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DI RIPRESA E RESILIENZA

INFN

Coatings deposition

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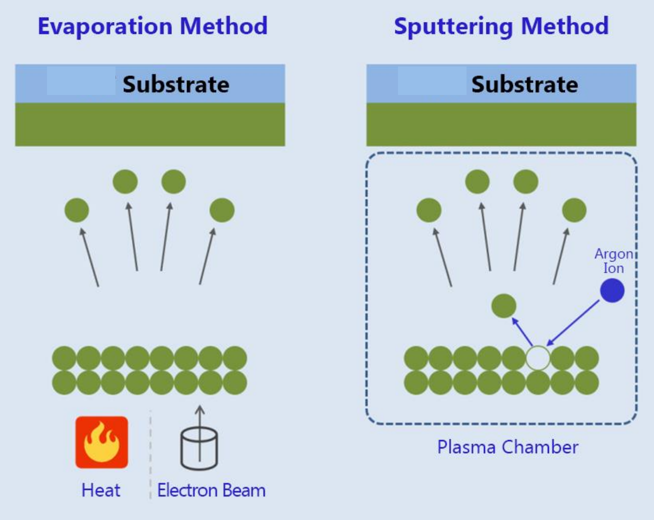
ETIC Technology School
Cagliari 12-13 dec. 2024

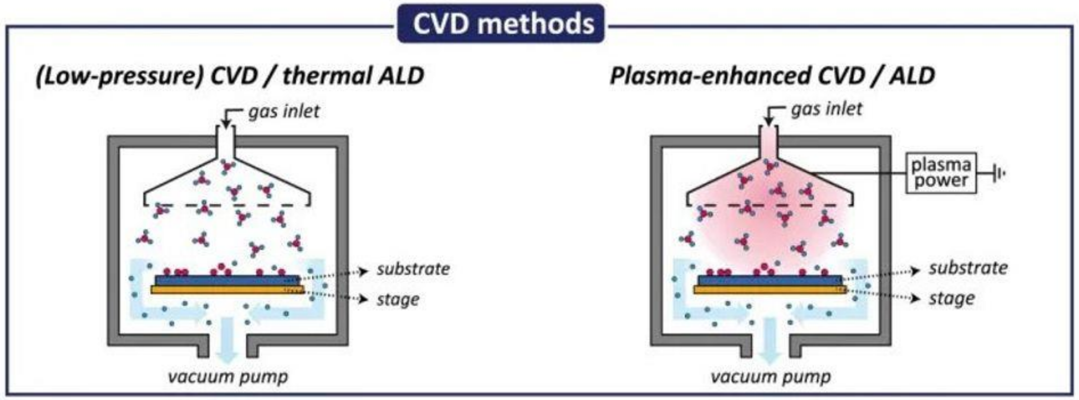
Vapor deposition

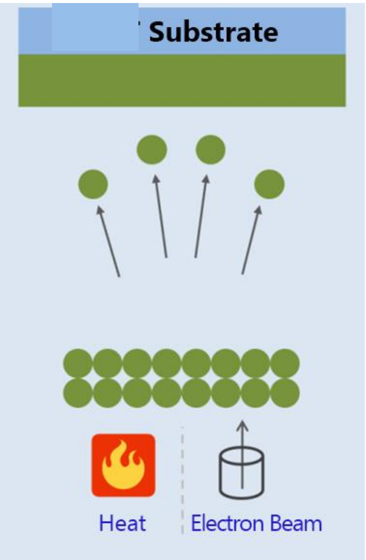
Vapor deposition describes any process in which a solid immersed in a vapor becomes larger in mass due to material transfer from the vapor onto the solid surface.

- if the vapor is created by physical means without a chemical reaction, the process is classified as **PVD**;
- if the material deposited is the product of a chemical reaction, the process is classified as **CVD**

PVD methods



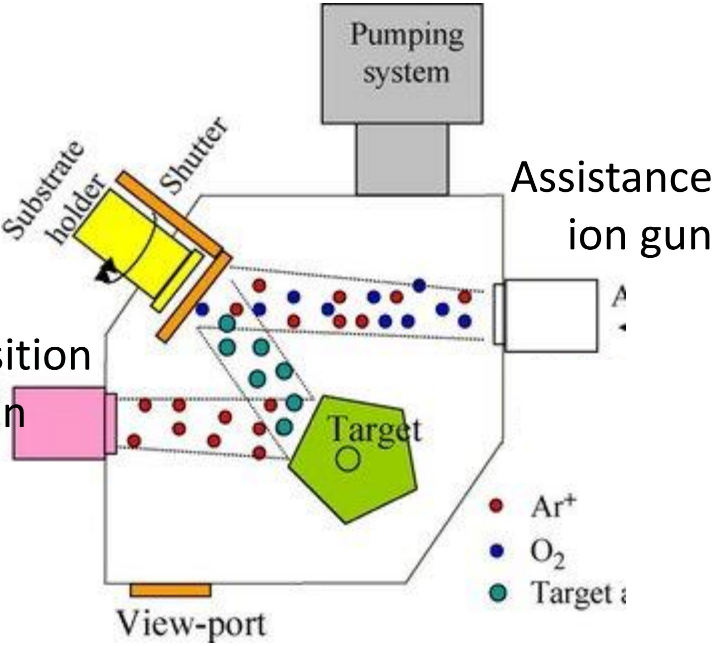




Transfer to vapor phase by heating in vacuum

- PVD
  - Evaporation
    - Heating
    - E-beam
  - Sputtering
    - DC, RF Diode
      - No magnetic confinement
    - DC, RF Magnetron
      - Efficiency magnetic confinement
    - IBS
      - Secondary beam

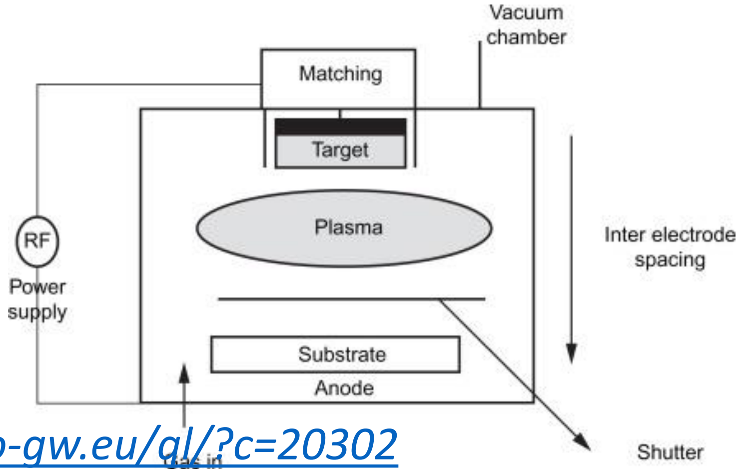
Transfer to vapor phase by plasma (ions bombardment)

