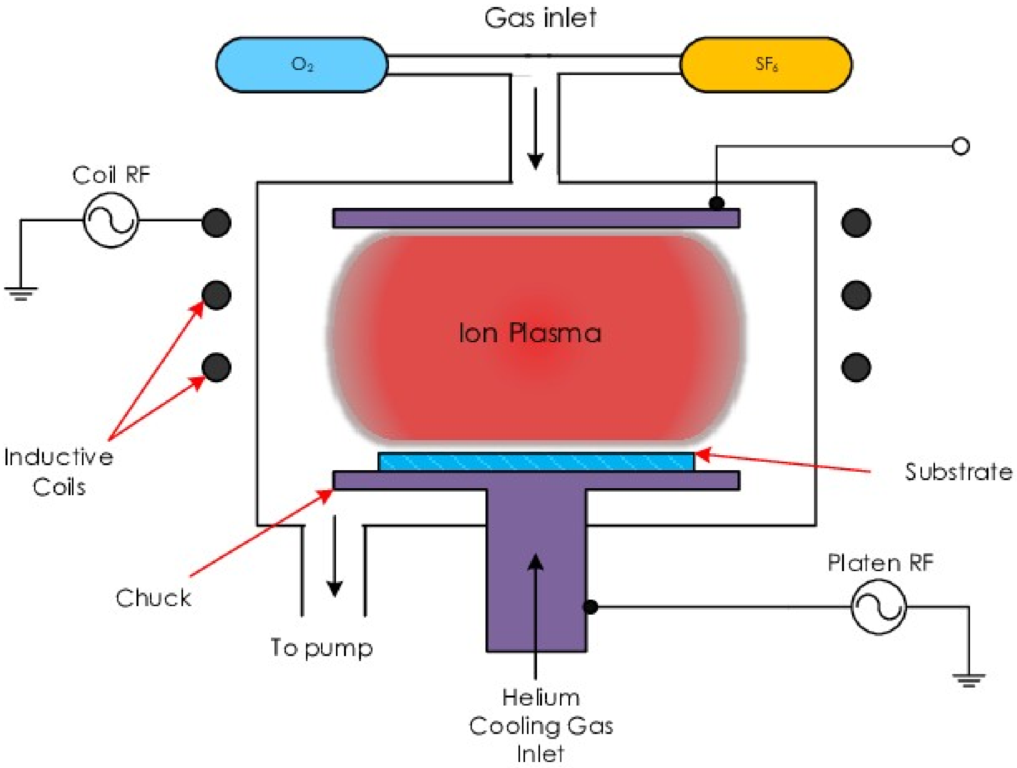
$$= \frac{h}{m_e \cdot \sqrt{2 \cdot \frac{e}{m_e} \cdot V_a}} = \frac{h}{\sqrt{2 \cdot m_e \cdot e \cdot V_a}}$$

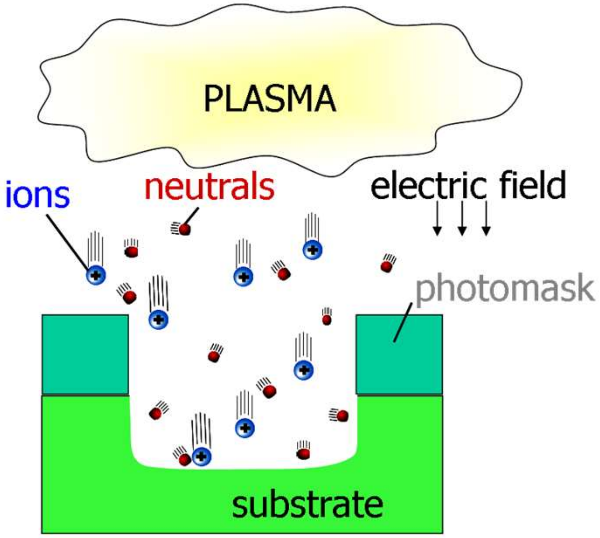
$V = 30\text{keV} \rightarrow \lambda = 7.06 \cdot 10^{-12}\text{m}$



