SEM sample preparation

6th CEMM workshop

Maja Koblar, Sc. Eng. Physics

Outline

- Before SEM characterization
- Preparation of:
  - bulk material – hard and soft
  - powders
- Mounting: holders and adhesives
- Coating: sputtering and evaporation
- Thin film growth

Before SEM characterization

Before you start the preparation?

- Is the sample vacuum compatible?
- Interested in morfology (shape of the particles or the microstructure, grain size, grain shape,..)
- Do you want to know the chemistry?
- Discuss different options of preparation…

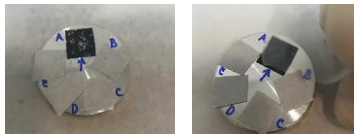
Figure removed for copyright reasons.

Sample as small as possible

Less is more!

Figure removed for copyright reasons.

Image: providr.com



The same goes for SEM samples

Figure removed for copyright reasons.

Image: more.com

Why?

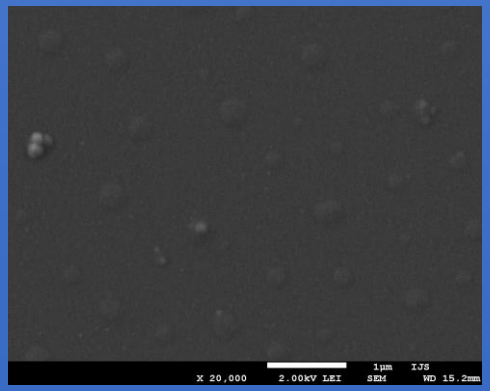
- Less pumping, less outgassing.
- Powder need special handling, otherwise they may get loose and fly off the holder. If magnetic it can damage the EM.
- We usually look at a very small area.

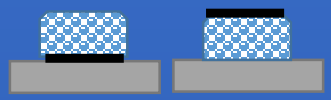
Preparation of bulk: hard material

Composites, metals, rocks, ceramics, glass,…

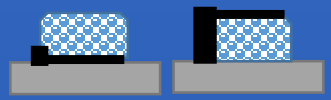
maja.koblar@ijs.si

Surface observation of the material „as it is“





Make **pathway** for e- on the adhesive layer and on the sample:



don't shake

Dry and clean

Use solvents depending on the sample.
If too big cut it with the leaf saw.

Surface topography (SE)

Imaging SE and BSE

Some samples are smooth and have a mirror surface.

Mounting securely on a SEM stub

SEM

If conductive, use high voltage, if nonconductive use low voltages or a variable pressure SEM.

coating

SEM

Make a path to ground.

etching

Avoid microanalysis.

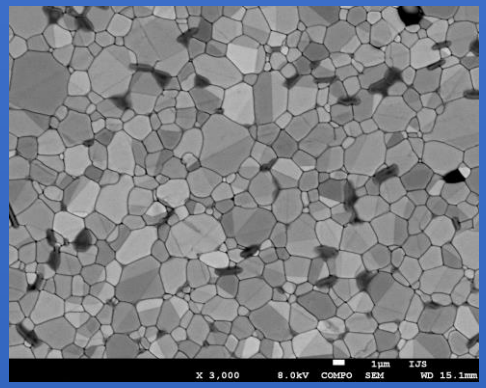
SEM

coating

SEM

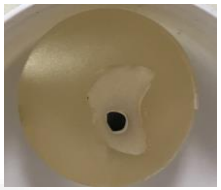
Surface observation and open surface of the sample´s inner structure

(for BSE, EDS, WDS, EBSD)



Cutting

Make sure that the selected part is representing the whole sample.



Mounting , embedding in resin…

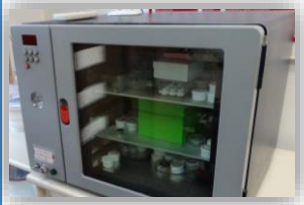
grinding

polishing

cleaning, mounting on a SEM stub

SEM

Storing the samples in a vacuum dryer.



coating

SEM

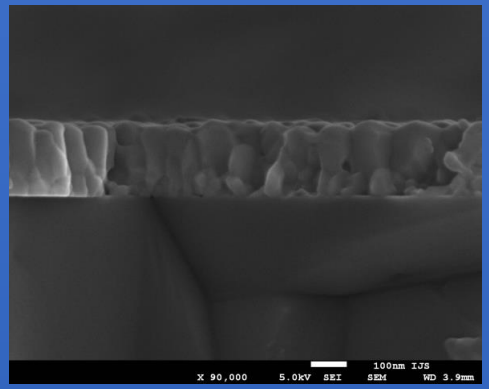
etching

SEM

coating

SEM

Fractures or cross sections, exposure by breaking, cutting, cleaving, snapping and pulling.

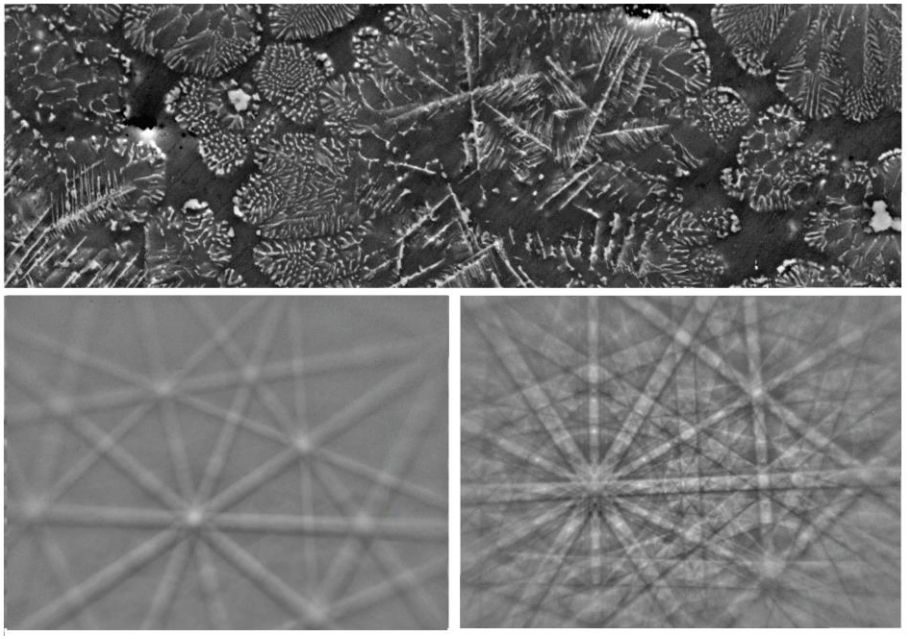


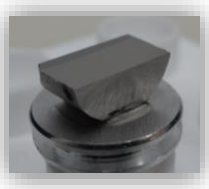
Unpolished:

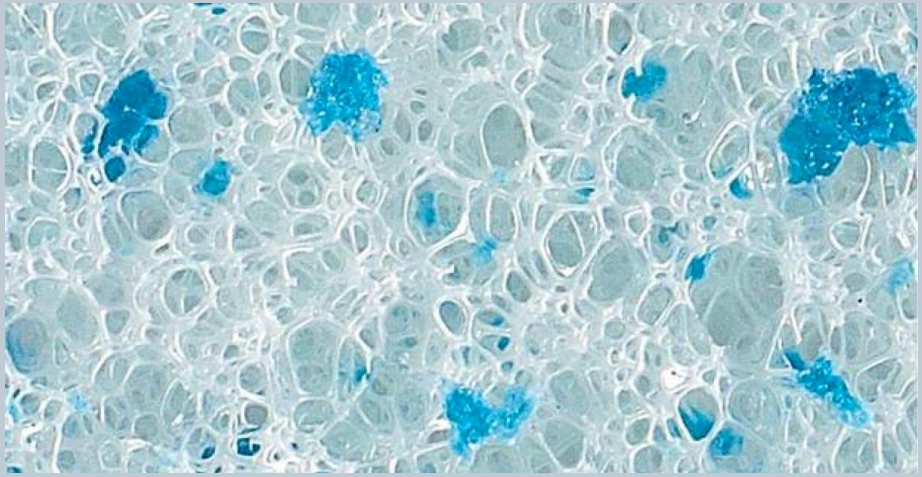
- Topography (SE)