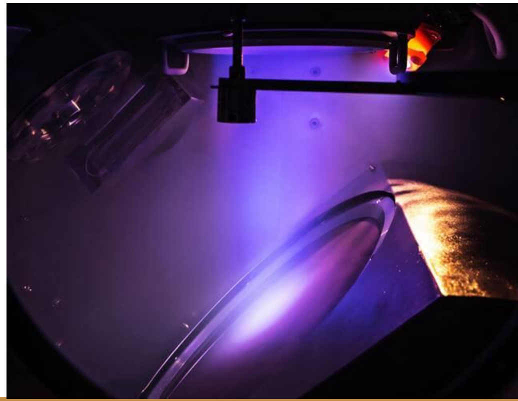


Assistance ion gun

Deposition ion gun

- Double e-beam 6 crucible
- IAD ion assistance source

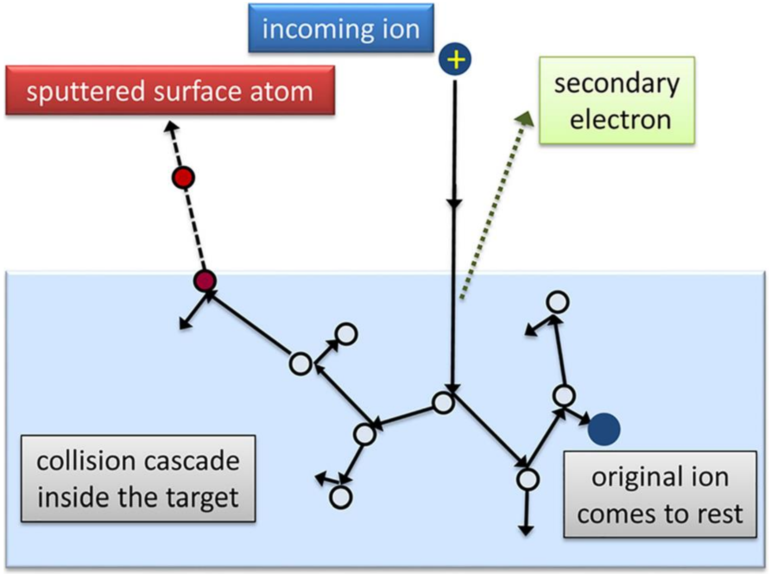


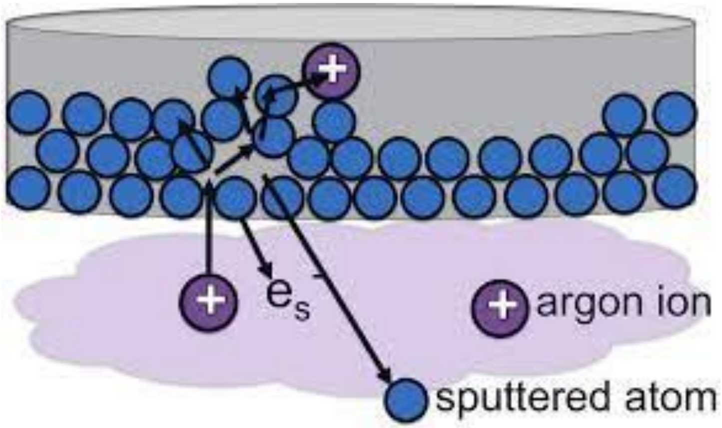


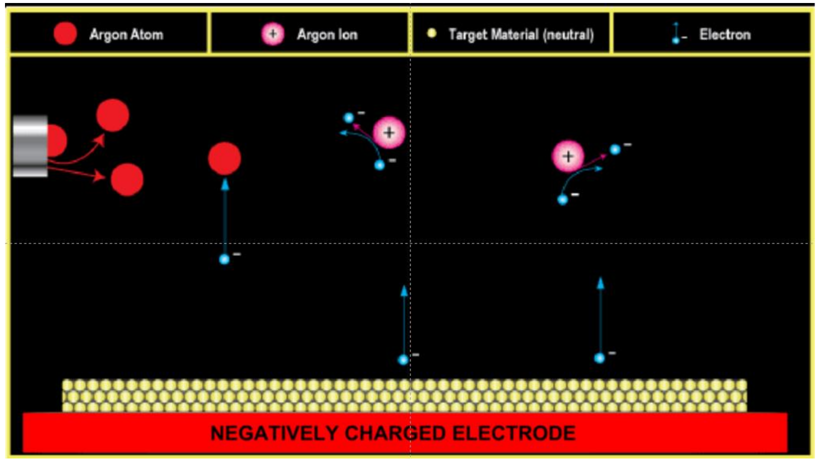
V.Granata VIR-0242A-24 https://tds.virgo-gw.eu/ql/?c=20302

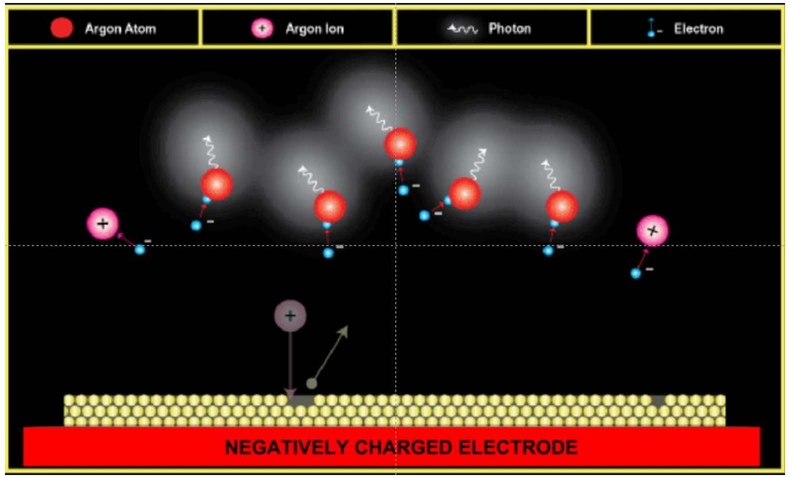
Sputtering

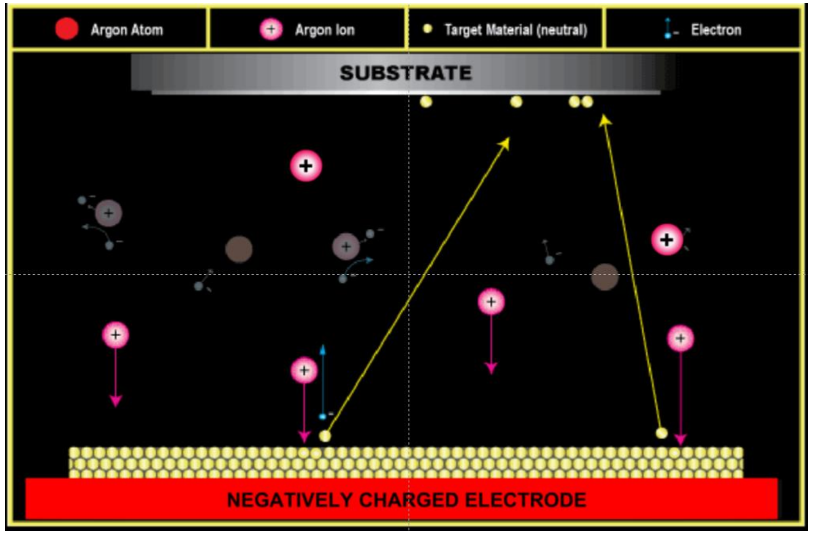
Sputtering is a phenomenon of the ejection of microscopic particles of a solid material from its surface when bombarded by energetic particles (plasma ions, energetic neutrals, accelerated particles)

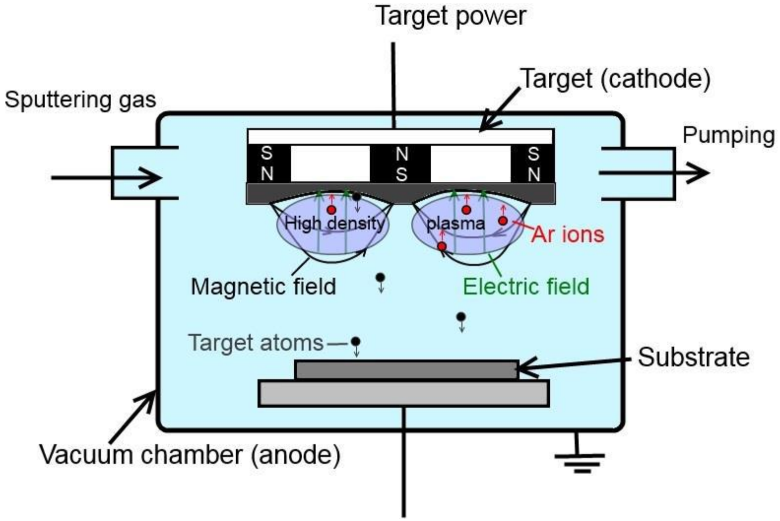




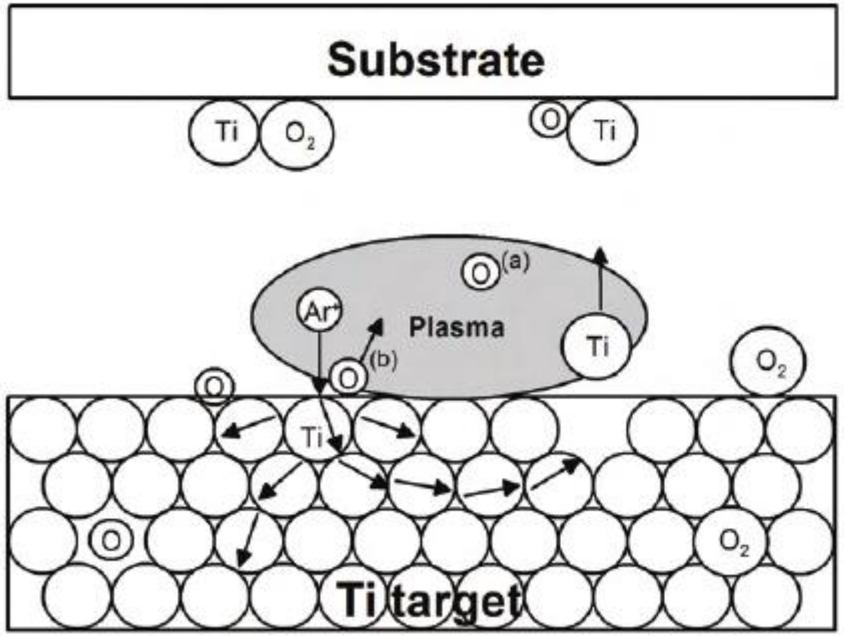

Argon Atom
Argon Ion
Target Material (neutral)
Electron
NEGATIVELY CHARGED ELECTRODE