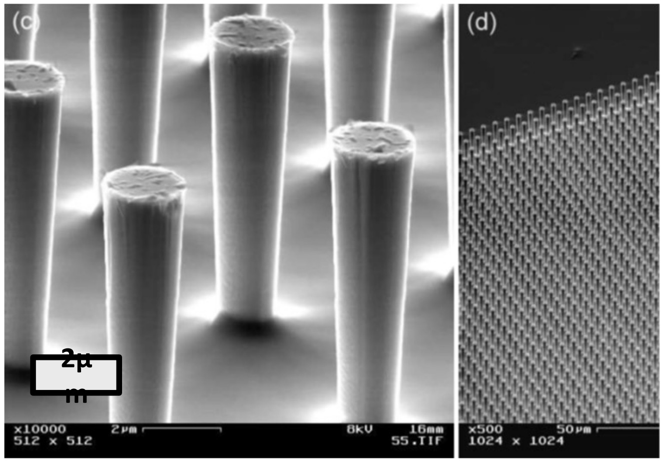
Main Principle of Reactive Ion Etching

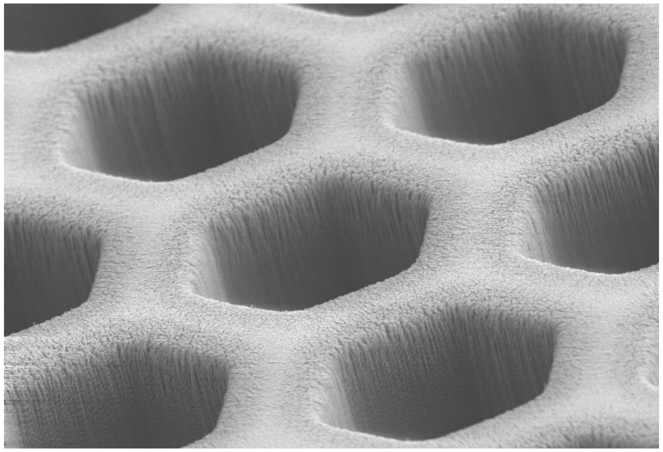
- Etching: breaking chemical bonds and turning atoms from solid state to volatile etching products
- Low pressure plasma (dissociation, ionisation): main mechanisms, physical component (Ions) weakens bonds on the surface, and chemical component (Radicals) forms volatile by-products
- Generating an ion flux: through charging of the plasma needs an RF generator @13.56 MHz: difference in mobility of ‚heavy‘ ions and ‚light‘ electrons
- Advantages of Reactive Ion Etching: potentially highly anisotropic, ions follow the field line. Good selectivity defined by the reactivity of the radicals. Controllable, stable etch rate.

