SEM sample preparation

5th CEMM workshop

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Outline

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

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Before SEM characterization

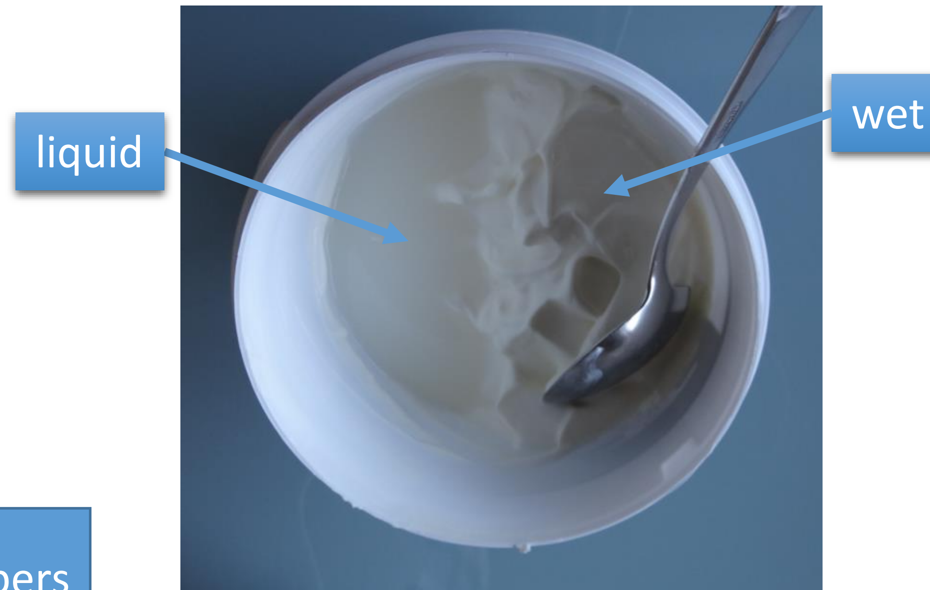
Clean and dry (no outgassing).

Some particles have natural tendency to absorb moisture from regul environment.

For biological samples – „critical drying“ to remove liquid without damaging the specimen.

Always check recent published research papers to check on current techniques being used.

Definition of dry sample



Liquid: paint, milk, mud

Wet: fresh leaf, fresh concreate, a fly

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Sample as small as possible

Less is more!

Figure removed for copyright reasons.

Image: providr.com

Figure removed for copyright reasons.

Image: more.com

The same goes for SEM samples

Why?

- Less pumping, less outgassing.
- Powder need special handling, otherwise they may get loose and fly off the holder.
- Magnetic samples also need experienced SEM operators, or damage on the SEM is possible.

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Bulk: hard material

Composites, metals, rocks, ceramics, glass,…

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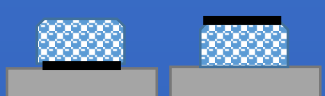
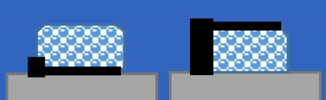
Bulk material