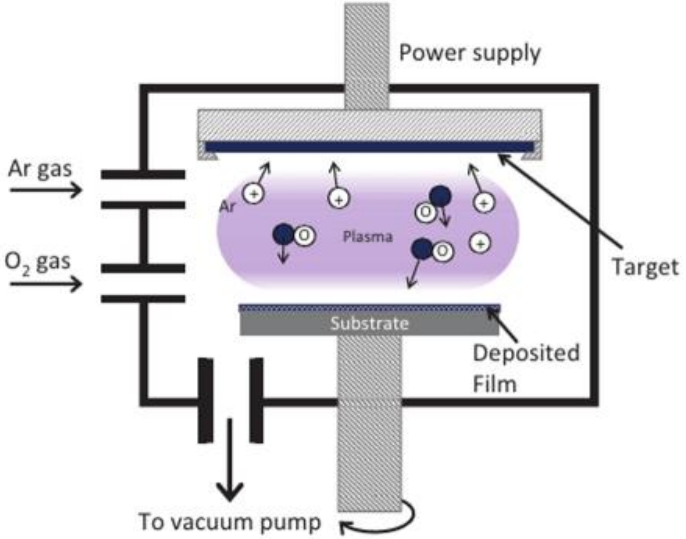

Argon Atom
Argon Ion
Photon
Electron
NEGATIVELY CHARGED ELECTRODE

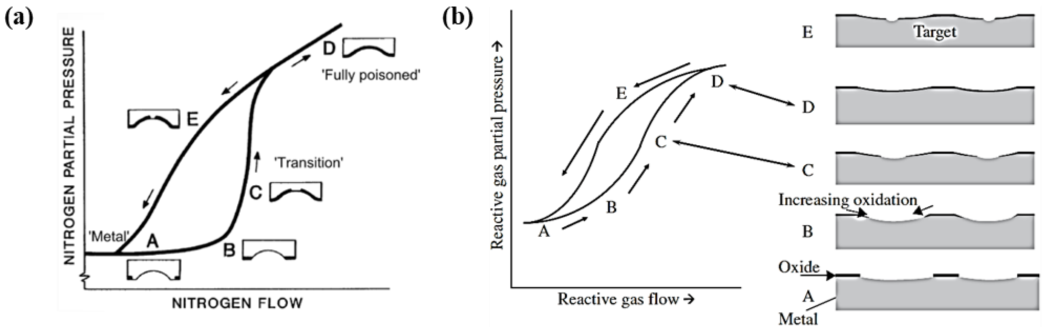

Argon Atom
Argon Ion
Target Material (neutral)
Electron
SUBSTRATE
NEGATIVELY CHARGED ELECTRODE


Target power
Target (cathode)
Sputtering gas
Pumping
S
N
N
S
S
N
High density
plasma
Ar ions
Magnetic field
Electric field
Target atoms
Substrate
Vacuum chamber (anode)

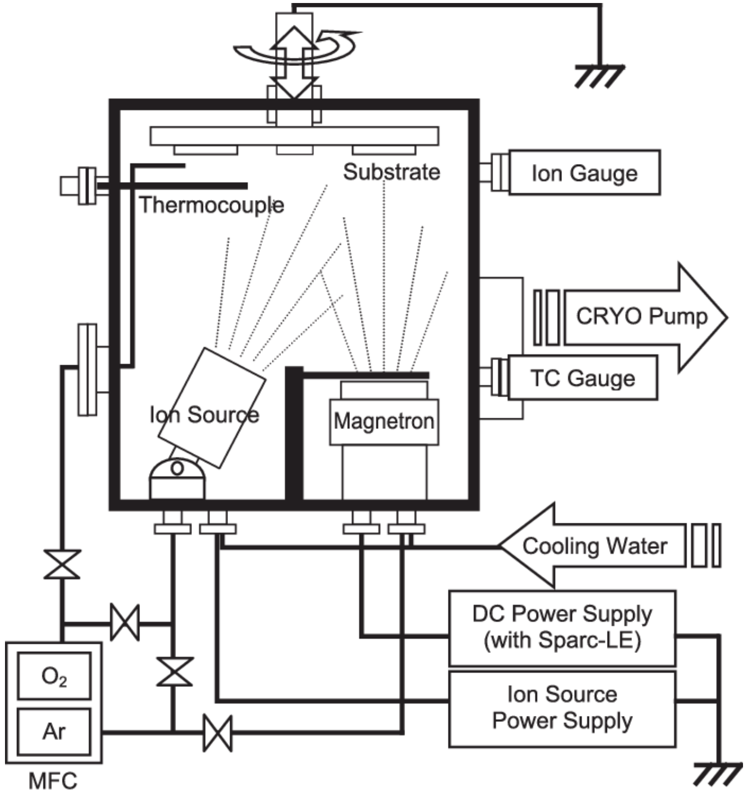
Reactive sputtering



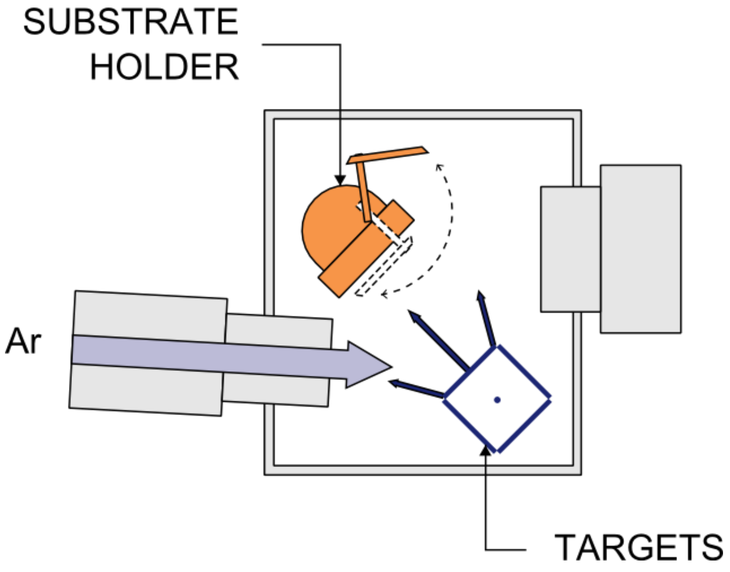




Sputtering setup



Example: components PVD desposition system



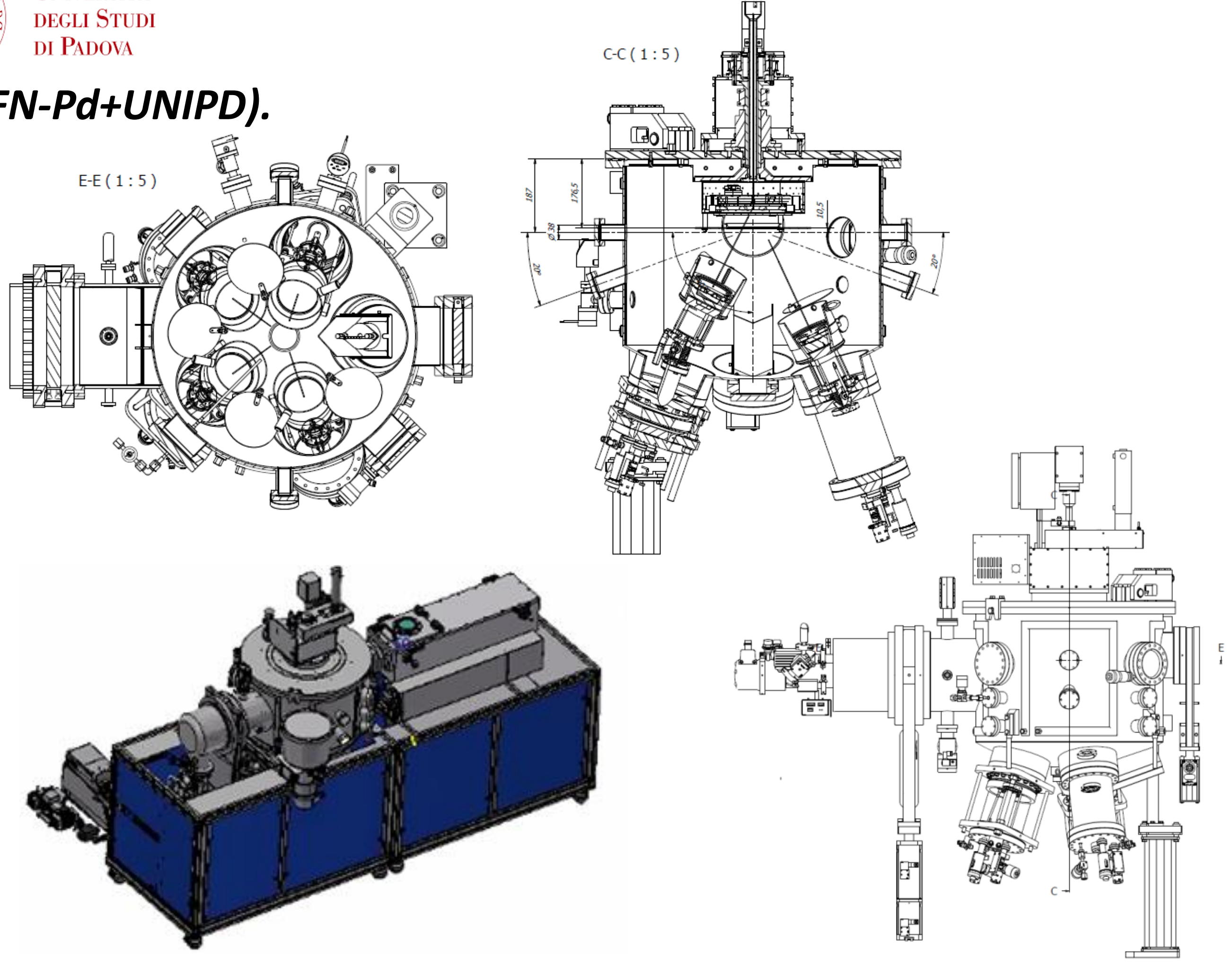
Example: IBS setup

MS setup example

INFN PADOVA
UNIVERSITÀ DEGLI STUDI DI PADOVA

Kenosistec customized cluster MS system (lab. CoMET, INFN-Pd+UNIPD).