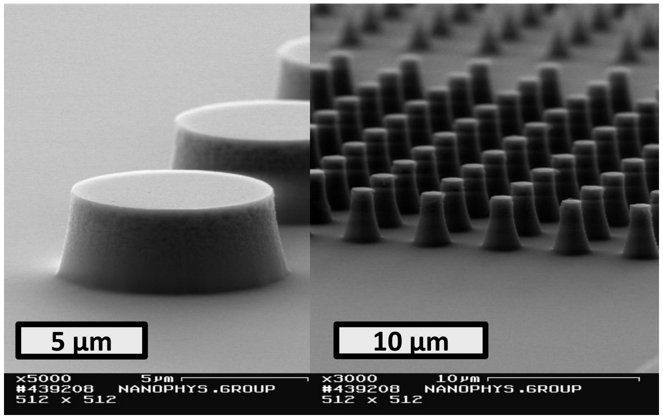




Chemistry of Reactive Ion Etching





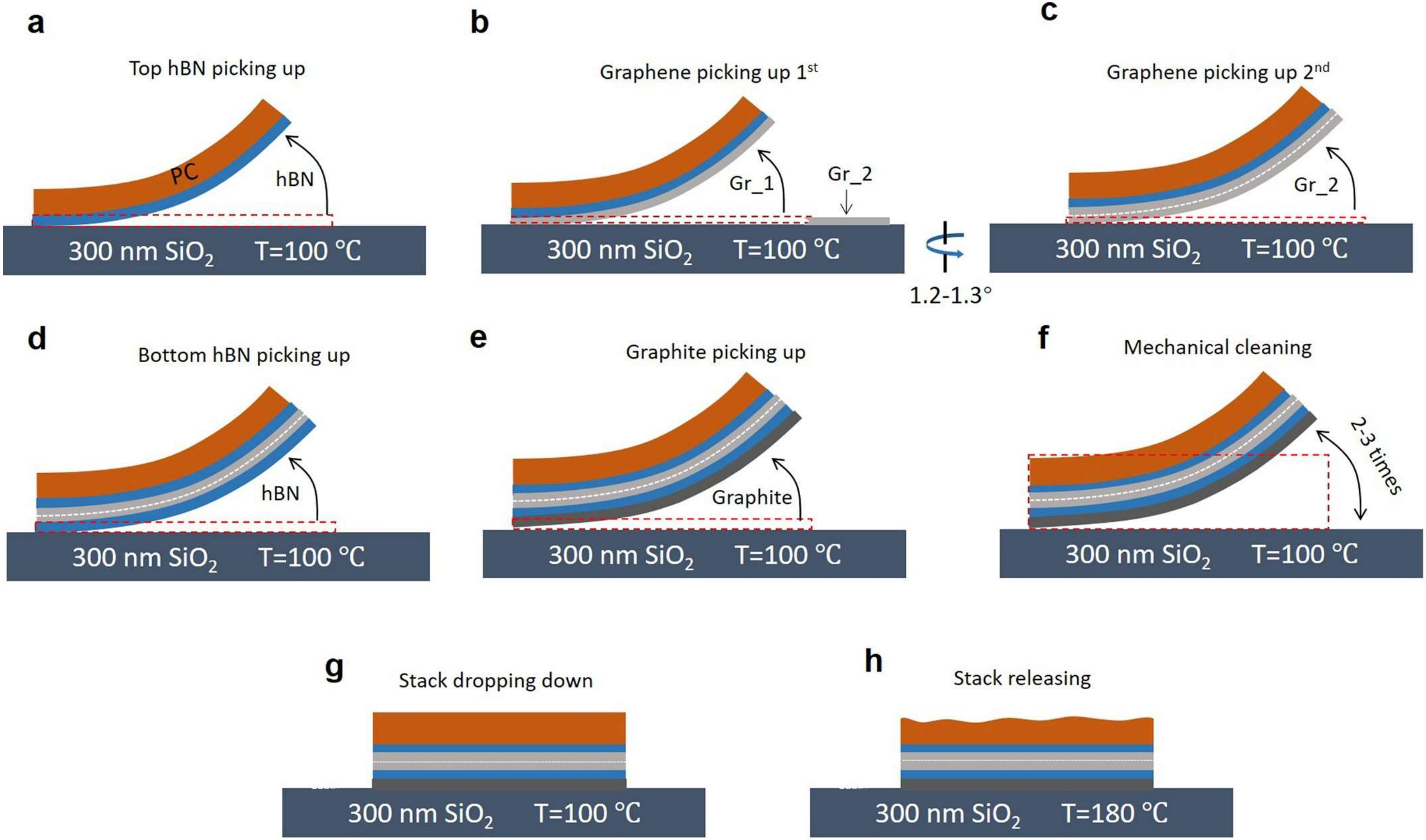


- **Chlorine based chemistry** (SiCl4, BCl3, Cl2…) for most **metals** (Al, Cr, Au, Pt) and **compound semiconductors** (III-V, e.g. GaAs)