Hard samples

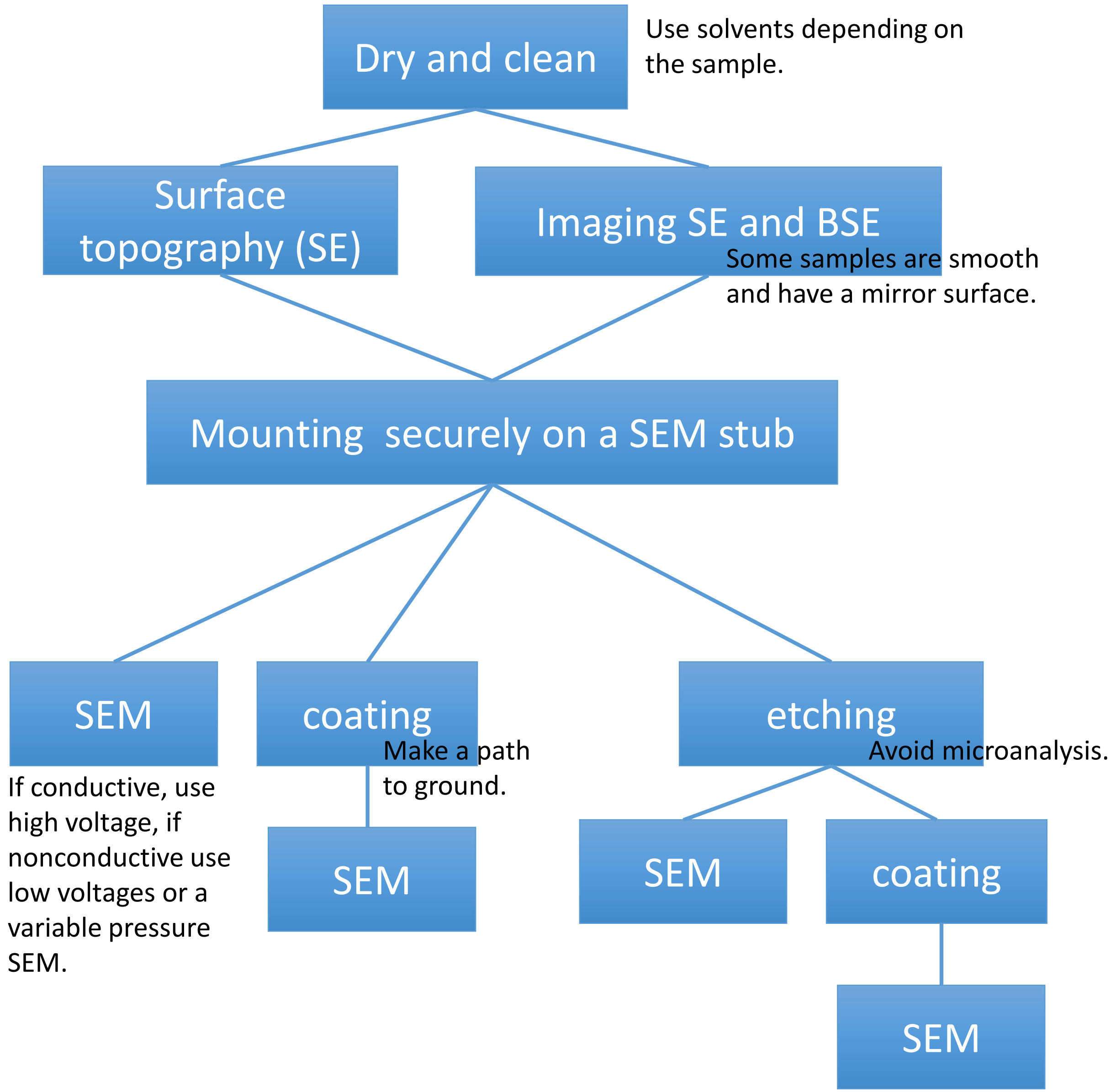
Surface observation of the material „as it is“