Polished to a smoth surface:

- Topography (SE)
- Compositional imaging (BSE)
- Microanalysis





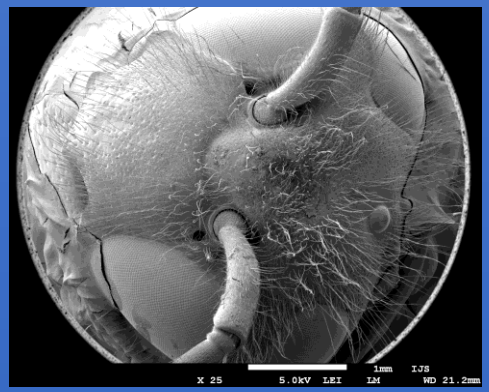


Preparation of bulk: soft and firm material

Biological samples, paper, polymers, foams, gels, wood, food, …

maja.koblar@ijs.si

Surface observation of the material „as it is“



fixation

Fixation stops cellular processes and aims to preserve the specimens as close as possible to its natural state.

chemical

vapour

critical point drying

chemically dry

freeze dry

The sample is fixated, dyhidrated and dry

Mounting on a SEM stub

SEM

coating

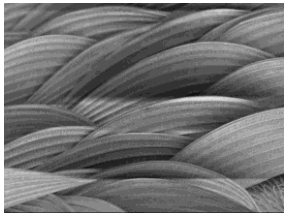
etching

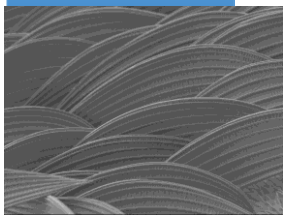
Storing the samples in a vacuum dryer.

SEM

SEM

coating





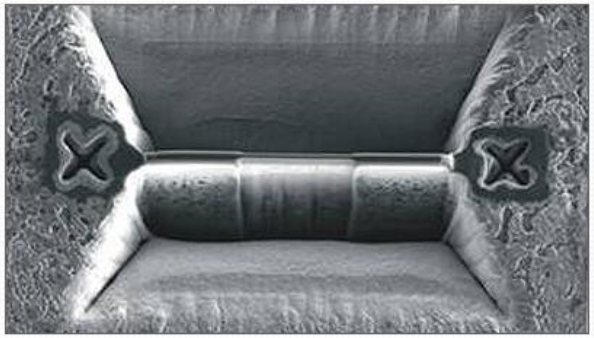
Sometimes charging can be a problem

Surface observation of samples inner sturture, cross sections and thin sections

Using: Scalpels, FIB, Ultramicrotome, …

Image: SCAN

Image: embalmers.com



Images: Nanocenter, FIB

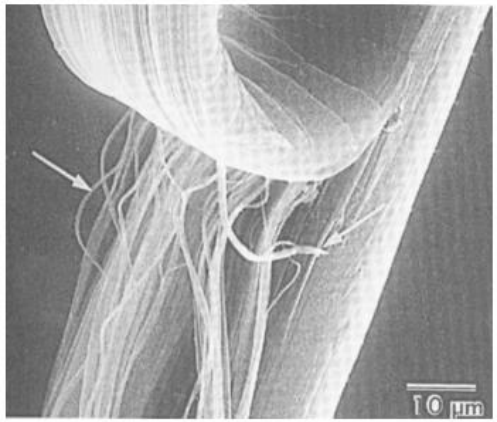


Image: Goldstein, fiber

Preparation of powders

Powders, colloids and magnetic particles

CEMM

CENTER FOR ELECTRON MICROSCOPY AND MICROANALYSIS

maja.koblar@ijs.si

Large particles:

Diameter above 10 mm.

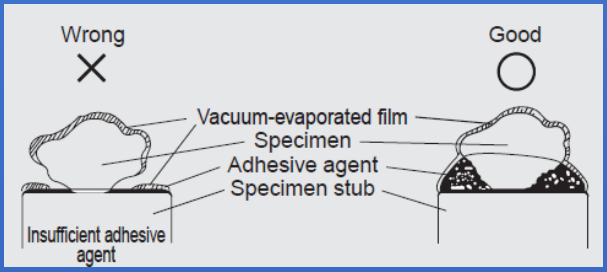


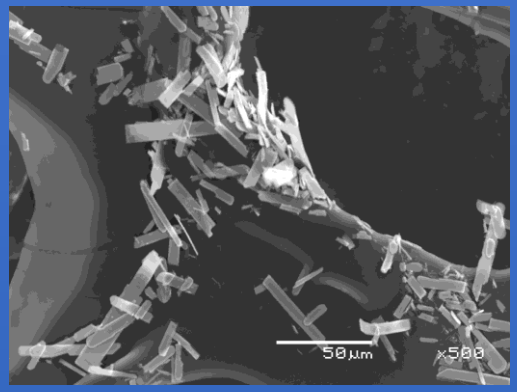
Image: Jeol

Particles diameter > 10 mm:

- Use carbon holders or aluminium holders
- Place a drop of **carbon paint** and spread the drop to form a layer
- Wait for the paint to evaporate to near dryness
- Collect a **small amount** of sample using a make sure that it is representable.
- While the paint is still tacky, place the particles:
  - With forceps and binocular microscope
  - Droop them – the momentum from falling will embed the particles into the paint
- Gently tab the stub on the table to remove excess sample.
- Blow away excess sample using air duster.
- If non conductive, use low voltage or variable pressure SEM or coat the sample.

Small particles:

Diameter between 5 µm and 10 mm.



10 mm > particle diameter > 5 µm:

- Use carbon holders or aluminium holders
- Place a double sided carbon / copper tape
- Collect a **small amount** of sample using a transfer pipet, make sure that it is representable.
- Apply a thin layer of powder on the tape.
- Gently tab the stub on the table to remove excess sample.
- Blow away excess sample using air duster.
- If non conductive, use low voltage or variable pressure SEM or coat the sample.

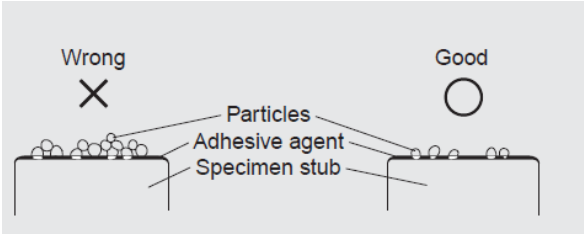
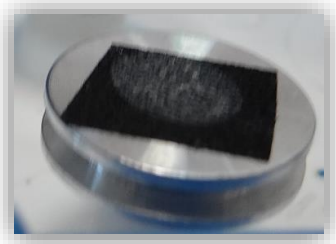
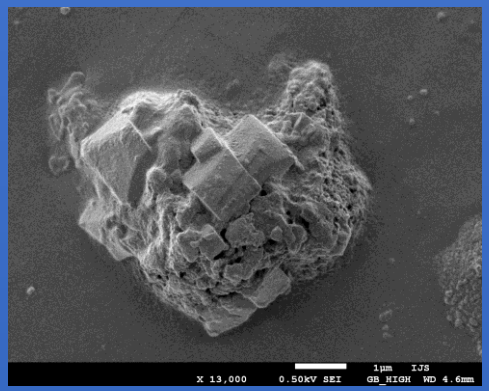


Image: Jeol



Smaller particles:

Diameter below 5 µm

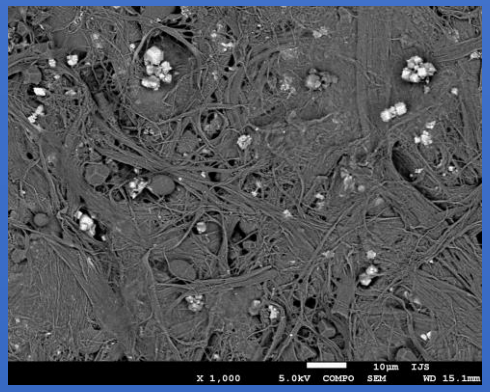


Particle diameter < 5 µm:

- Use carbon holders or aluminium holders
- Add **small amount** of powder in solvent, make sure that is representable (particle settling).
- Put it in the ultrasound to disperse the particles well.
- Place a **small drop** of suspension on a flat surface, again make sure that is representable (particle settling).
- Wait till it dries.
- Coat the sample with carbon to provide an additional adhesion.