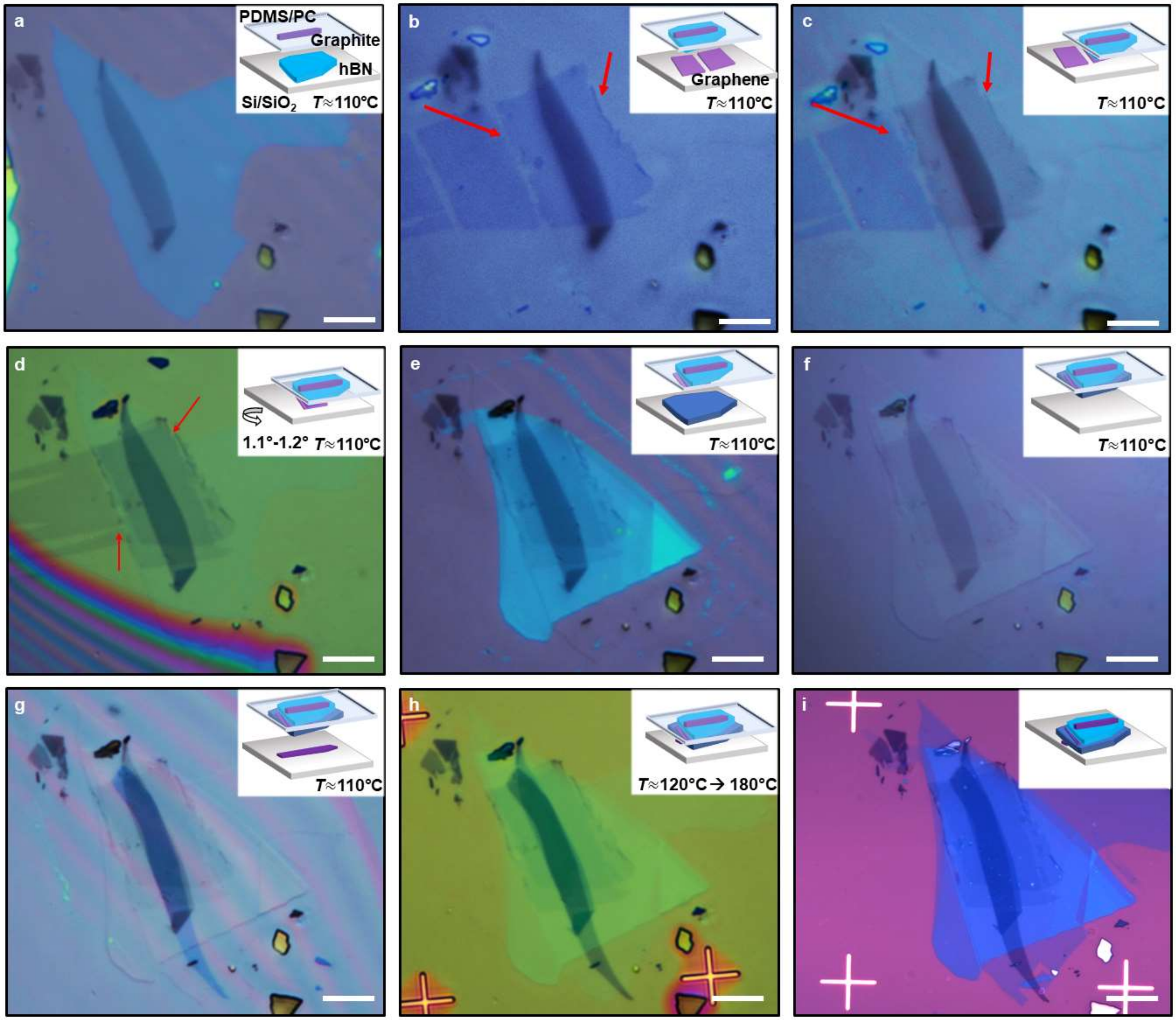
- **Oxygen (O2)** for Carbon based materials (mostly Polymers) by forming CO and CO_2

- **Fluorine based chemistry** (SF6, CHF3, CF4…) for **Silicon**, Silicondioxide etc. and some **metals** (Ti) Si + 4 F → SiF4 (gas)

Typical process flow for graphene device - stacking

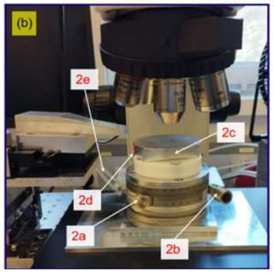


Typical process flow for graphene device - stacking

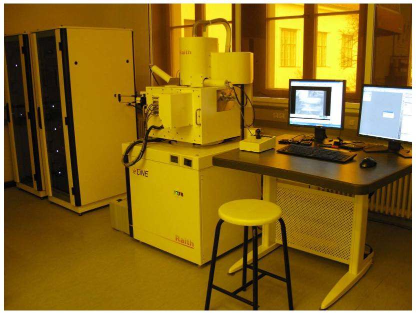


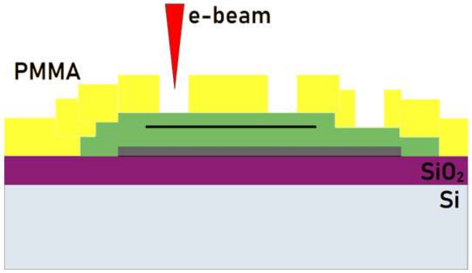
Typical process flow for graphene device - nanofab