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



Surface observation of the material „as it is“

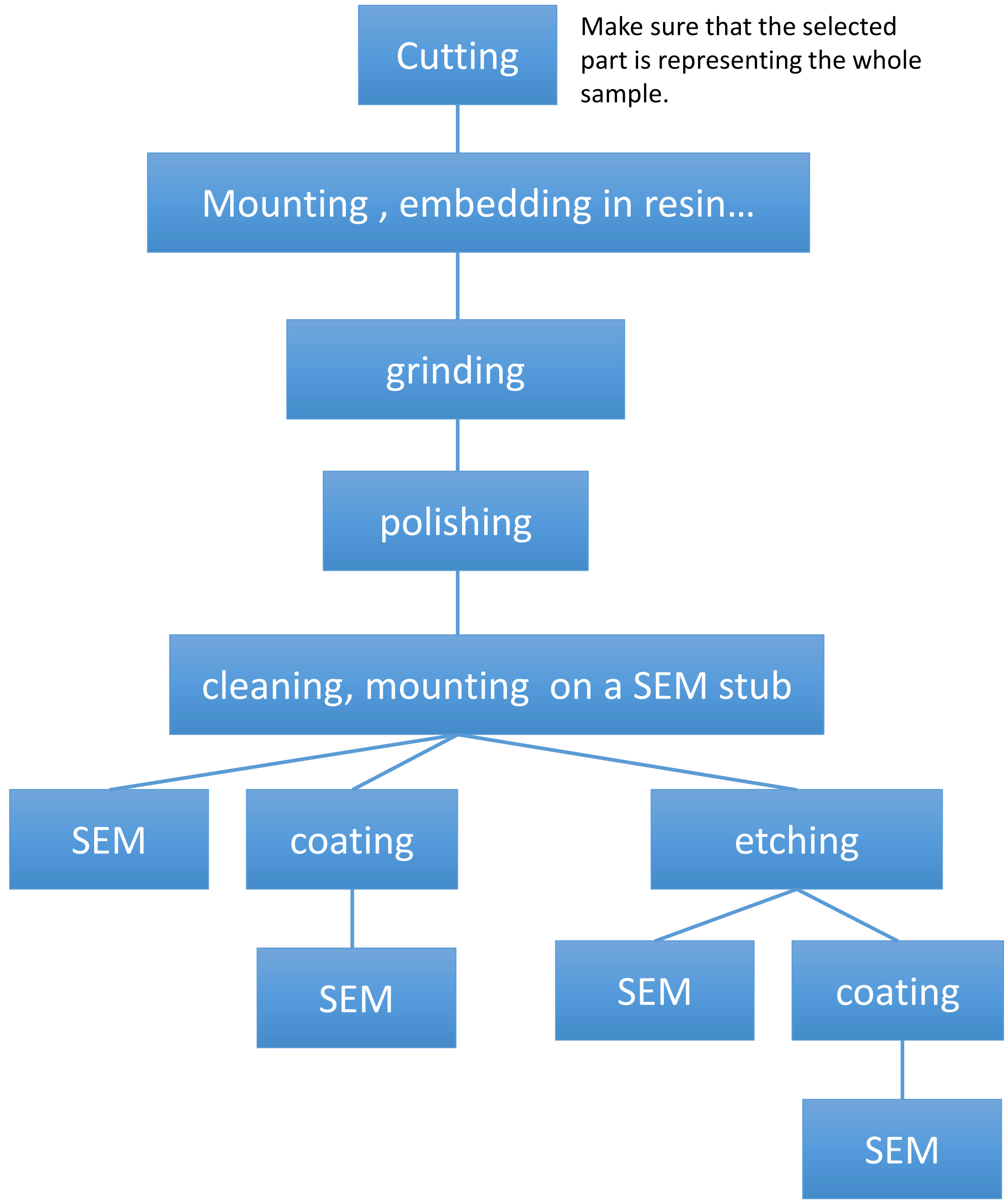


Bulk material

Hard samples

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

(for BSE, EDS, WDS, EBSD)



Bulk material

Hard samples

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

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Images: Dense and highly textured coatings obtained by aerosol deposition method from Ti3SiC2 powder: Comparison to a dense material sintered by Spark Plasma Sintering J. Henon et al. / Journal of the European Ceramic Society 35 (2015)

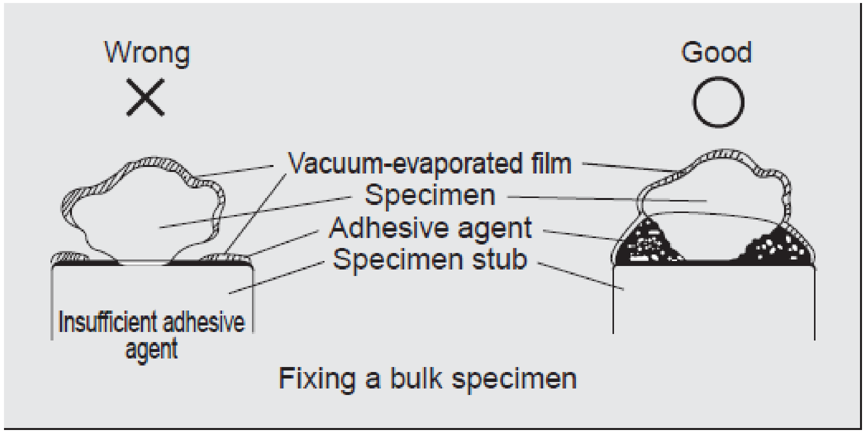

Image: Jeol

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Bulk: soft and firm material