Dispersion can also bi made with Freon. The faster the solution evaporates, the less particle aggregation will occur.

BSE or inner surface

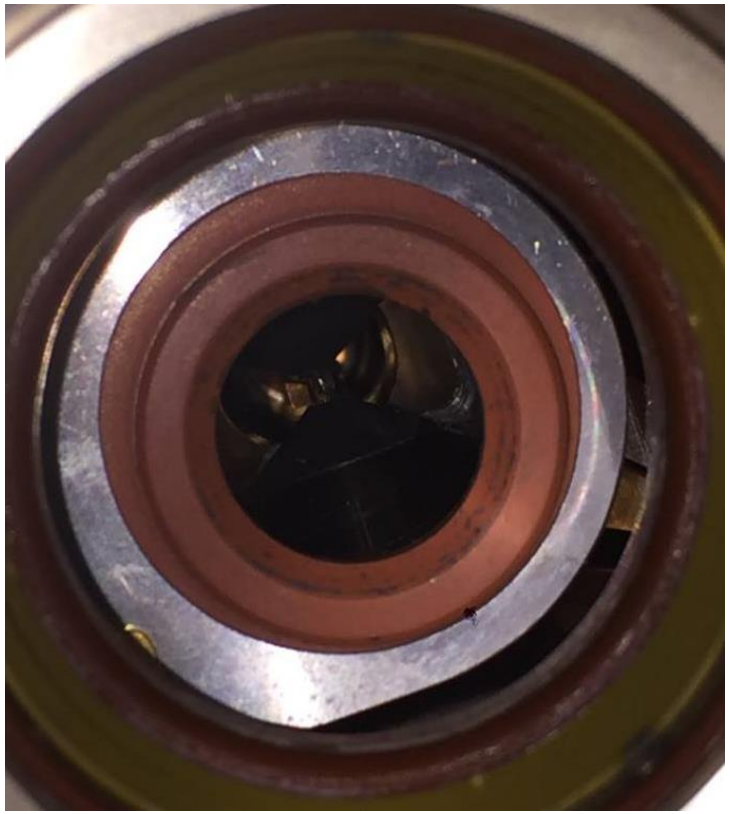


BSE imaging:

- The powder should be hot-mounted in resin or cold-mounted (embedded) in epoxy resin, polymerised
- Polished to a smooth mirror surface
- If non conductive, use low voltage or variable pressure SEM or coat the sample.
- Do not use silver paint, due to the size of the silver particles and the high Z

Magnetic materials require special care!

Magnetic materials can be investigated, but **care is needed** so that the powder is not attracted to the objective lens pole piece where it could disrupt the electron optics.



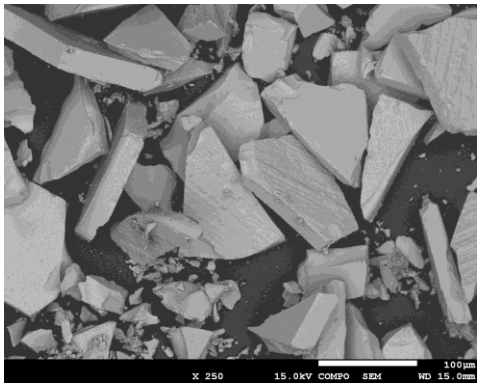


Image: Spela K7

Figure removed for copyright reasons.

Mounting: holders and adhesives

Holders

Mounting stubs are made of:

- Aluminium:
  - has good conductivity, low cost, easy shaping
  - for SE and BSE (topography and compositional)
  - for semi-quantitative (standardless) EDS

- Carbon:
  - for particles and powder
  - for EDS, BSE

- “made with epoxy”:
  - The upper surface polished
  - for BSE, ESD, WDS

Adhesive

Must firmly fix the sample (no mechanical drift when tilted or thermal drift when irradiated by the beam).

Must be vacuum compatible and no outgassing.

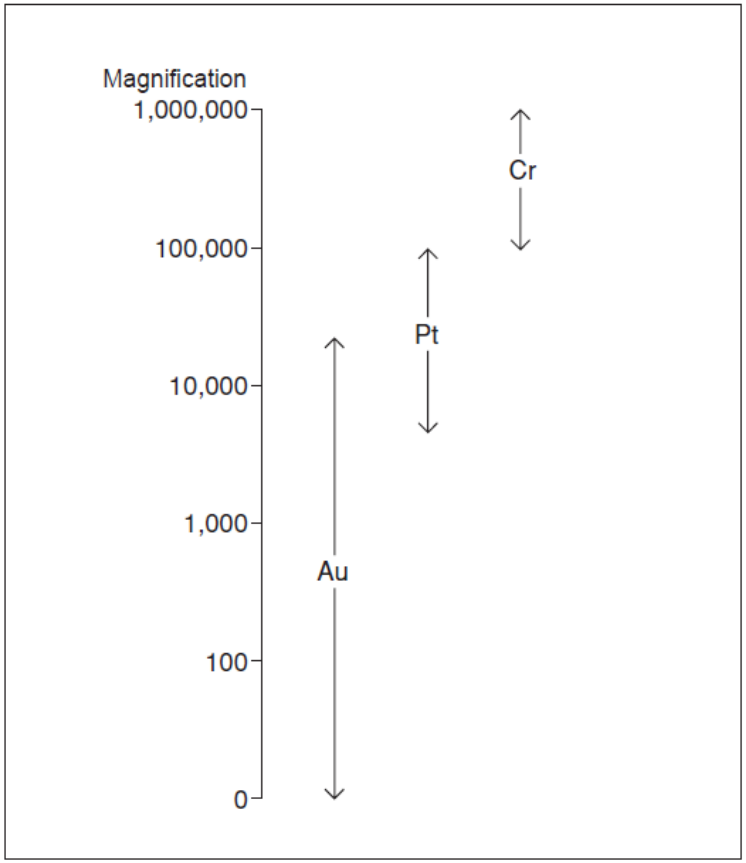
- No super glues – outgassing

- Double sided tape
  - Carbon or copper
- Fast drying paint
  - Carbon or silver
- Plastic conductive carbon cement
  - For irregular shapes
- Two component silver filed epoxy
  - Needs to be heated!

Conductive coating

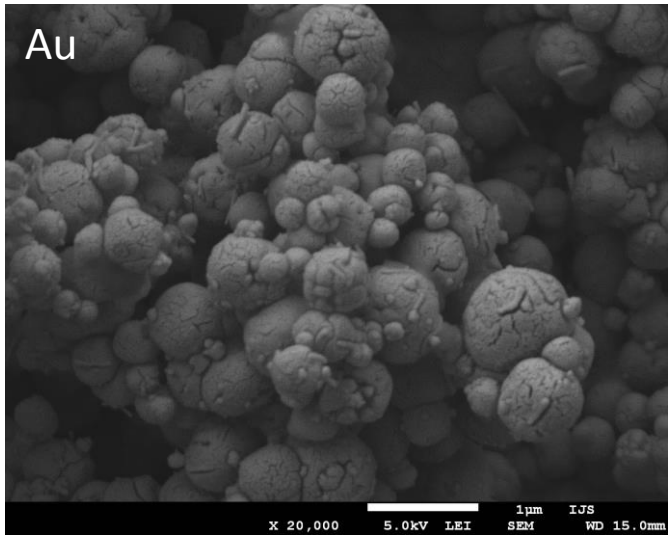
Charging leads to variations in surface potential: deflected secondary e-, increase secondary emission e-, Ddflection of electron beam, spurious x-ray signal

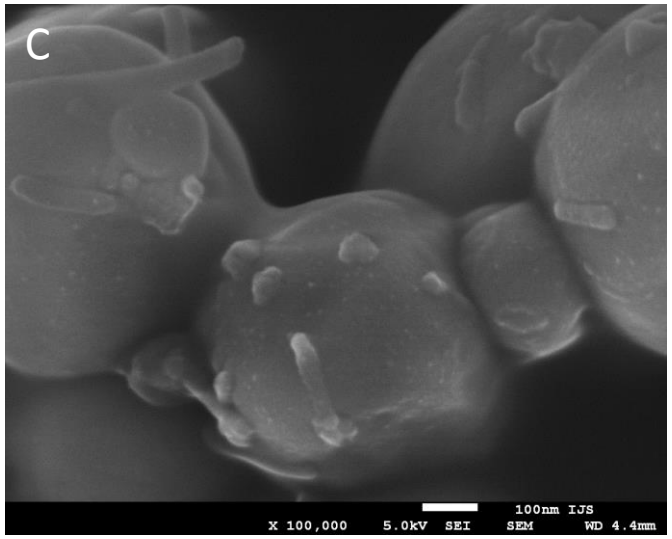
What material should I coat with?