- 4 4’’ **magnetron sources** for co-deposition (for now 1 pulsed DC, 2 RF power supplies)
- **Assistance ion source** eH200HC (non yet, but additional funds were requested)
- High **vacuum** (< 6×10^{-8} mbar)
- 5 **gas lines** (Ar, O2, N2, ..) each can be used near magnetron or/and near substrate
- **Uniformity** better than 1% on 100 mm diameter
- **Substrates** up to 125 mm diameter, up to 20 mm thickness
- Rotated **substrate holder**, heating up to 700°C
- Predisposition for RF susbtrate **bias**
- Predisposition for several **in-situ diagnostics**: RGA, ellipsometry, *energy-mass spectrometry, stress/curvature measurement, optical(photon) emission monitor, ..*



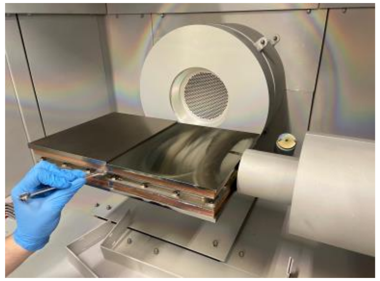
IBS setup example

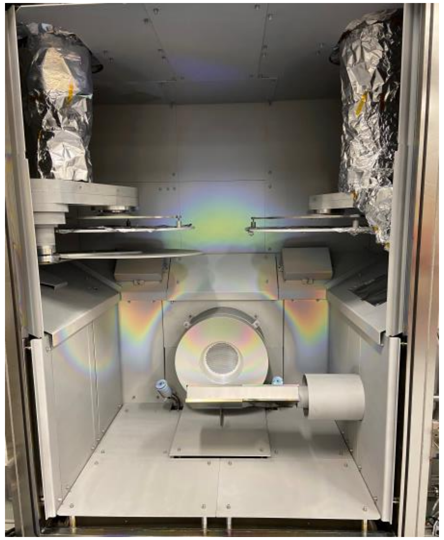
EPOC NAVIGATOR system

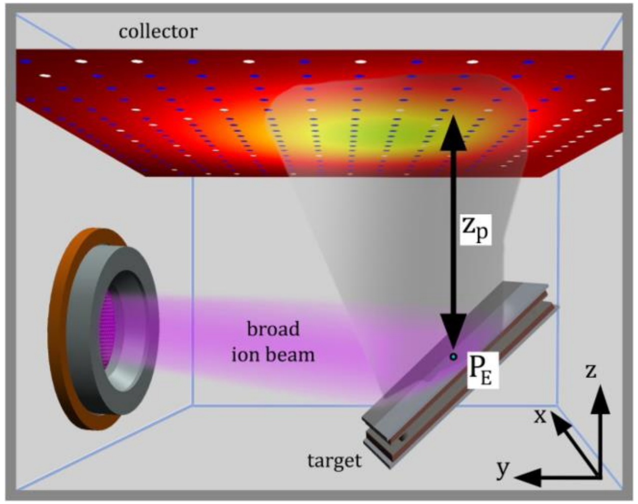
University of Strathclyde Glasgow

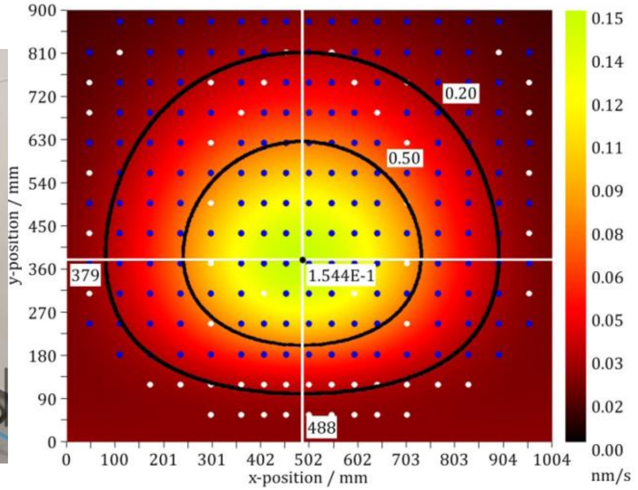
- <1% uniformity in 62 cm diameter without masks
- One main ion source and (coming next) one assist source
- Two substrate holders
- Process is monitored by BBOM
- 2 IR heaters
- Base pressure ~ 10^{-8} mbar
- 4 targets (20 cm x 25 cm) – metallic
- Mixed materials achieved easily by horizontal translation of target stage
- Optical designs by Optilayer, process allows for on-the-fly redesigning

https://dcc.ligo.org/LIGO-G2300222
https://epoc.scot/









https://doi.org/10.1364/AO.389883

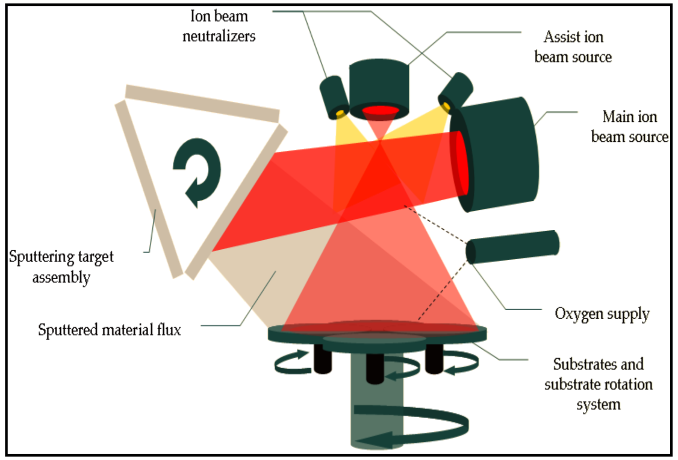
IBS setup example

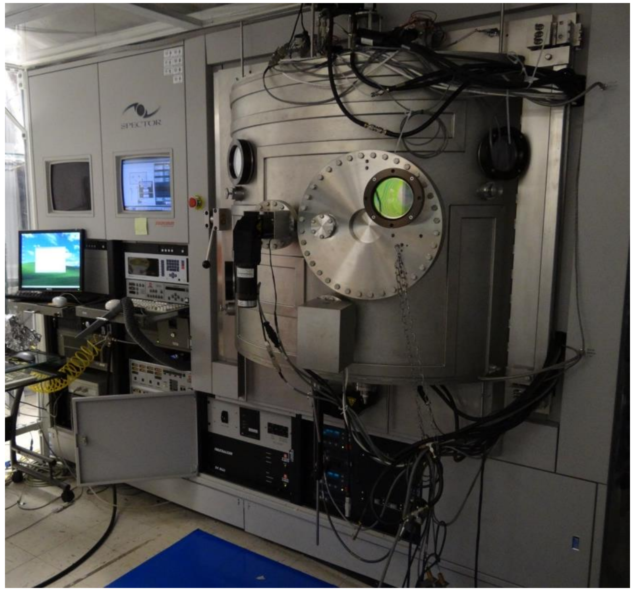
Colorado State University

Veeco SPECTOR deposition system

- Main source: Ar+: ~ 600eV – 1250 eV, up to 600 mA
- Assist source: Ar+, O2+, N+ or their combination; ~ 600eV – 1250 eV, up to 600 mA
- Base pressure: 10-6 Torr
- Primary ions impact the target at ~ 45 deg - Sputtered material leaves the target at ~45 deg
- Planetary rotates the substrates through the plume during film growth
- Oxygen is provided when sputtering a metal target
- Three targets available during a coating run. One used at a time

https://dcc.ligo.org/LIGO-G1702195



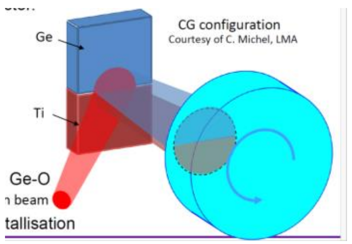


IBS setup example

LMA LABORATOIRE MATÉRIAUX AVANCÉS

LMA Grand Coater deposition system

- Size: 2.2 x 2.2 x 2 m^3
- Pumping system: 2 dry pumps 4 cryopumps
- Pressure: 1.10-7 mbar in 3 hours
- 16 cm RF Ion source + RF Neutralizer
- Thickness monitoring: 4 XTC
    - 2 multi targets:
    - 2 substrate holder:
- single rotation/planetary motion
- corrective coating robot
- deposition speed: 0.1 à 3 Å/s
- capability: up to Ø 1m in single rotation