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

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Bulk material

Soft and firm material

Surface observation – as it is

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

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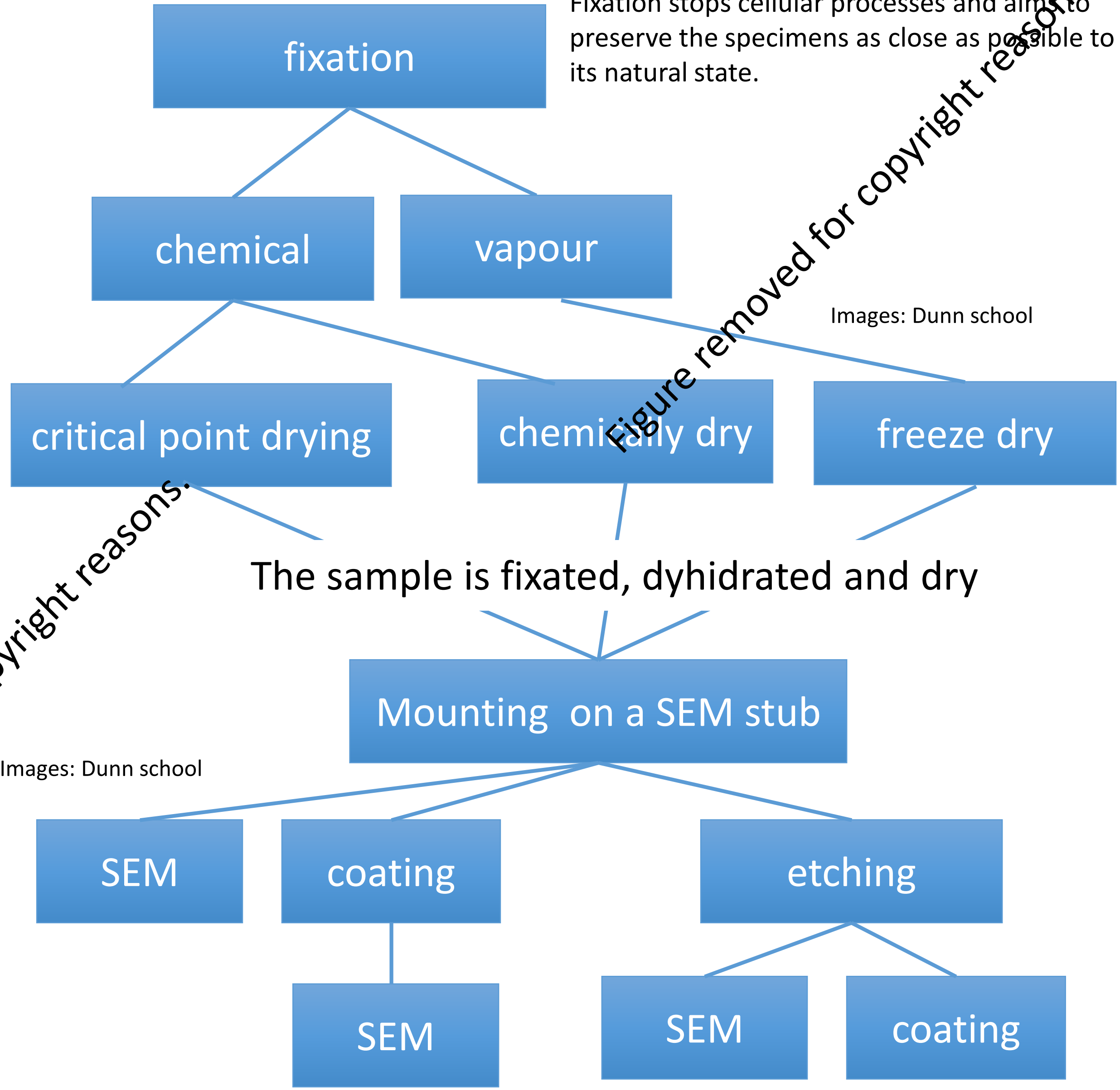
Images: Dunn school



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Images: Dunn school

Bulk material

Soft material

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

Using: Scalpels, FIB, Ultramicrotome, …

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Image: embalmers.com

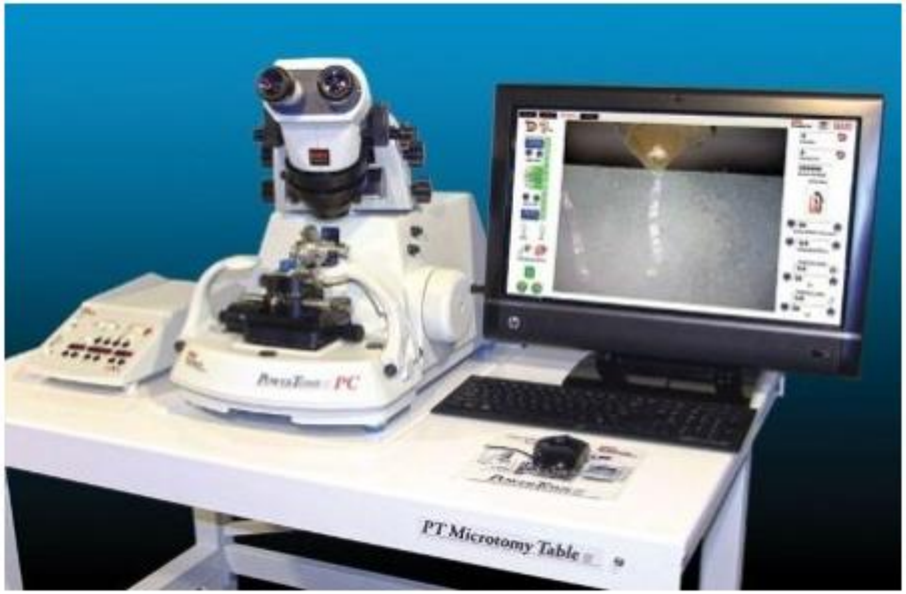

Image: SCAN

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Powders

Powders, colloids and magnetic particles

Powders

Large particles:

Diameter above 10 mm.

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.

Powders

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.