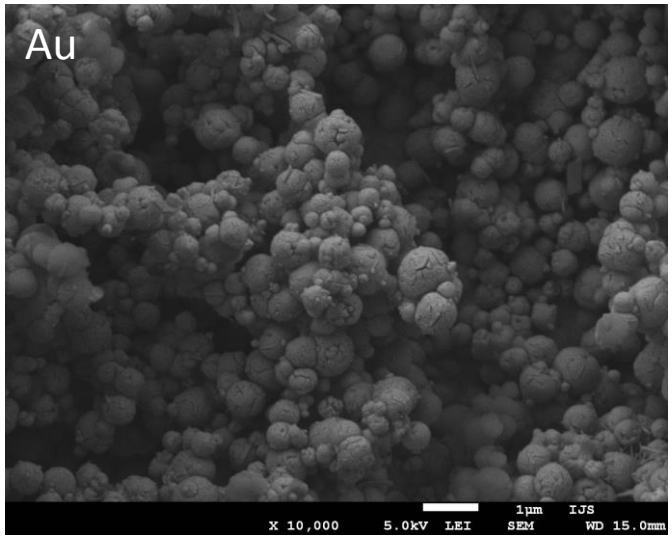
- **Au:** standard for sputtering, easy to coat, inert, stable under electron beam
- **Au/Pd:** all the advantages of Au bus smaller grain size
- **Pt:** highly inert, very conductive but medium grain size
- **Cr:** smallest grain size, but oxides quickly
- **C:** best for X-ray microanalysis and EBSD