CG configuration
Courtesy of C. Michel, LMA

GC view from rear side

GC view from clean room ISO 3

photos: C. Fresillon – photothèque CNRS / E. Le Roux / LMA

C. Michel OCLA Symposium 2017

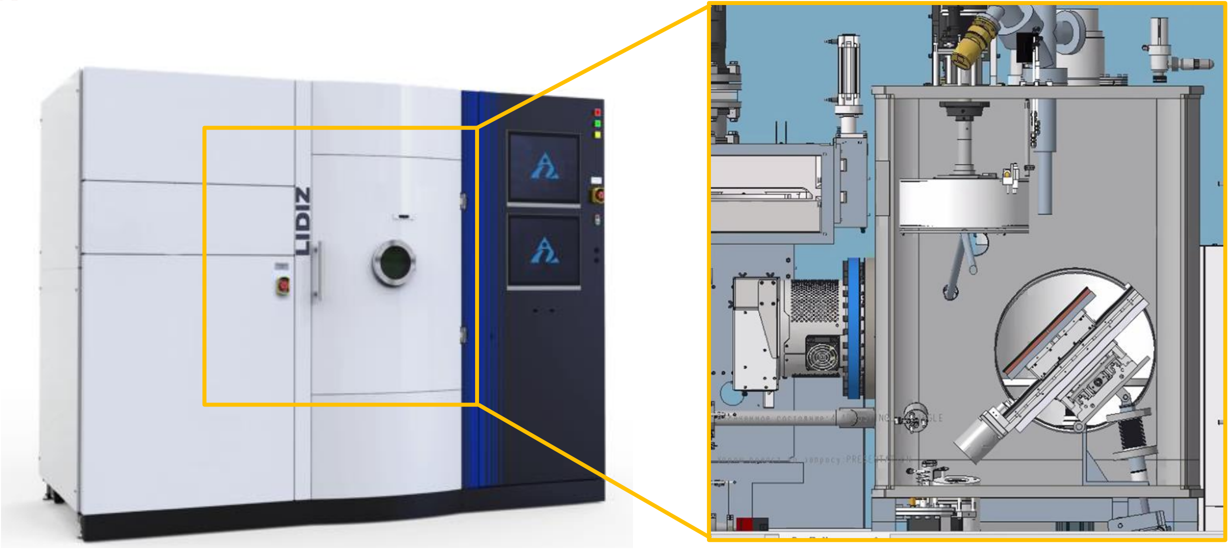
[G.Cagnoli LVK 2023 https://dcc.ligo.org/LIGO-G2300658](https://dcc.ligo.org/LIGO-G2300658)

IBS setup example

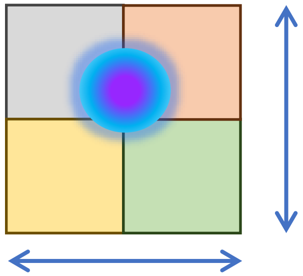
Università degli Studi di Padova

I-Photonics LIDZ customized IBS system (lab. CoMET, UNIPD).

- Multimaterial deposition with compositional control <u>up to 4 different **targets** (each can be multicomposition).</u>
- **Primary ion source** 400 mA 2000 eV,
- **Assistance ion source** 2A, 450eV
- High **vacuum** ($< 1 \times 10^{-7}$ mbar)
- **Substrates** up to 125mm in diameter, several mm thickness
- **Uniformity** better than 0.5% on 100 mm diameter substrates
- Rotating **sample holder**, heated up to 700°C, 10cm ΔZ position
- **In-situ diagnostics**: ellipsometer, RGA, *mass-energy spectrometer,* optical thickness monitor



Target movements for composition and angle control



Deposition systems are very different

I would even say that each setup is unique.

To compare results or scale up the process you need to take into account all process parameters

PVD process parameters include, but are not limited to

Deposition system and geometry

- Base pressure (base vacuum level)
- Additional impurities trapping methods
- Deposition pressure
- Deposition geometry (arriving particles incidence angle, path length, or target-substrate distance)
- System geometry (position reactive gas inlet), horizontal-vertical, up-down or down-up, etc.)
- Magnetic field involved
- Masks

gasses

- Inert gas flow
- Reactive gas flow

materials

- Used materials, microstructure, purities (gasses, targets, substrates)

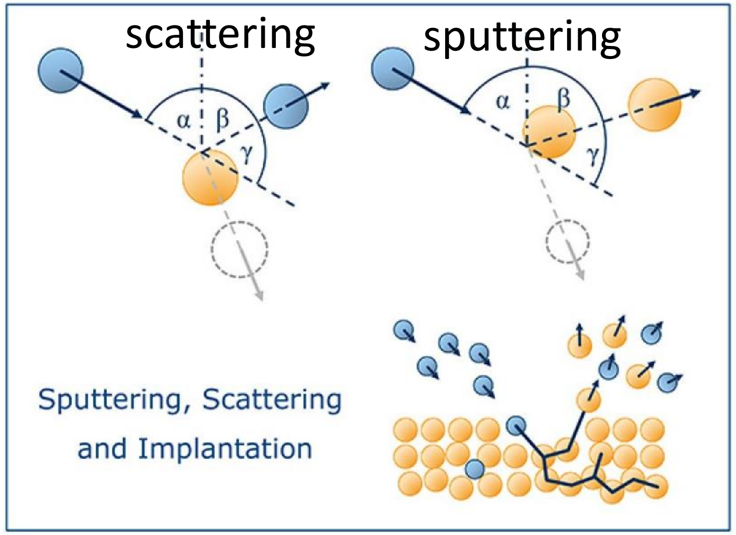
source

- Type of ion source or evaporation source
- Source parameters: Power, Voltage, Current and others (depends on type of source) or heating/e-beam parameters on an evaporator, neutralizer parameters for IBS
- Any movements, rotations available on target
- Any purification procedures: system backing, plasma etching, pre-sputtering, etc.

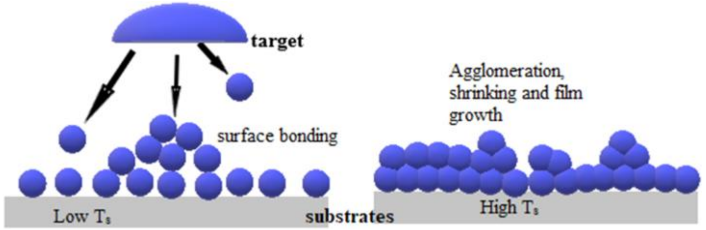
substrate

- Substrate temperature (heating, cooling, floating)
- Substrate bias: DC (positive, negative), RF
- Substrate positioning
- An ion beam assistance on the substrate and it's parameters

on the target & substrate



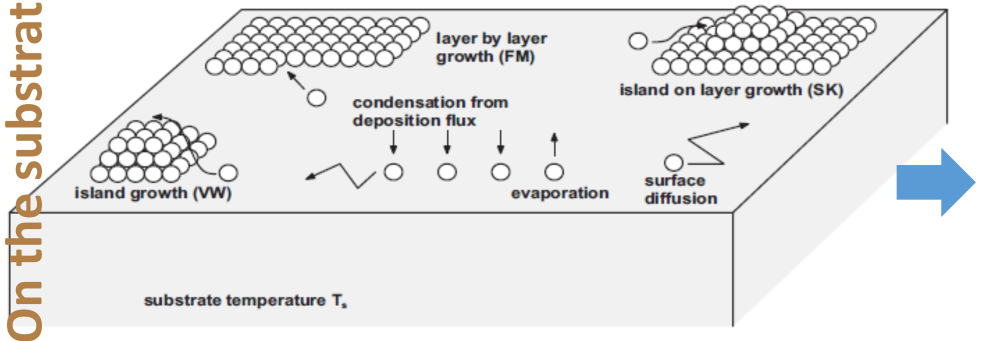
Process parameters → Thin film properties

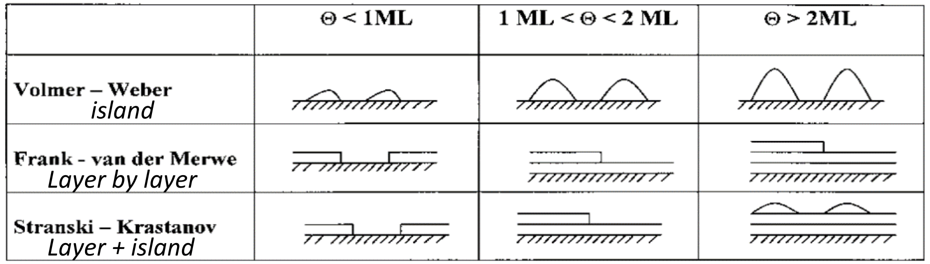


Under ideal growth conditions, the growth mode depends on:

- the materials involved,
- the temperature of the substrate,
- the degree of supersaturation of the vapor(≈deposition velocity).