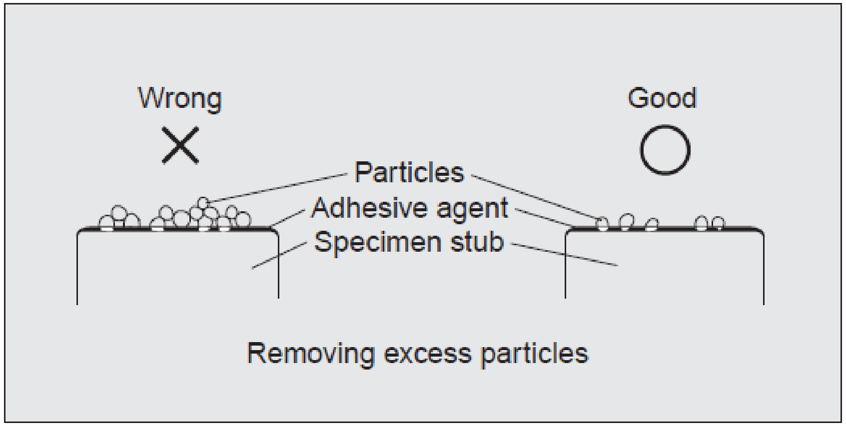

Image: Jeol

Powders

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. So a lamp can help but take care not to damage the organic or biological samples.

Powders

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.

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(Image: IOM)

Ion beam

Cleaning or etching

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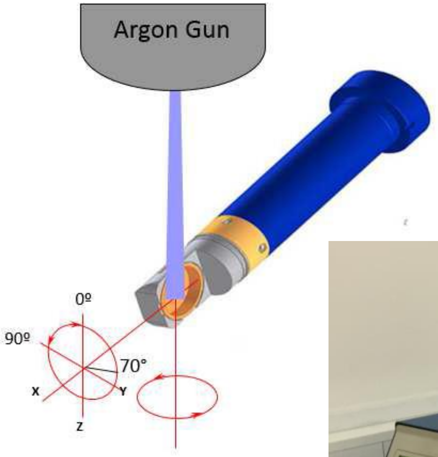
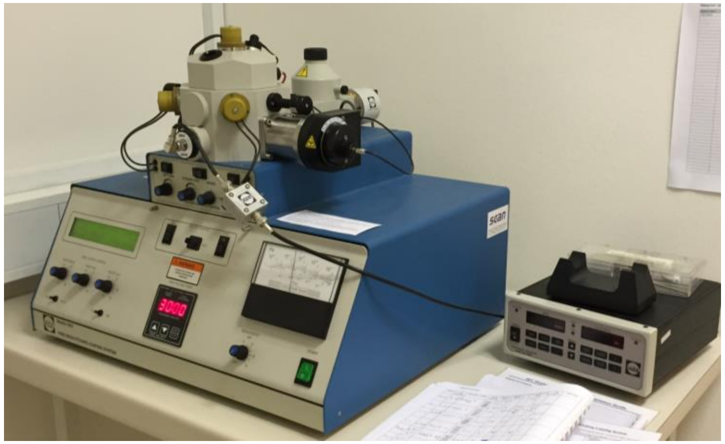
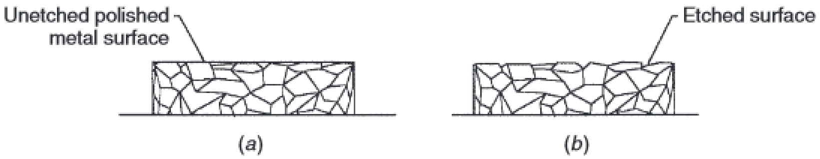
Cleaning



- ion etching



- bench-top instrument
- broad beam - 1 cm^2 area
- Beam energy: 1 – 10 keV



(Image: spectroscopynow)

- For EBSD measurement:
  - 60 ° -70° tilt
  - best results obtained with low kV (2 kV - 3 kV)

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Sample preparation tools

Holders

Mounting stubs are made of aluminium, carbon or epoxy.

- 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

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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!

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Conductive coating

charging leads to variations in surface potential:

Deflected secondary e-

Increase secondary emission e-

Deflection of electron beam

Spurious x-ray signal

(Image: Manfred Kage)