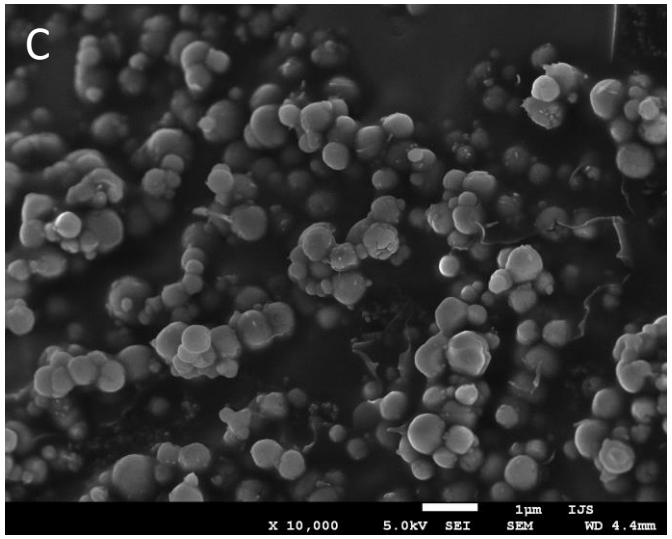
Polimer coated with Balzers





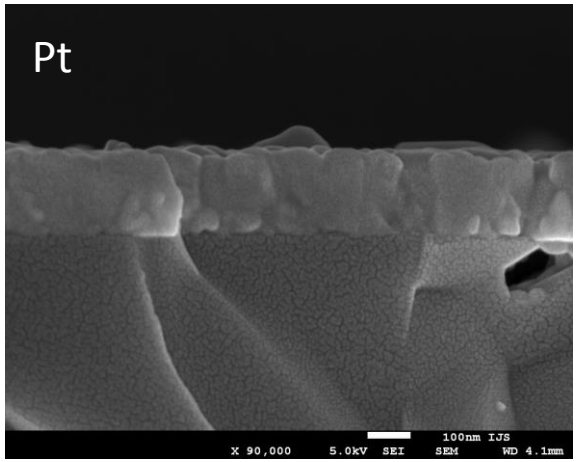
HIGH MAG

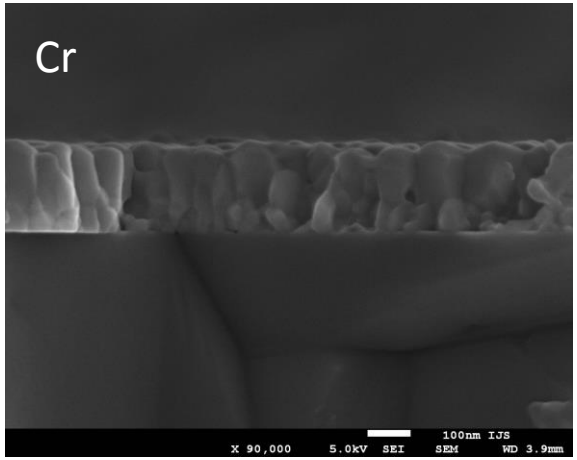


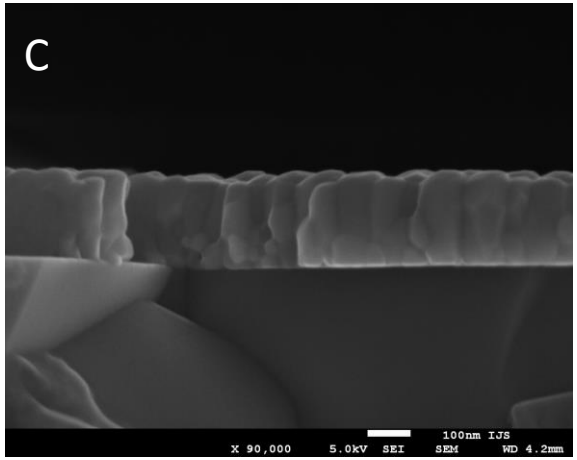


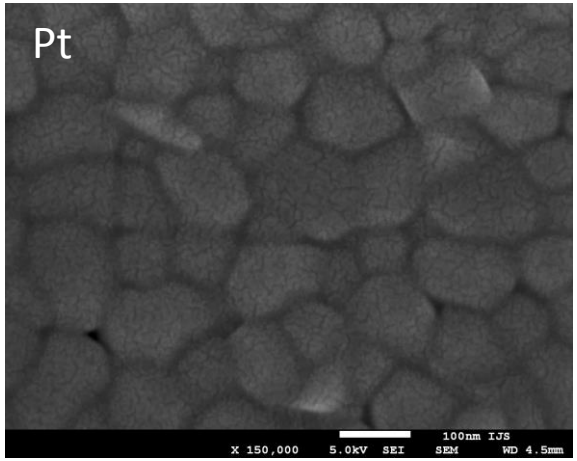
LOW MAG

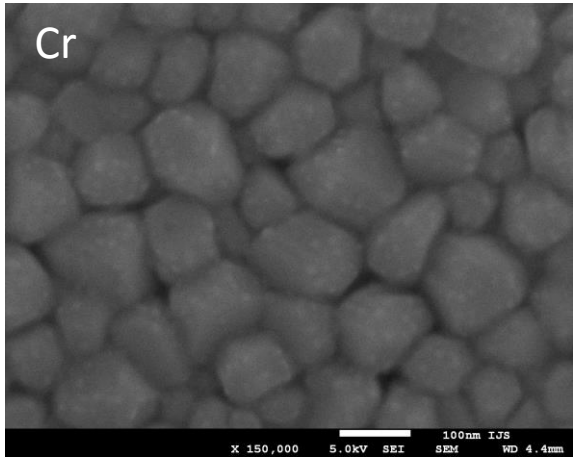
Cheramics coated with PECS

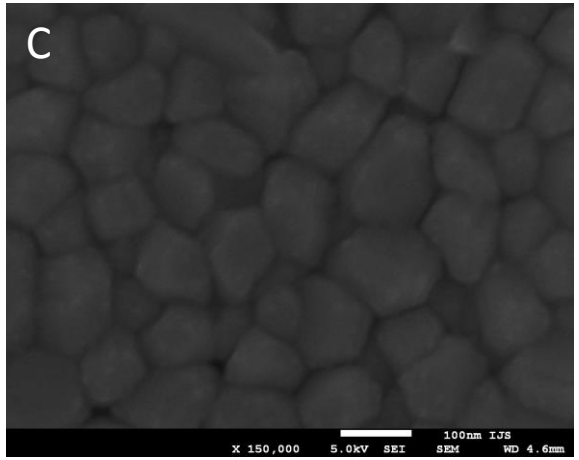




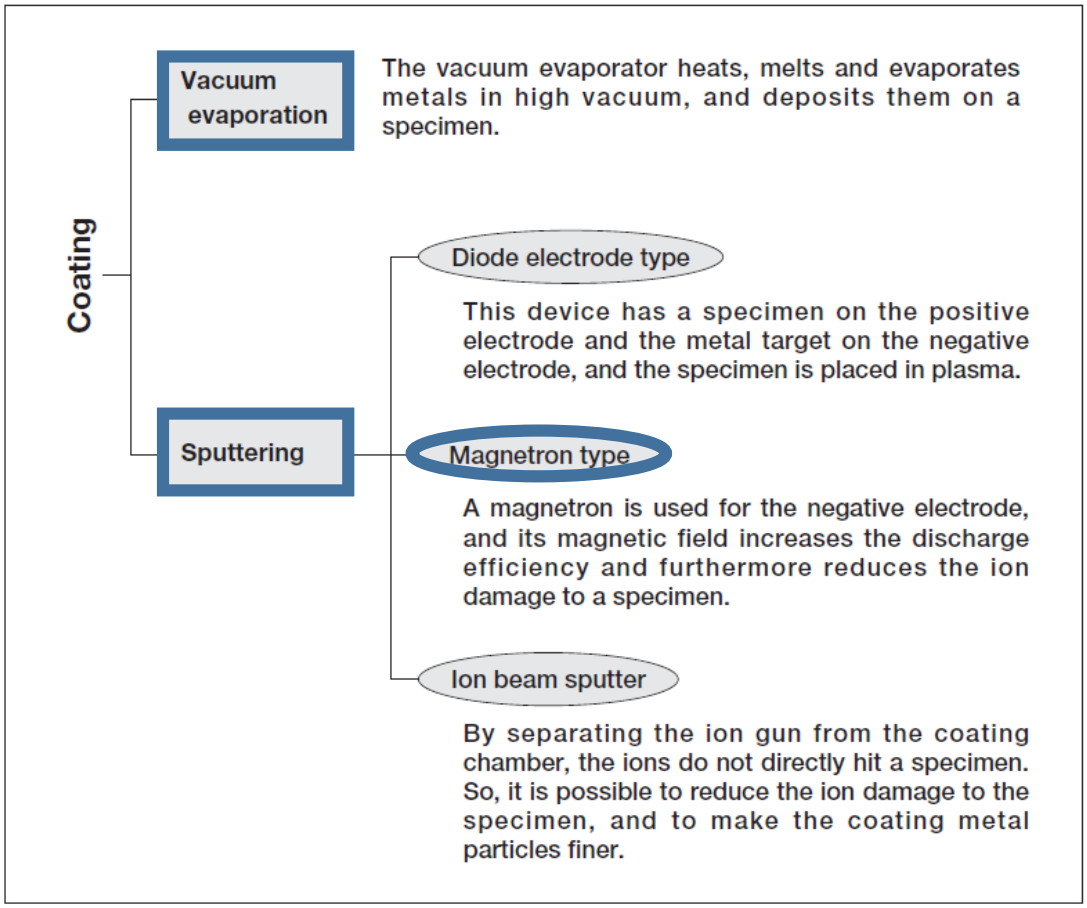




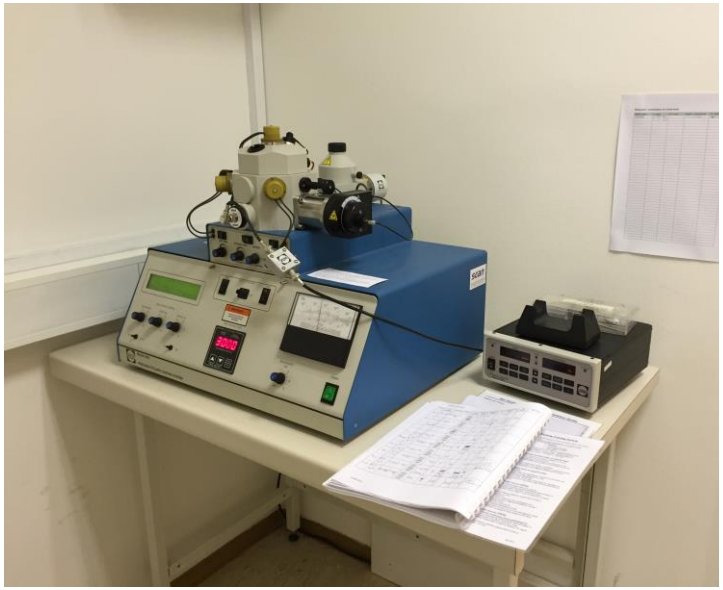


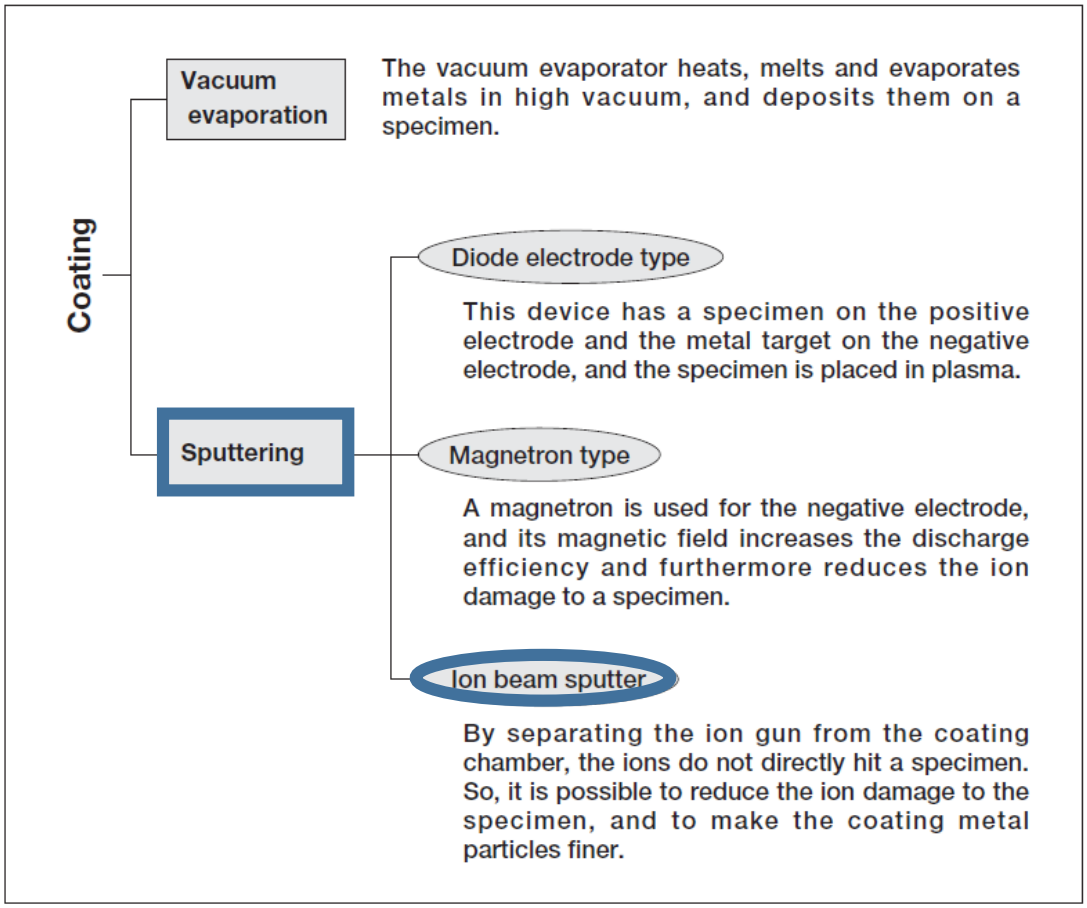


Coating the sample – Balzers



Coating the sample - PECS





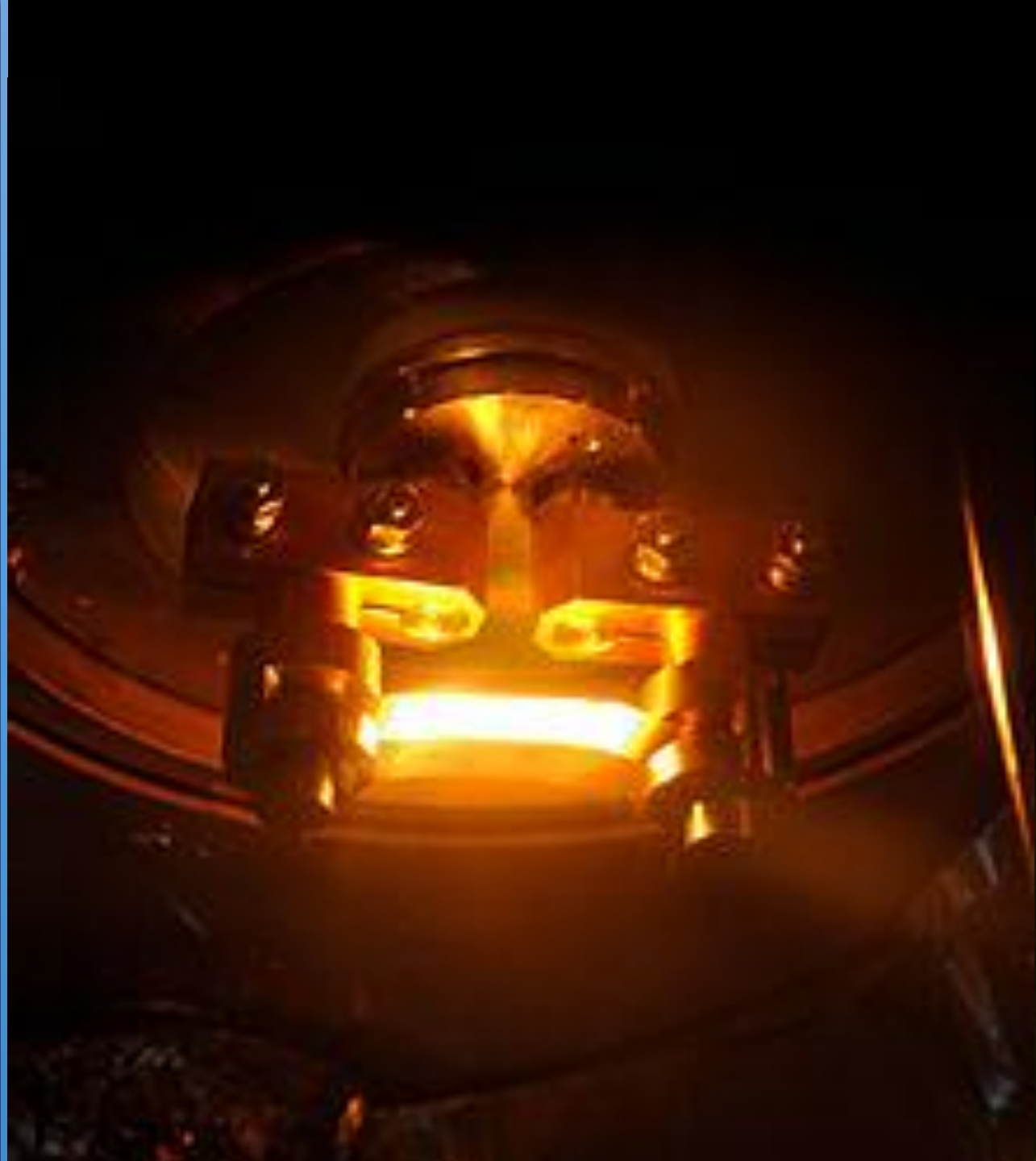
EVAPORATION

The source is **heated** directly or indirectly until the point is reached where it efficiently sublimes or **evaporates**.

The atoms or molecules start to leave the surface of the source and **travel in more or less straight** path until they reach another surface (substrate, wall).

Since these surfaces are at **lower T**, the molecules will transfer there energy to the substrate, lower their T and **condense**.

Other than pressure and temperature, the placement of the heater, source and substrate are important factors