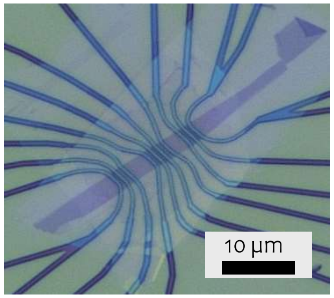
1. Exfoliation and stacking



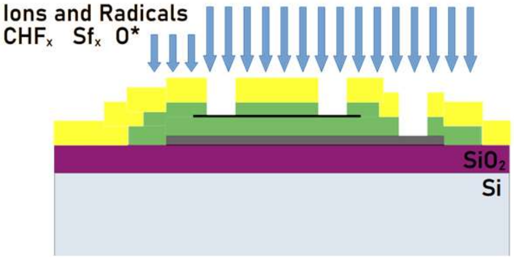
2. E-Beam Lithography

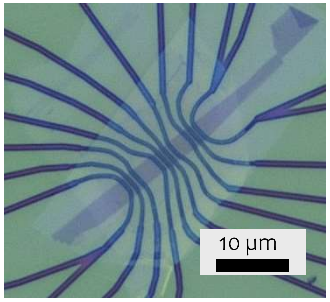






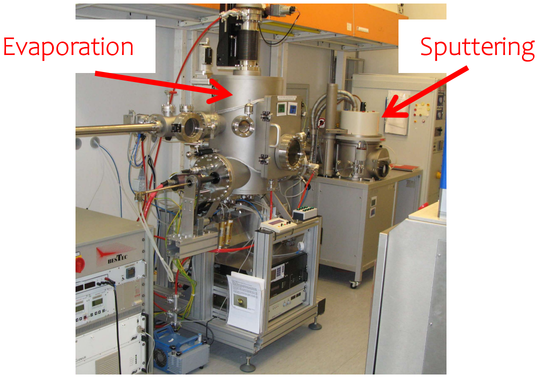
3. Reactive Ion Etching

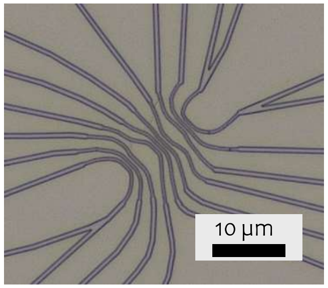




4. Deposition

5. Lift-Off



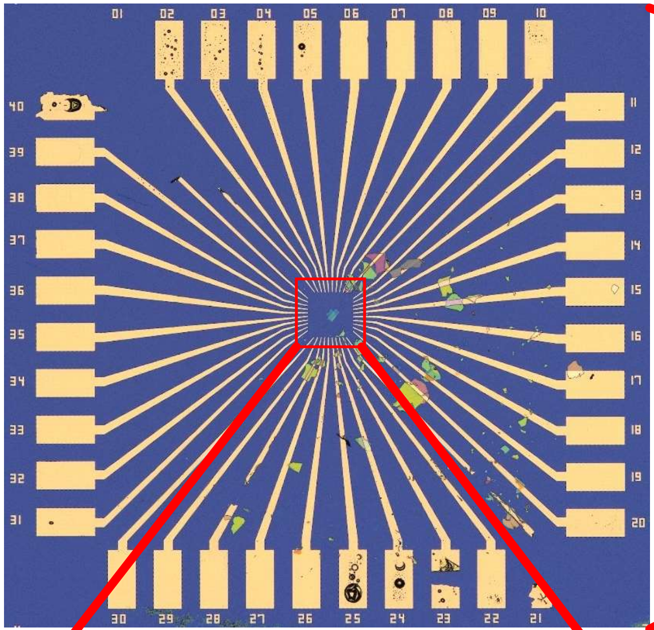


Typical key words for description of process flow

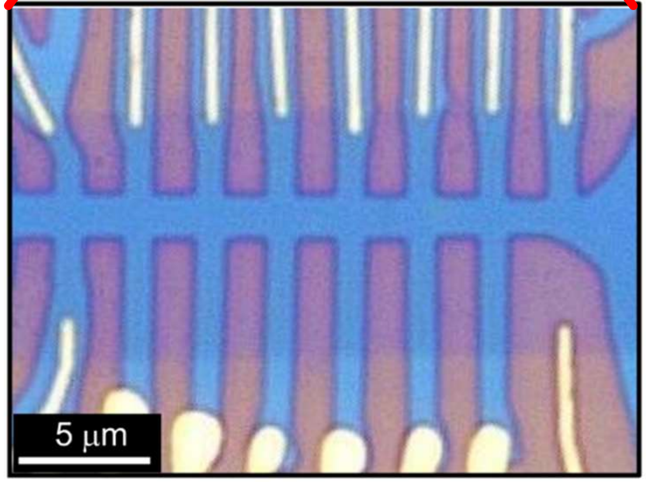
- Pretreatment, adhesion promotion, Surface chemistry: hydrophilic ⟶ hydrophobical (non-polar) by means of: using a primer, heating, oxygen-plasma