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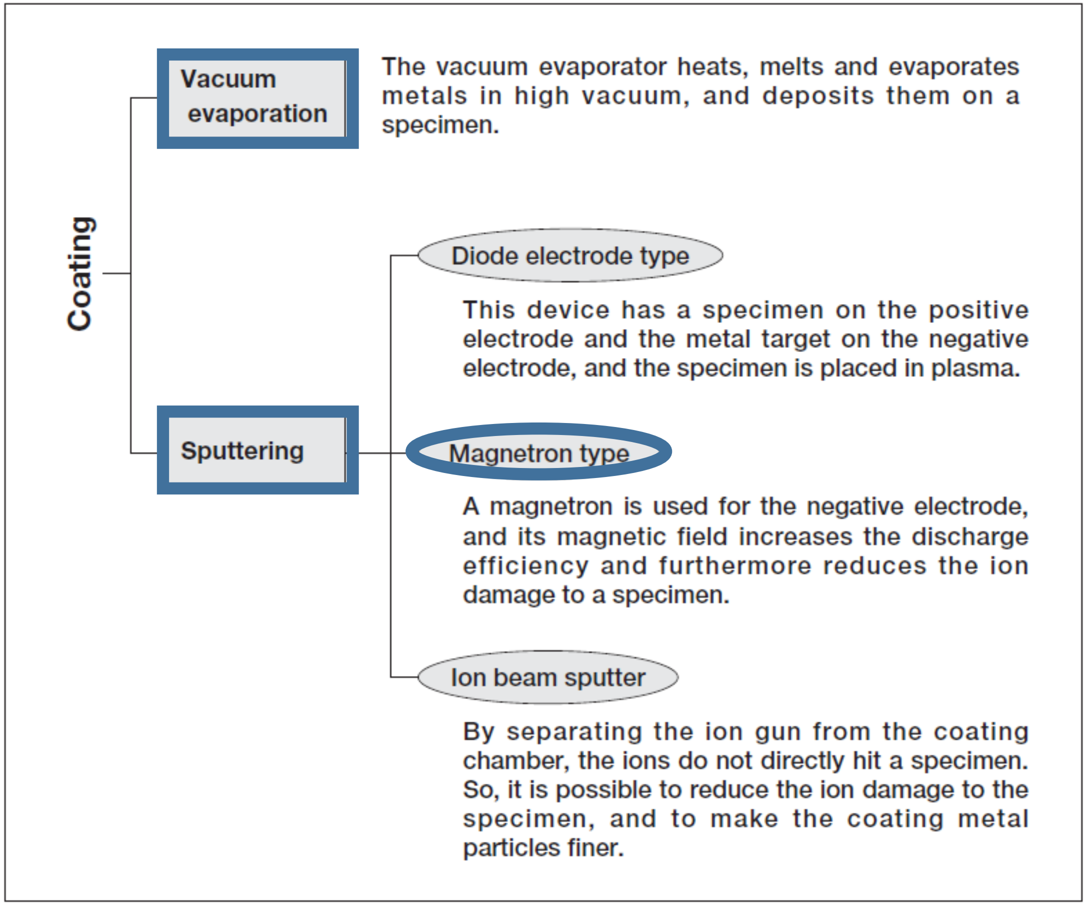
Coating the sample – Balzers



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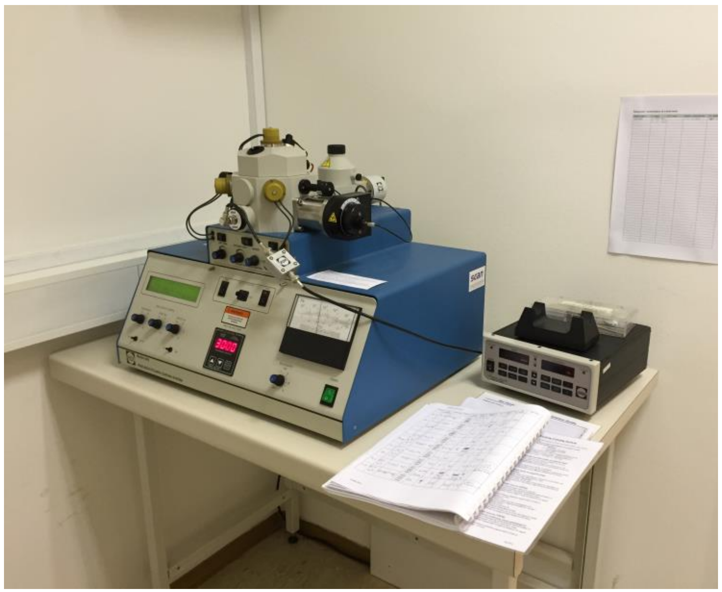
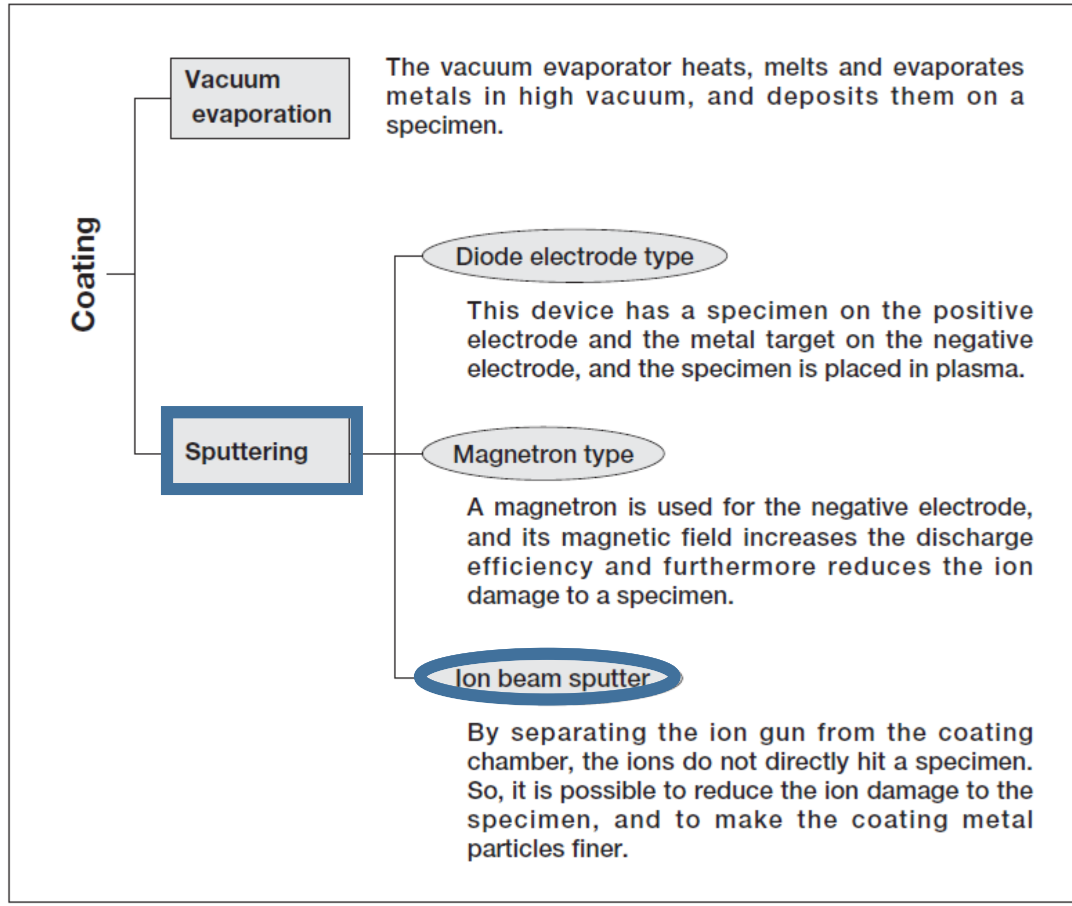
Coating the sample - PECS