Vapor pressure of elements

Below the vapour pressure surface evaporation is faster than condensation, above it is slower.

The vapor pressure of any substance increases non-linearly with temperature according to the Clausius- Clapyeron equation:

$$\frac{dP}{dT} = \frac{\Delta H(T)}{\mathrm{T}\Delta V}$$

P – pressure,
T – temperature,
H – entropy,
V – specific volume

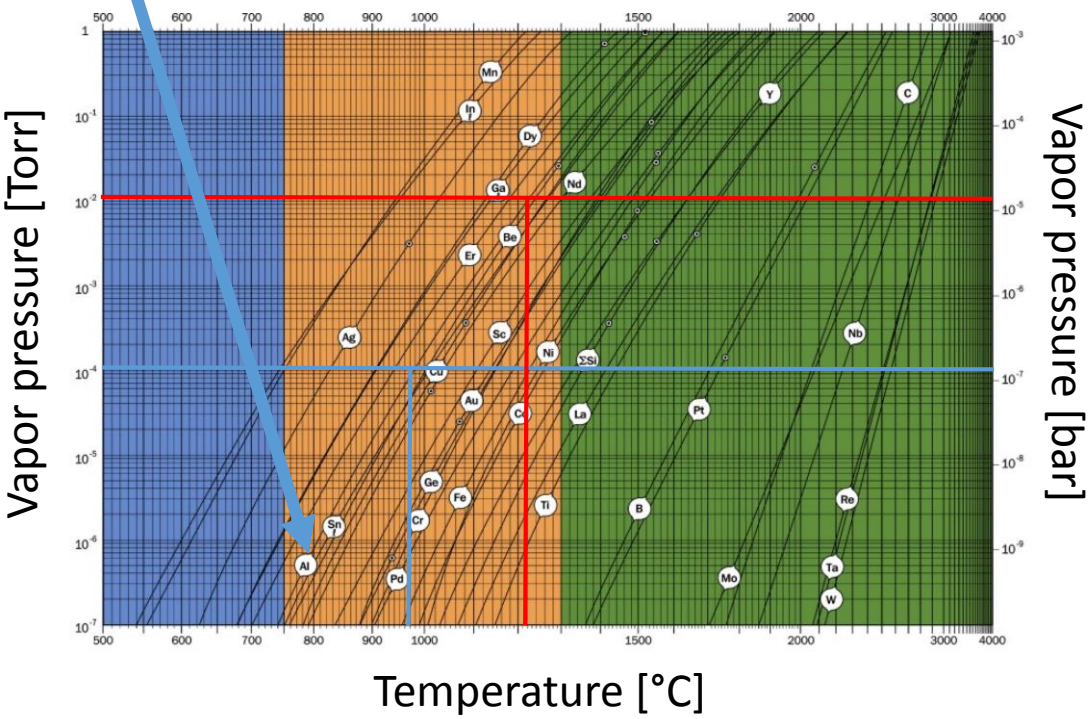
Mass evaporation rate:

$$\Gamma_e = 5{,}84 \cdot 10^{-2} \sqrt{\frac{M}{T}} P_v \frac{g}{cm^2 s}$$

M – molar mass
P_v – vapor pressure

Evaporation rate for aluminium

- Al: M=27 g/mol



$P_v = 10^{-7}$ bar = 10^{-4} Torr → 980°C:

$$\Gamma_e = 5{,}84 \cdot 10^{-2} \sqrt{\frac{27}{980}}\, 10^{-4}\, \frac{g}{cm^2 s} = \mathbf{9{,}694 \cdot 10^{-7}}\, \frac{g}{cm^2 s}$$

$P_v = 10^{-5}$ bar = 10^{-2} Torr → 1220°C:

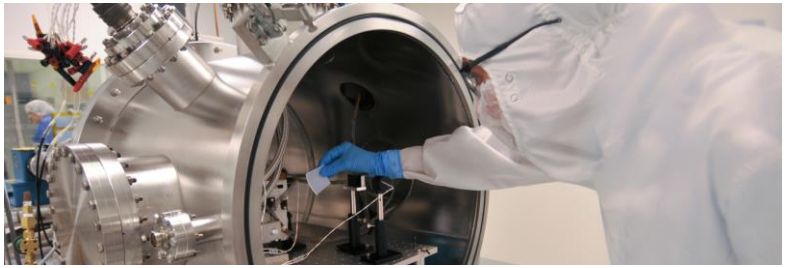
$$\Gamma_e = 5{,}84 \cdot 10^{-2} \sqrt{\frac{27}{1220}}\, 10^{-2}\, \frac{g}{cm^2 s} = \mathbf{8{,}688 \cdot 10^{-5}}\, \frac{g}{cm^2 s}$$

Sources of impurity

What can effect film purity?