On the substrate



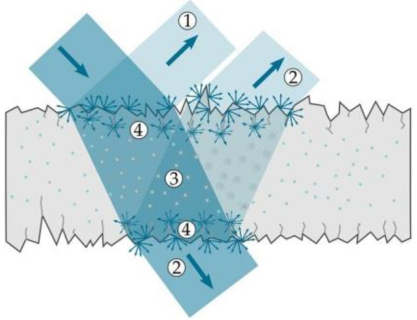


	$\Theta < 1ML$	$1\ ML < \Theta < 2\ ML$	$\Theta > 2ML$
Volmer – Weber *island*			
Frank - van der Merwe *Layer by layer*			
Stranski – Krastanov *Layer + island*			

interaction between atoms of the film is stronger than with substrate atoms

the interaction between substrate-atoms and film-atoms is stronger than between the atoms of the film

Process-dependent coating properties for GW interferometer mirrors (Part I)



- **Density** → optical & mechanical properties,
- **Roughness** → scattering
- **Adherence** → scattering, mechanical properties, thermal losses
- **Stress** → mechanical properties, density, birefringence, adherence

All these properties are energy-dependent

Energy in game:

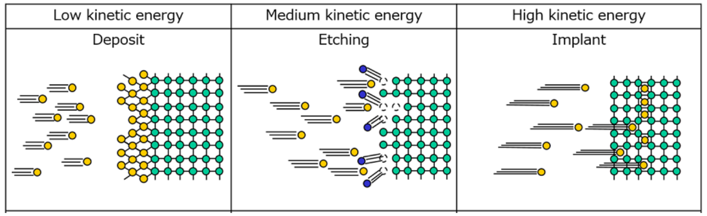
☐ kinetic energy of arriving particles
☐ thermal energy of substrate heating

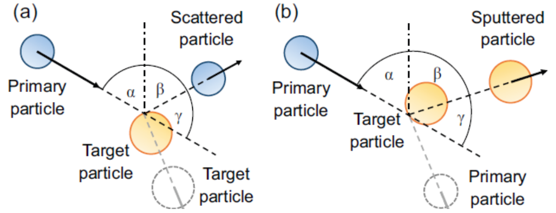
Process parameters → Particle properties → Thin film properties

Particles bombarding thin film
Adatom mobility

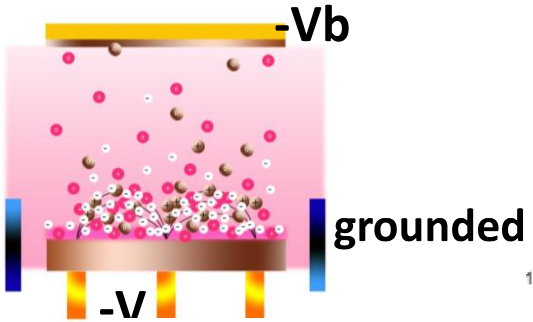
Methods to play with the energy

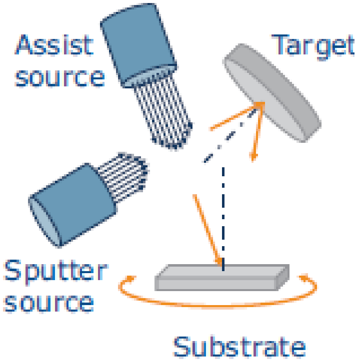
- ☐ **kinetic energy of arriving particles**
  (Particles bombarding thin film)
- ☐ **thermal energy of substrate heating**
  (Adatom mobility)

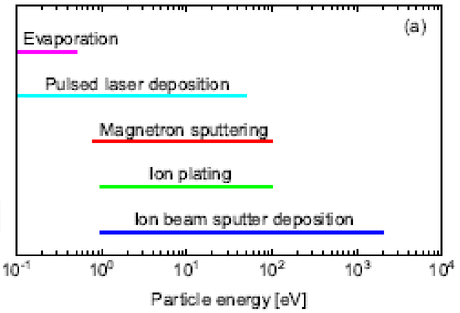


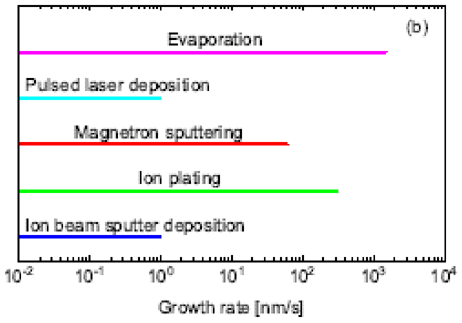


- Sputtering pressure (MS, IBS)
- Substrate bias (MS, IBS)
- Ion gun assistance (evaporation, MS, IBS)
- Substrate heating/cooling (evaporation, MS, IBS)
- Sputtering gas type-projectile (MS, IBS)
- Angle of incidence of projectiles (MS, IBS)
- Depsotion rate (MS; IBS; evaporation)









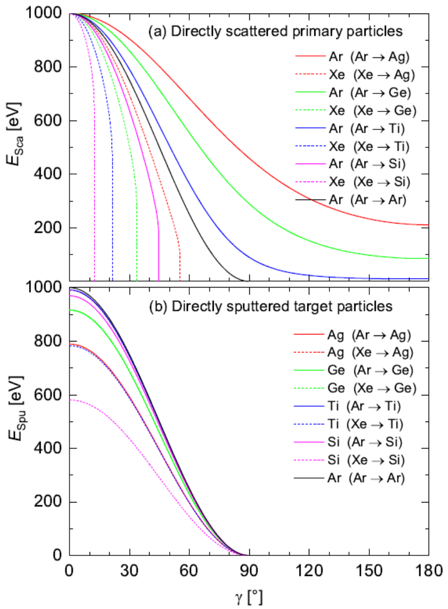


FIG. 11: Calculated energy of directly scattered primary particles [$E_{\rm Sca}$, panel (a), Eq. 11] and directly sputtered target particles [$E_{\rm Spu}$, panel (b), Eq. 12] versus scattering angle γ for interaction of primary particles Ar (solid lines) or Xe (dotted lines) with target particles Si, Ti, Ge, or Ag. The energy of the primary particle is assumed to be $E_{\rm Ion} = 1000\,\text{eV}$.

https://doi.org/10.1063/1.5054046.

Structure Zone Models (SZM): multiple parameters influence

Process parameters → Thin film microstructure

Structure Zone Models (SZM) represent relations PVD process parameters vs microstructure

The idea is to **reduce all possible** deposition **parameters** to **as few as possible** and illustrate their influence film structure
Usually, it is realized in terms of ENERGY

T^* - corresponds to potential energy
E^* - corresponds to kinetic energy
t^* - net thickness

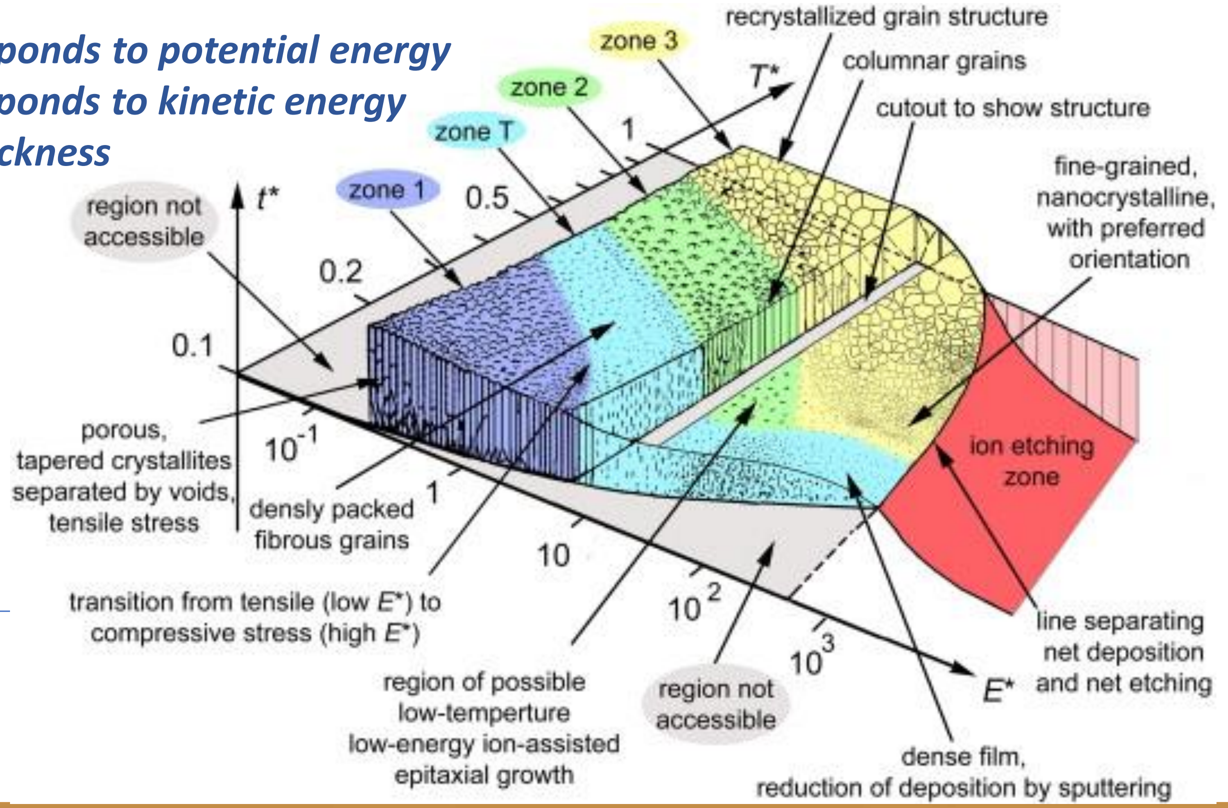
Homologous temperature

$$T_h = \frac{T}{T_m} \qquad T_{pot} = \frac{E_{pot}}{k \cdot N_{moved}} \qquad E_{pot} = E_c + (E_i - \phi)$$