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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

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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:

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

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

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

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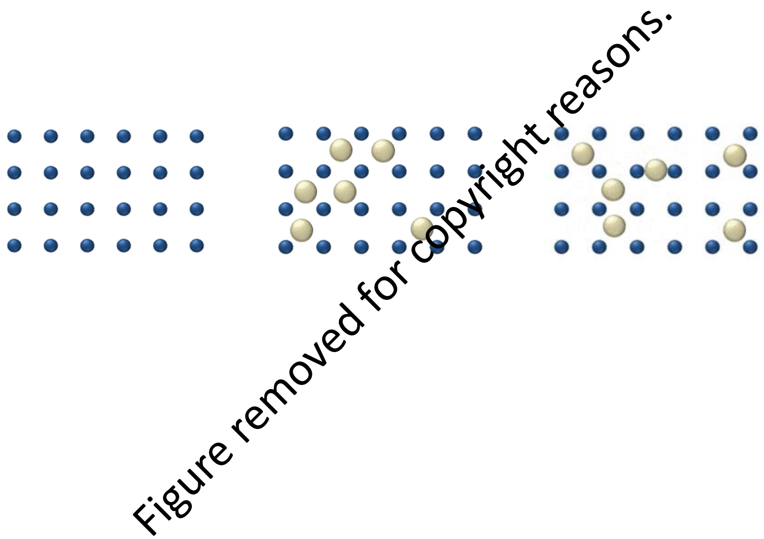
Sources of impurity

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)

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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.

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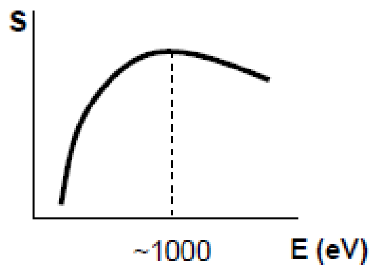
Sputtering yield – S [atoms/ions]

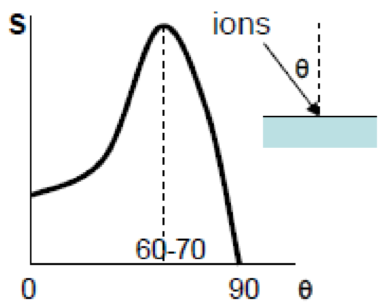
Number of ejected (sputtered) atoms, molecules from target divided with the incident particle, ions