- Contamination of source materials- use high purity of source material
- Contamination of the heater - use material with low diffusion
- Residual gas in the chamber- better vacuum, higher deposition rate

(Image: wiki)



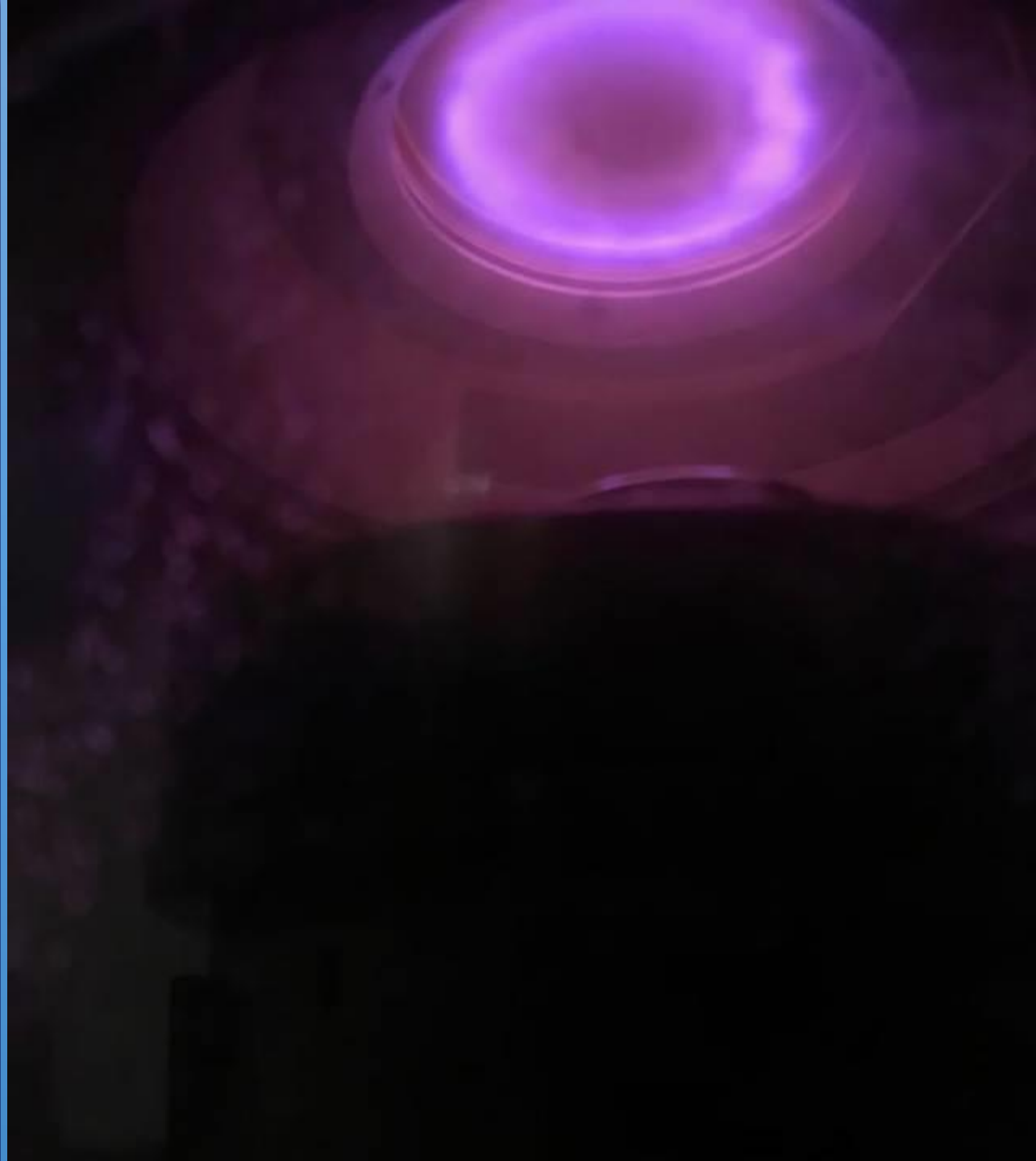
(Image: lasp)

SPUTTERING

Instead of using heat to eject material from a source, we can bombard them with **high speed particles.**

The **sputtering gas bombards** the target and sputters off the material we would like to deposit.

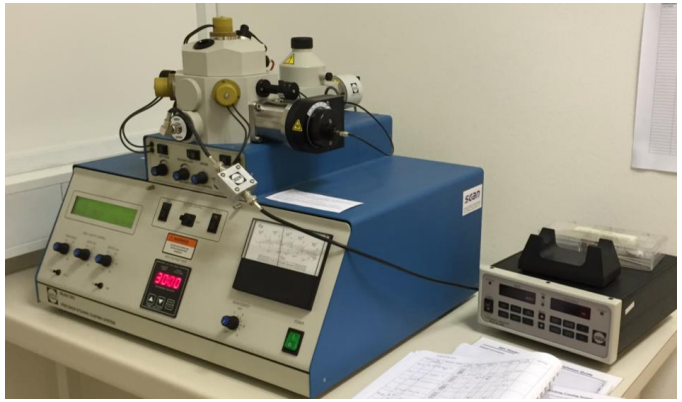
Once ejected, these atoms (or molecules) can travel to a substrate and **deposit as a film**.



Sputtering yield – S [atoms/ions]

Number of ejected (sputtered) atoms, molecules from target divided with the incident particle, ions

$$S = \frac{Number\ of\ sputtered\ atoms}{Number\ of\ incident\ ions}$$

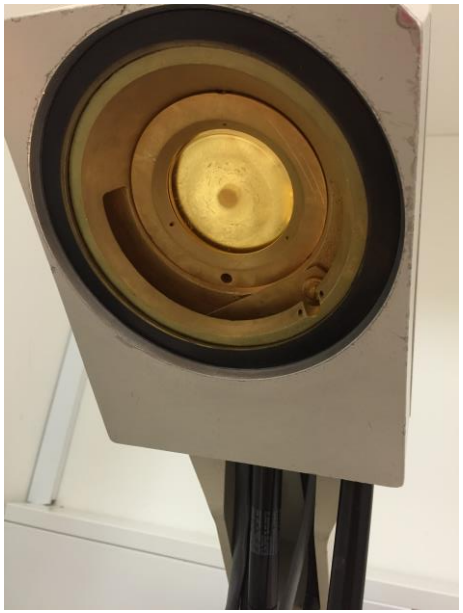


S depends on:

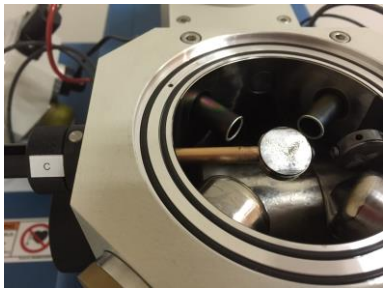
1. type of target atom and the binding energy
2. relative masses (of ions and atoms)
3. angle of incidence of ions and kinetic energy