$$T^* = T_h + T_{pot}$$

$$E^* = E_0 + Q \cdot e \cdot V_{sheath}$$

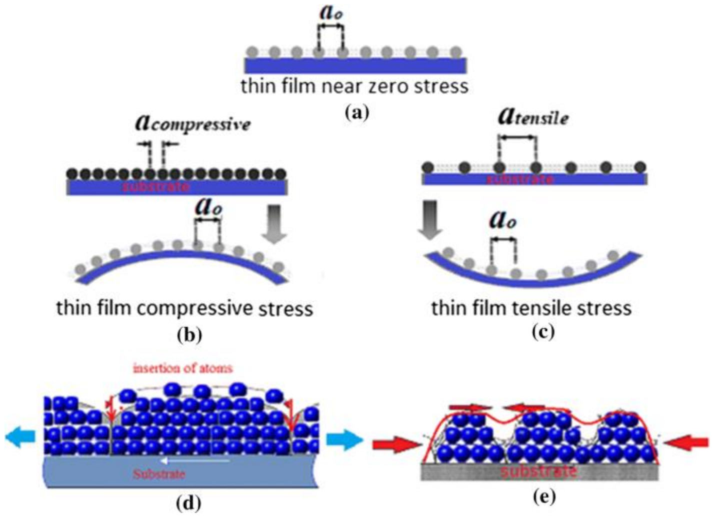
$$T^* = T_h + \frac{\frac{1}{k}\sum_\alpha \frac{E_{pot,\alpha} \cdot J_\alpha}{N_{moved,\alpha}}}{\sum_\alpha J_\alpha} \qquad E^* = \frac{\sum_\alpha \frac{E_{kin,\alpha} \cdot m_\alpha \cdot J_\alpha}{E_c \cdot m_s}}{\sum_\alpha J_\alpha}$$

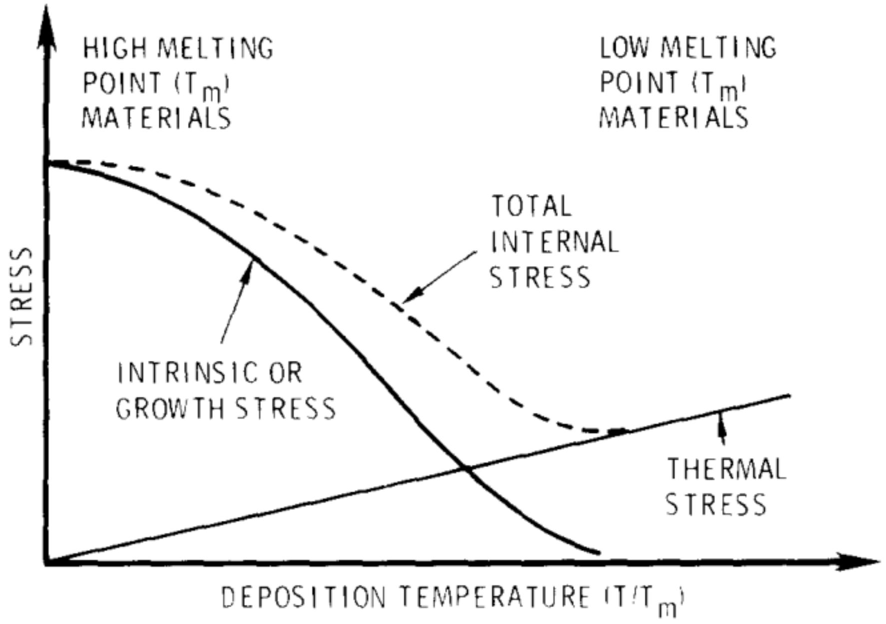


B. A. Movchan and A. V. Demchishin (1969). *Phys. Met. Metallogr. 28: 83–90.*
J.A. Thornton (1974) *J. Vac. Sci. Tech. 11: 666–670.*
R. Messier, A. P. Giri, and R. A. Roy *J. Vac. Sci. Tech. A 2, 500* ***(1984)***
A. Andre *" Thin Solid Films 518(15), 4087–4090* ***(2010)***

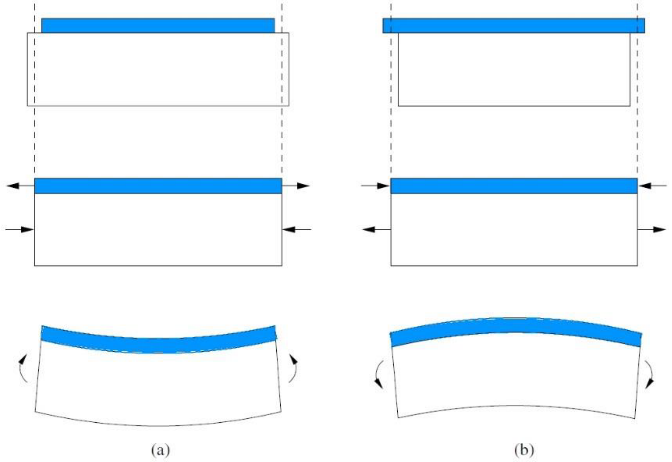
Stress in thin films.