- Spin coating (defines the thickness of the PR layer depending on viscosity, speed)
- Softbake (evaporation of solvents)
- Exposure (photo-chemical activation)
- Post-Exposure-Bake, PEB
  (increasing degree of crosslinking; finishing the photo-reaction)
- Development (dissolve either exposed or non-exposed resist)
- Hardbake (better adhesion and chemical-mechanical stability)
- De-scumming (Etching of resist residues)

Backend processing – final device assembly

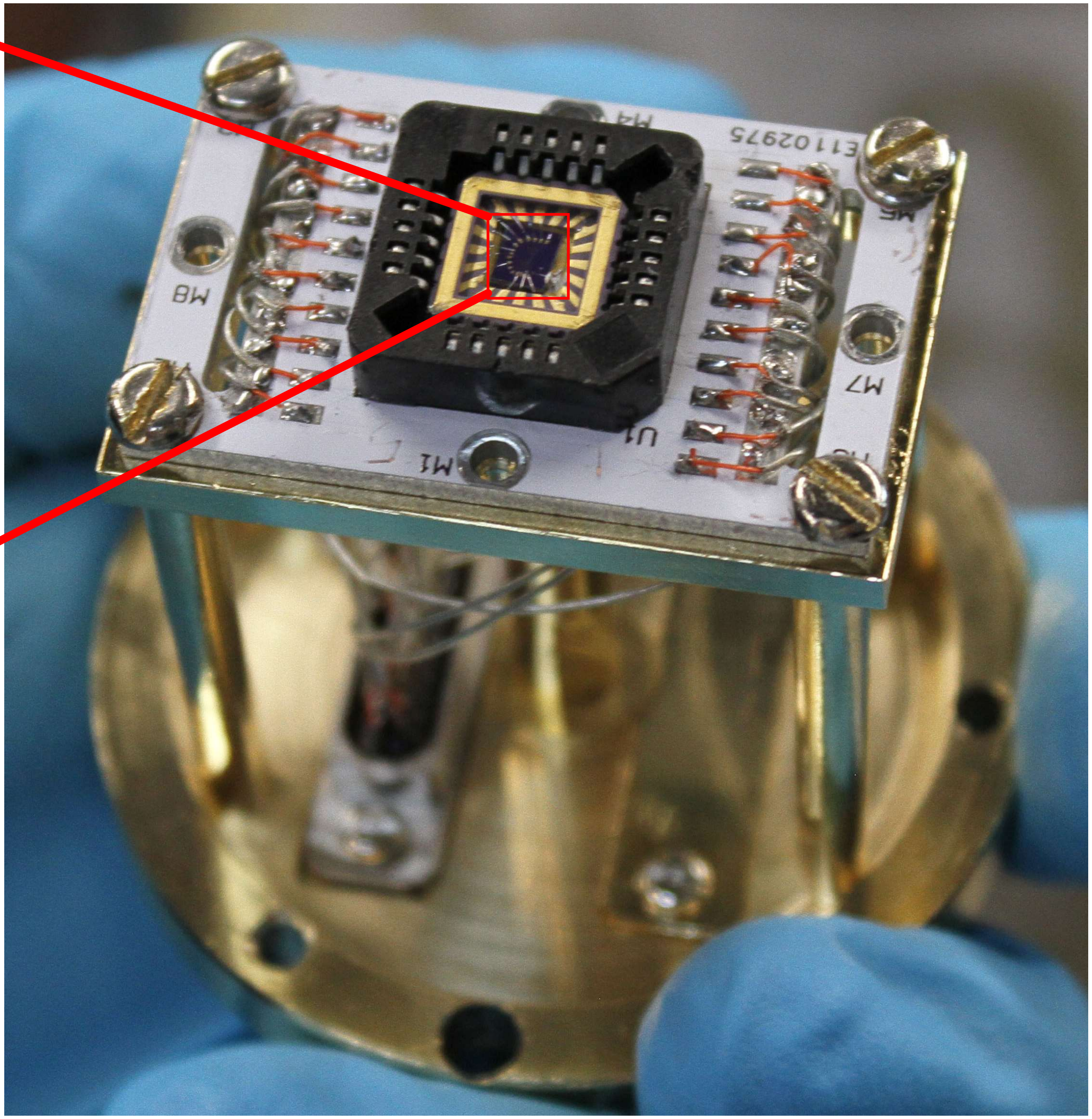
On-chip electrodes:



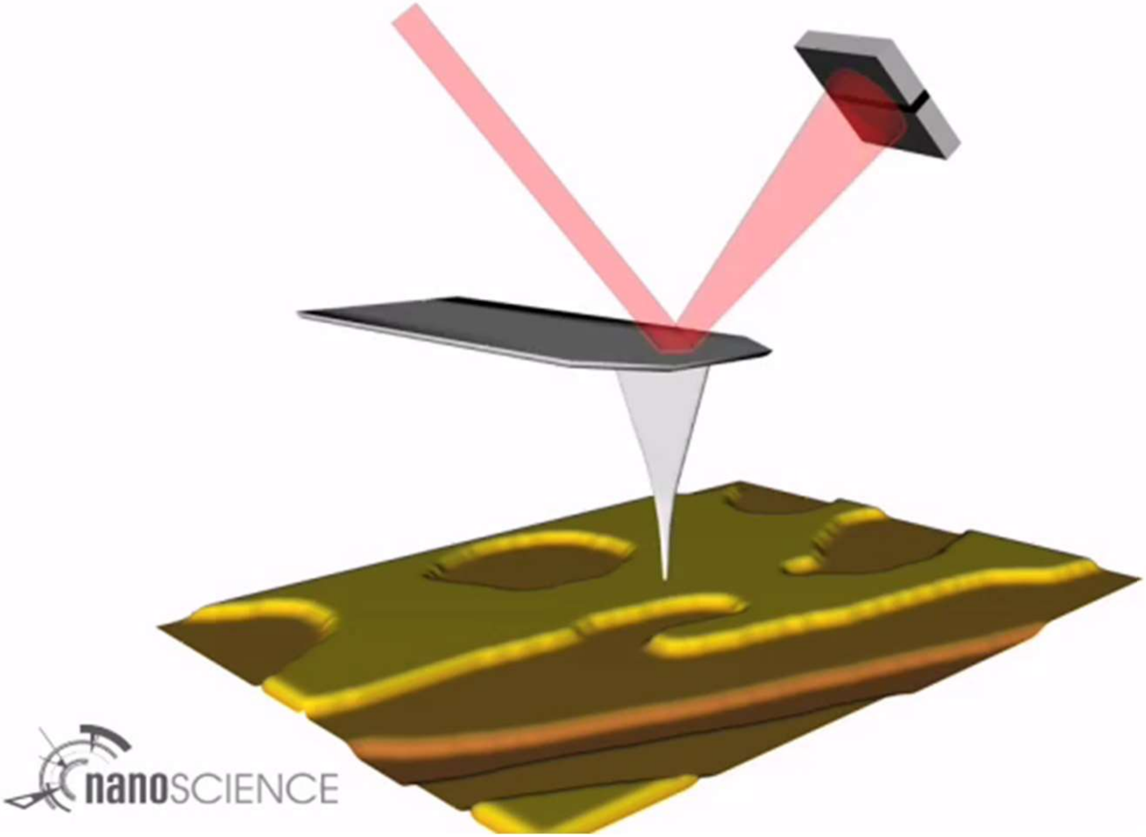
Graphene device:

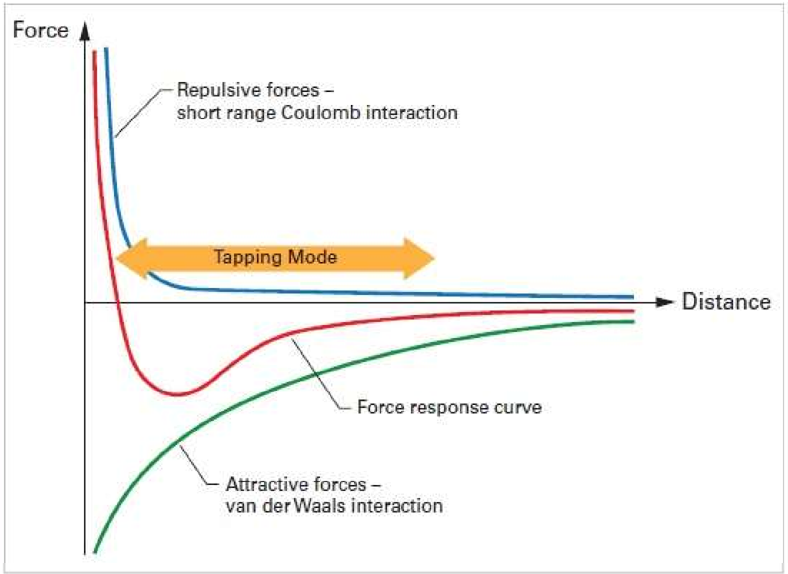


Wire bonded chip in carrier and insert:

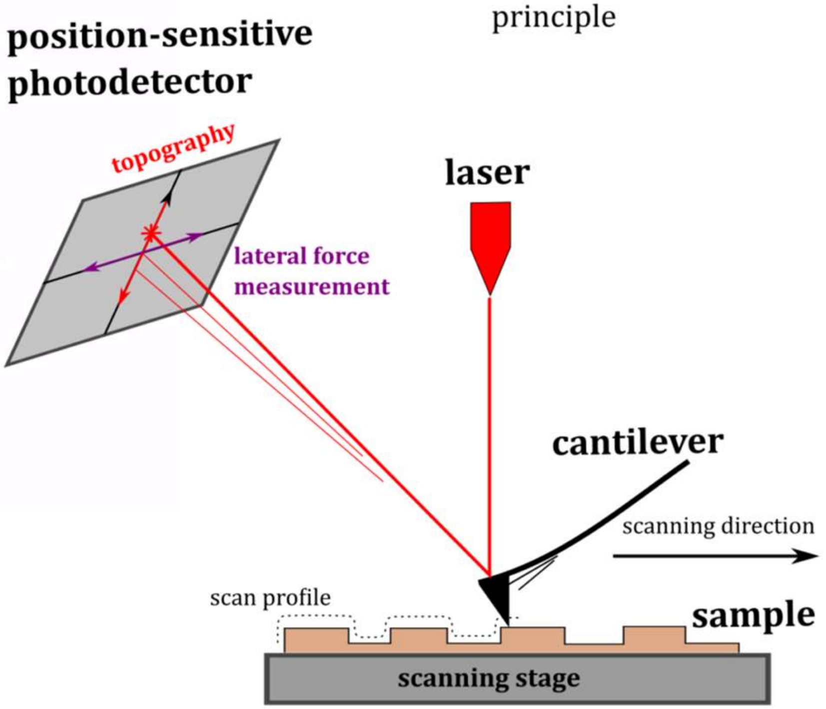


Atomic force microscopy





Atomic Force Microscope principle



Atomic force image of final graphene device