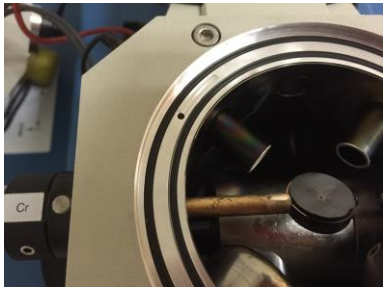
Hints:
C: 8-9 keV, L&D 280 -300 µA, 0,2 - 0,4 Å/s,
min 4 nm (2 min) **max 8 nm** (4 min)
Au/Pd: 6-7,5 keV, L&D 200 - 220 µA, 1,2 - 1,3 Å/s,
min 3 nm (25 s) **max 6 nm** (50 s)
Pt: 6-7 keV, L&D 200 - 220 µA, 0,7 – 0,8 Å/s,
min 3 nm (40 s) **max 6 nm** (80 s)
Cr: 6-7 keV, L&D 200 µA, 0,5 – 0,7 Å/s,
min 3 nm (1 min) **max 8 nm** (2,5 min) + **LN_2 trap**

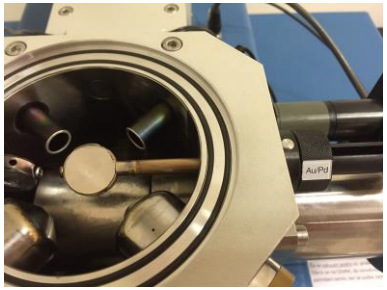
Targets on SCD and PECS



Magnetron target (Au, Pt) demonstrating racetrack erosion profile







PECS targets (Cr, Au/Pd, C and Pt)

Physical mechanisms of thin film growth

Figure removed for copyright reasons.

(Video: Barna, 1967)

Thickness measurment

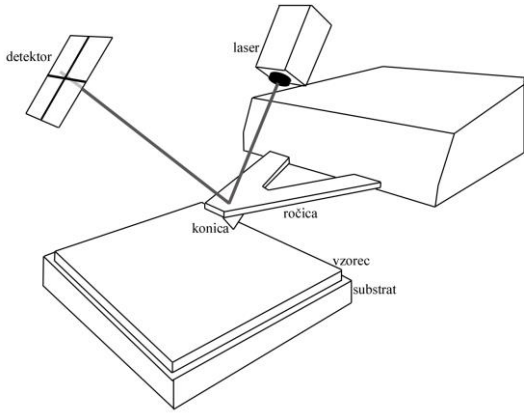
Gravimetric method

- Measure substrate weight before and after coating
- Calculate thickness from known substrate dimensions
- Not real-time but surprisingly accurate

(Image: wiki)

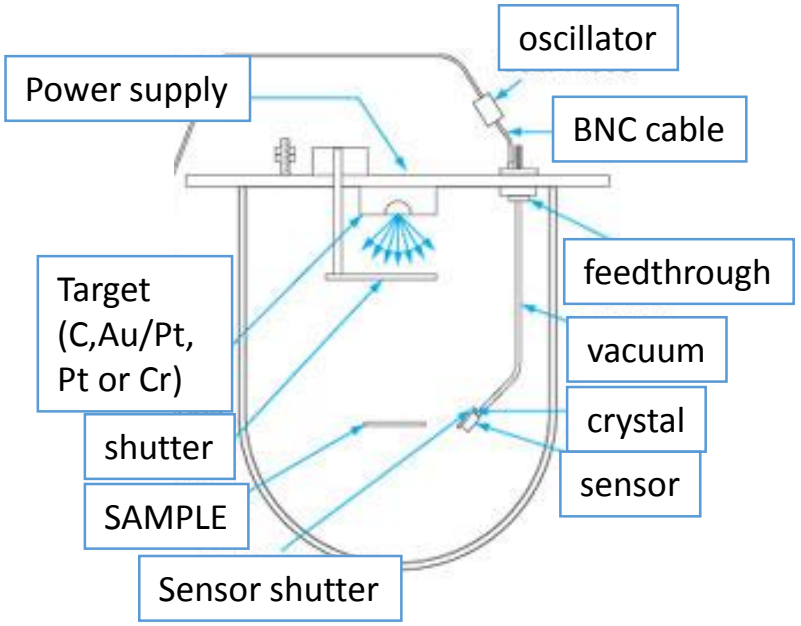
Stylus method – profilometer or AFM

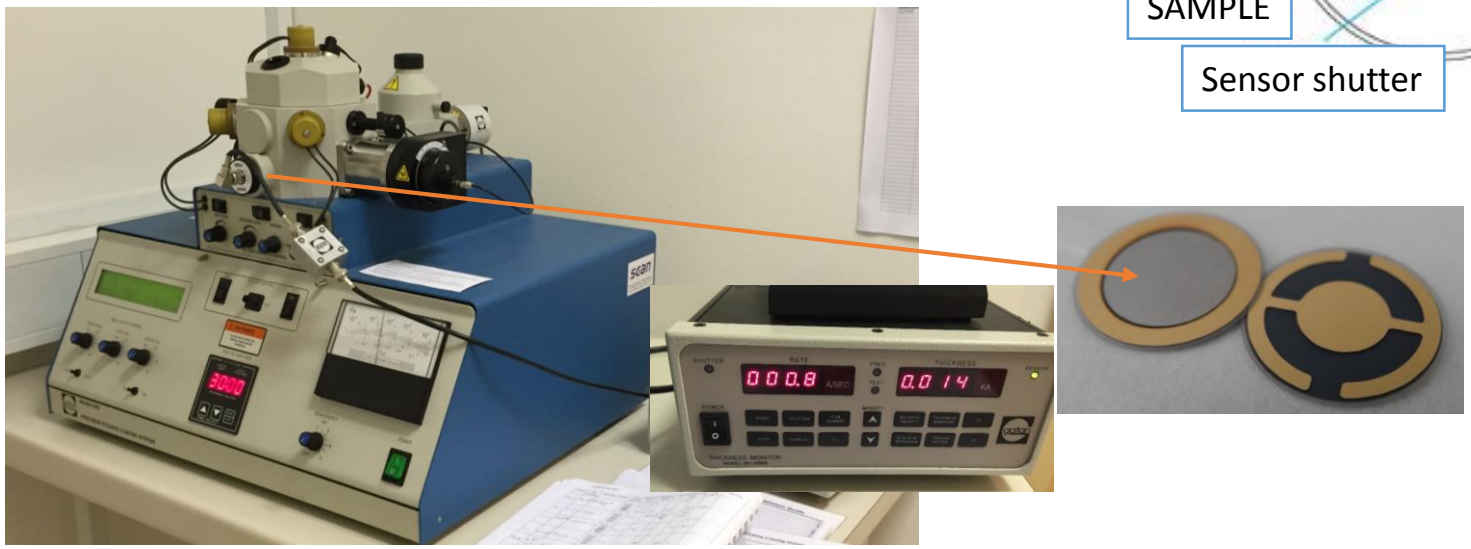
- A stylus is drawn across a step in the film
- Scratching can occur
- Needs calibration
- Can do repeated measurements
- Not real-time



Thickness measurment

- Specific oscillation frequency
- Expose one side of wafer to the vapor
- As the vapor coats the wafer the oscillation frequency changes
- Different parameters, proper calibration, quartz quality and proper usage





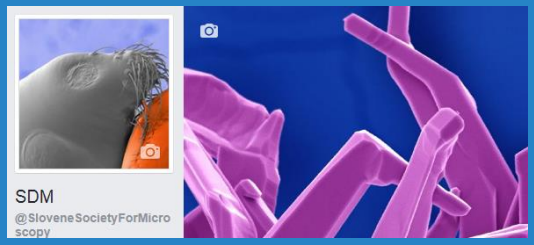
Take home information

- Before SEM characterization
  - Check recent published papers
- Preparation
  - Make sure that the samples **are firmly attached**
- Mounting: holders and adhesives
  - Different holders (carbon, aluminum)
  - Different adhesives, always wait for the adhesives to dry, check if it has to be heated…
- Coating: sputtering and evaporation
  - Always **make ground** (electrostatic charging)
  - Choose the coating depending on the imaging and sample, for BSE use carbon, for biological specimens, polimers use higher Z.
- Thin film growth
  - Different ways how the film growths
  - Be sure that the **surface image is not the coating**, use some uncoated sample to check

Slovene Society for Microscopy

http://mikroskopsko-drustvo.si/

www.facebook.com/SloveneSocietyForMicroscopy/



maja.koblar@ijs.si


QUESTIONS?
COMMENTS?
CONCERNS?