Intrinsic stress
Growth stress





Extrinsic stress
Example: thermal mismatch

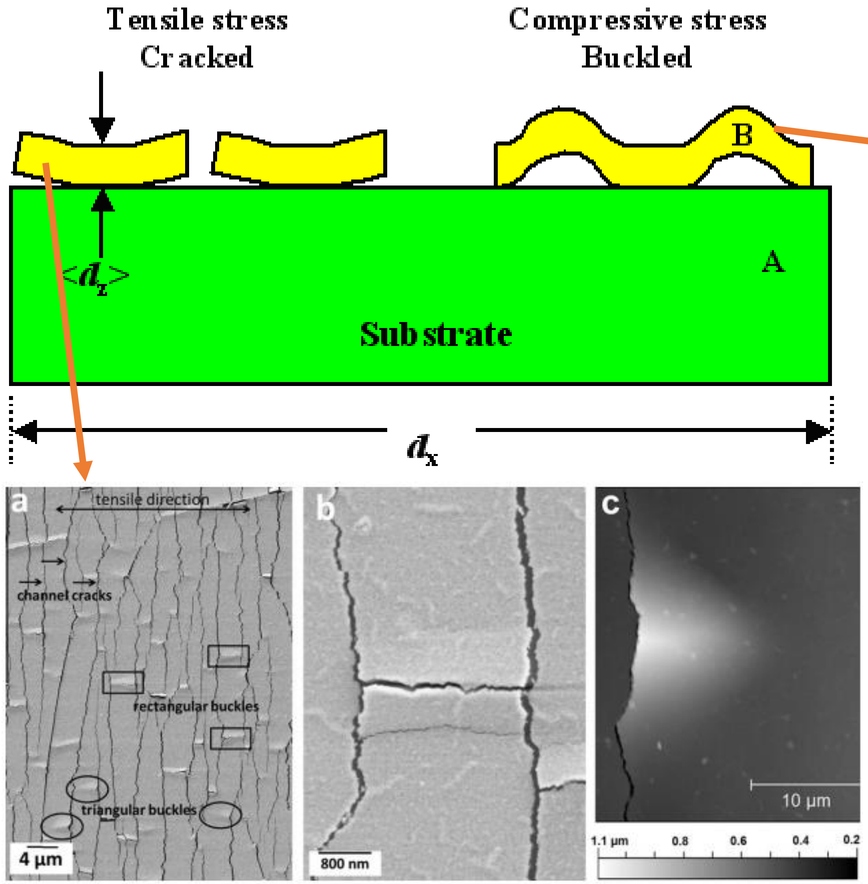


Residual stress (after deposition) = intrinsic stress + extrinsic stress

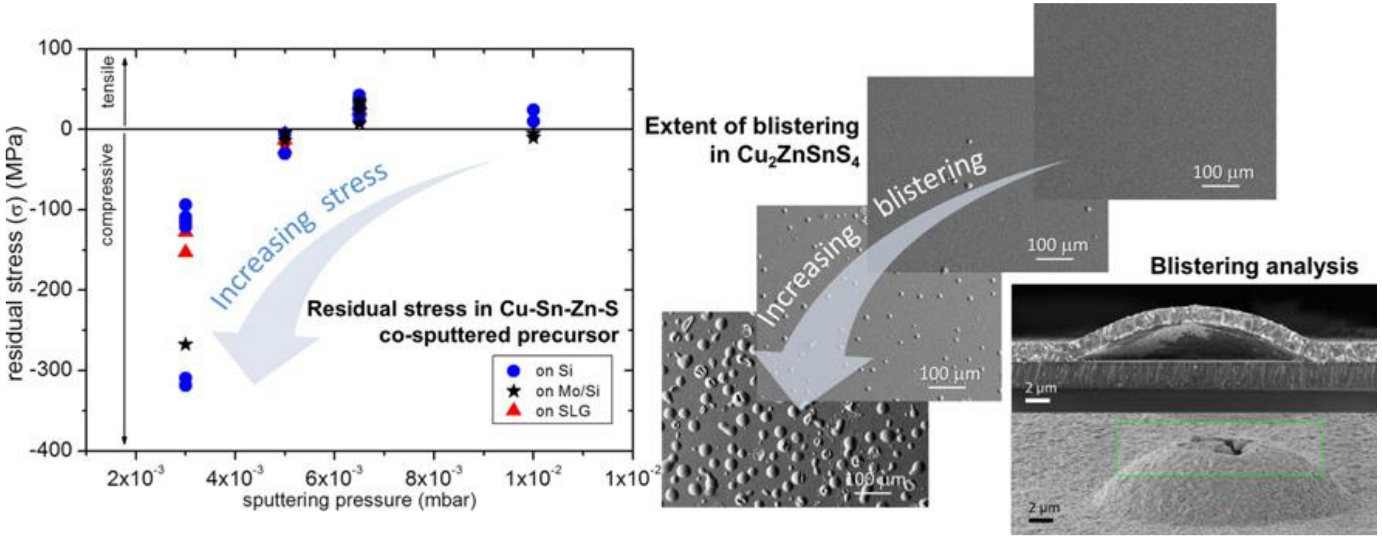
$$\sigma_{f,mismatch} = \frac{E_f}{1-\nu_f}(\alpha_f - \alpha_s) \cdot \Delta T$$

J. Laconte, et al. Micromachined Thin-Film Sensors for SOI-CMOS Co-Integration 2006

Film failure due to residual stress in thin films



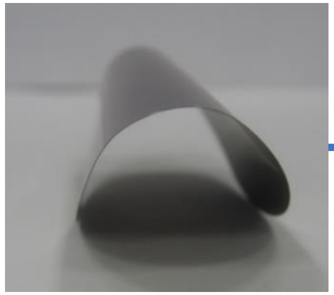
https://doi.org/10.1016/j.actamat.2013.01.014



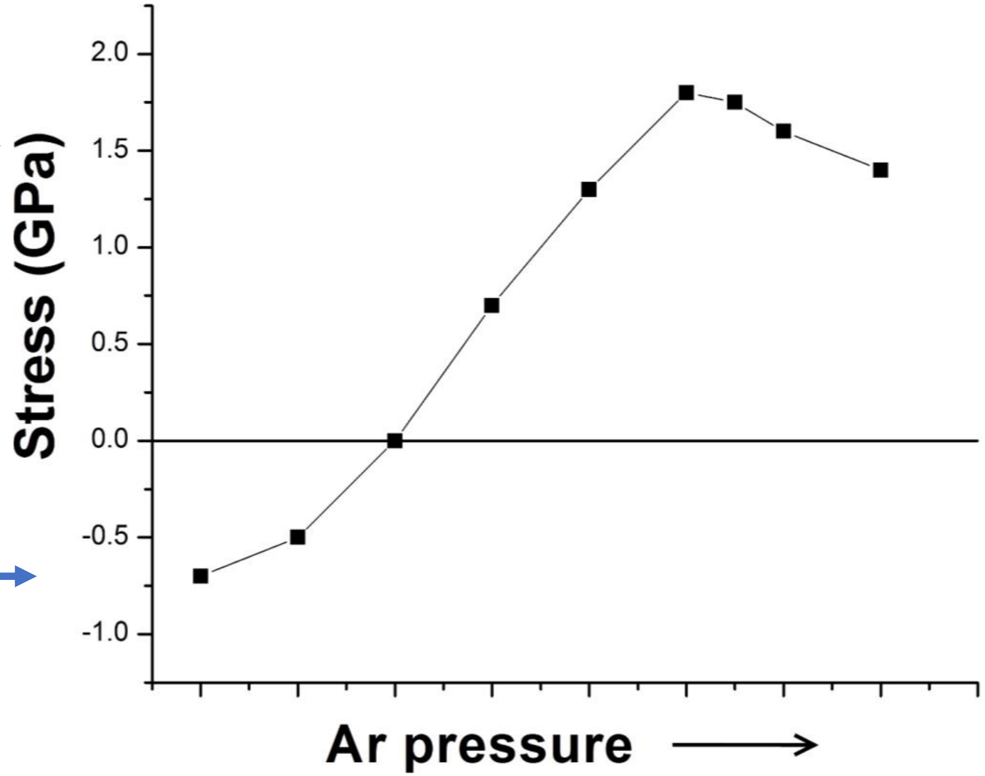
http://dx.doi.org/10.1016/j.matdes.2016.07.019

Optimizing sputtering pressure for minimal residual stress in thin films

Tensile stress

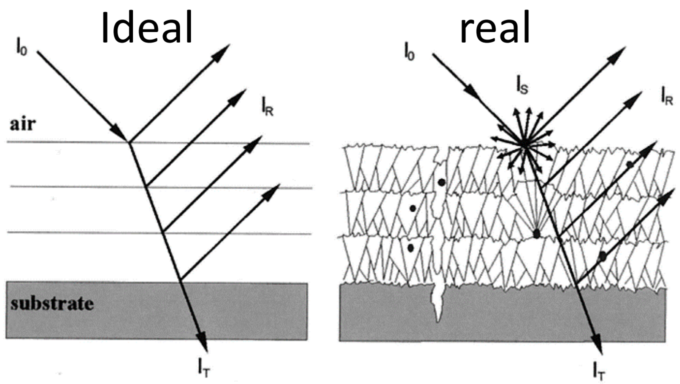


Compressive stress

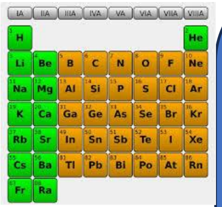


Process-dependent coating properties for GW interferometer mirrors (Part II)

- **Stoichiometry** → optical & mechanical properties
- **Amorphous microstructure**→ scattering
- **Transparency, minimal porosity, no pinholes** → Internal scattering
- **Minimal chemical impurities** → optical absorption

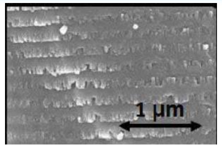


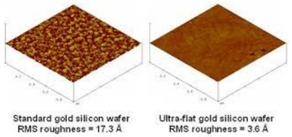
! **To keep under control:** substrate preparation, system cleaning, defects associated with macroparticles, different plasma etching methods



Elemental composition

- Rutherford Backscattering Spectroscopy (RBS)
- X-ray Photoelectron Spectroscopy (XPS)
- Energy Dispersive X-ray spectroscopy (EDX)
- …





Standard gold silicon wafer RMS roughness = 17.3 Å

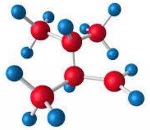
Ultra-flat gold silicon wafer RMS roughness = 3.6 Å

Morphology

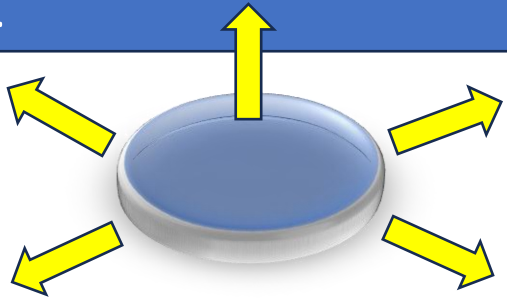
- Optical microscopy
- Scanning Electron Microscopy
- Stress measurements
- Atomic Force Microscopy
- …

Structure

- X-Ray Diffraction (XRD)
- X-Ray Reflectivity (XRR)
- Syncrotron radiation techniques (EXAFS, total X-ray scattering, anelastic scattering etc.)
- Raman spectroscopy
- Infrared spectroscopy (FTIR)
- …

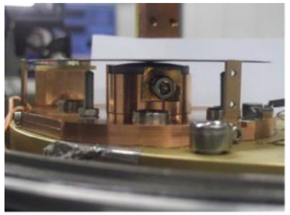


First order properties



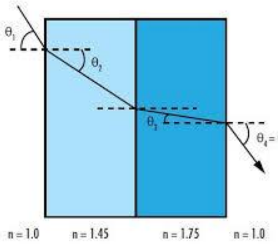
Mechanical properties & thermal noise

- Gentle Nodal Suspension system (GENS)
- Mech. Losses on cantilever
- Quadrature phase differential interferometer (QPDI)
- Direct measurement in optical cavity



Optical Properties

- Ellipsometry
- Optical Absorption Spectroscopy
- Optical Scattering
- …



Functional performance

Thank you for your attention!