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



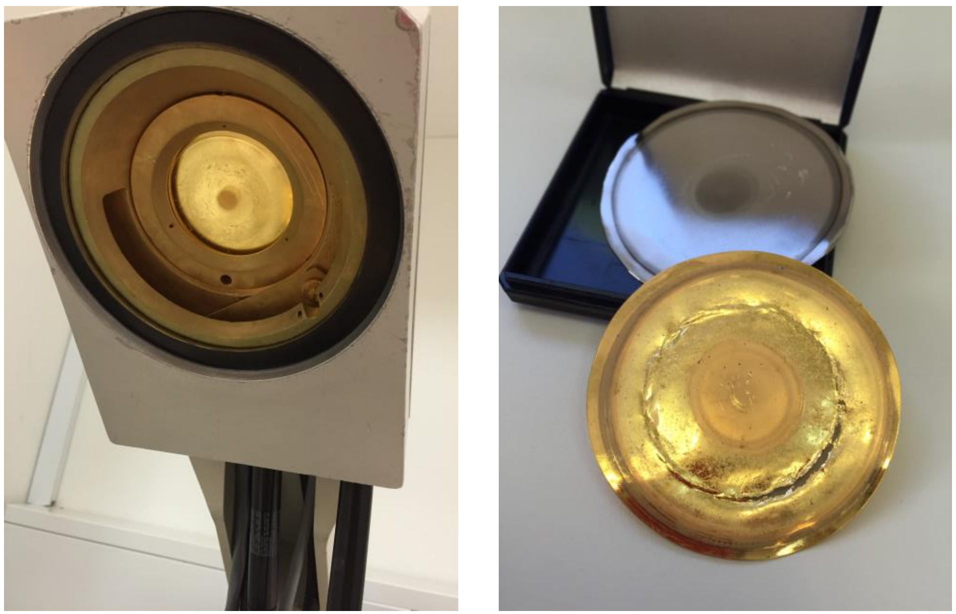
S depends on:

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



Namigi:
C: 8-9 keV, L&D 350 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 past**

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

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(Video: Barna, 1967)

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Thickness measurment

Gravimetric method

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

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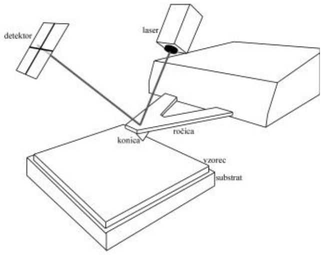
(Image: wiki)

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



(Image: l-w)

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Thickness measurment

- Quartz crystal monitors - Quartz oscillator
- 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
- If **6MHz** oscillator is used – nanogram changes can be measured in turn that means to **0,1A**

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(Image: lesker)

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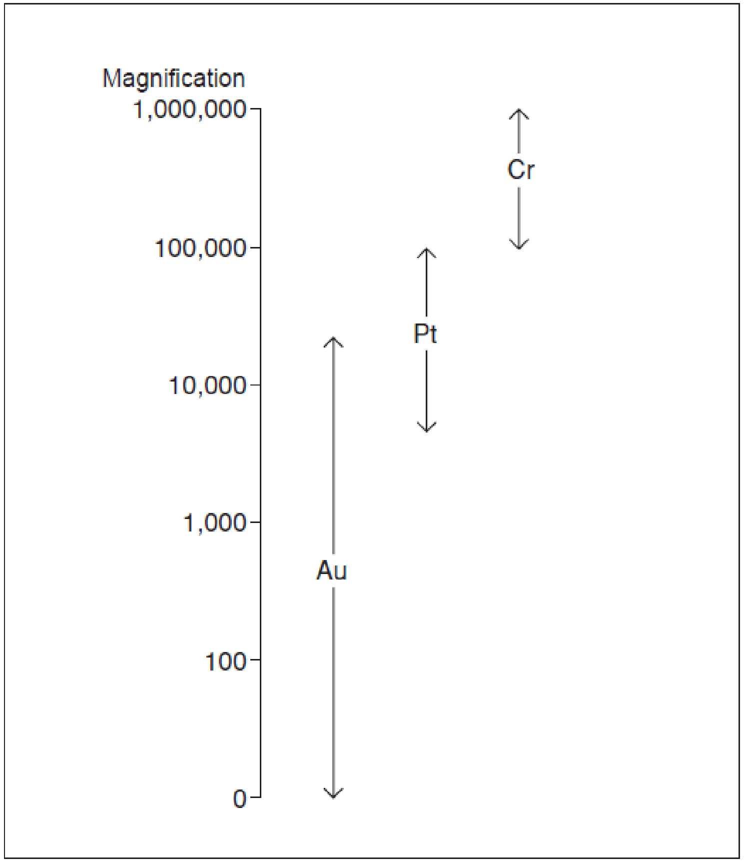
What material should I coat with?



- **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 large grain size
- **Cr:** smallest grain size, but oxides quickly
- **C:** best for X-ray microanalysis and EBSD

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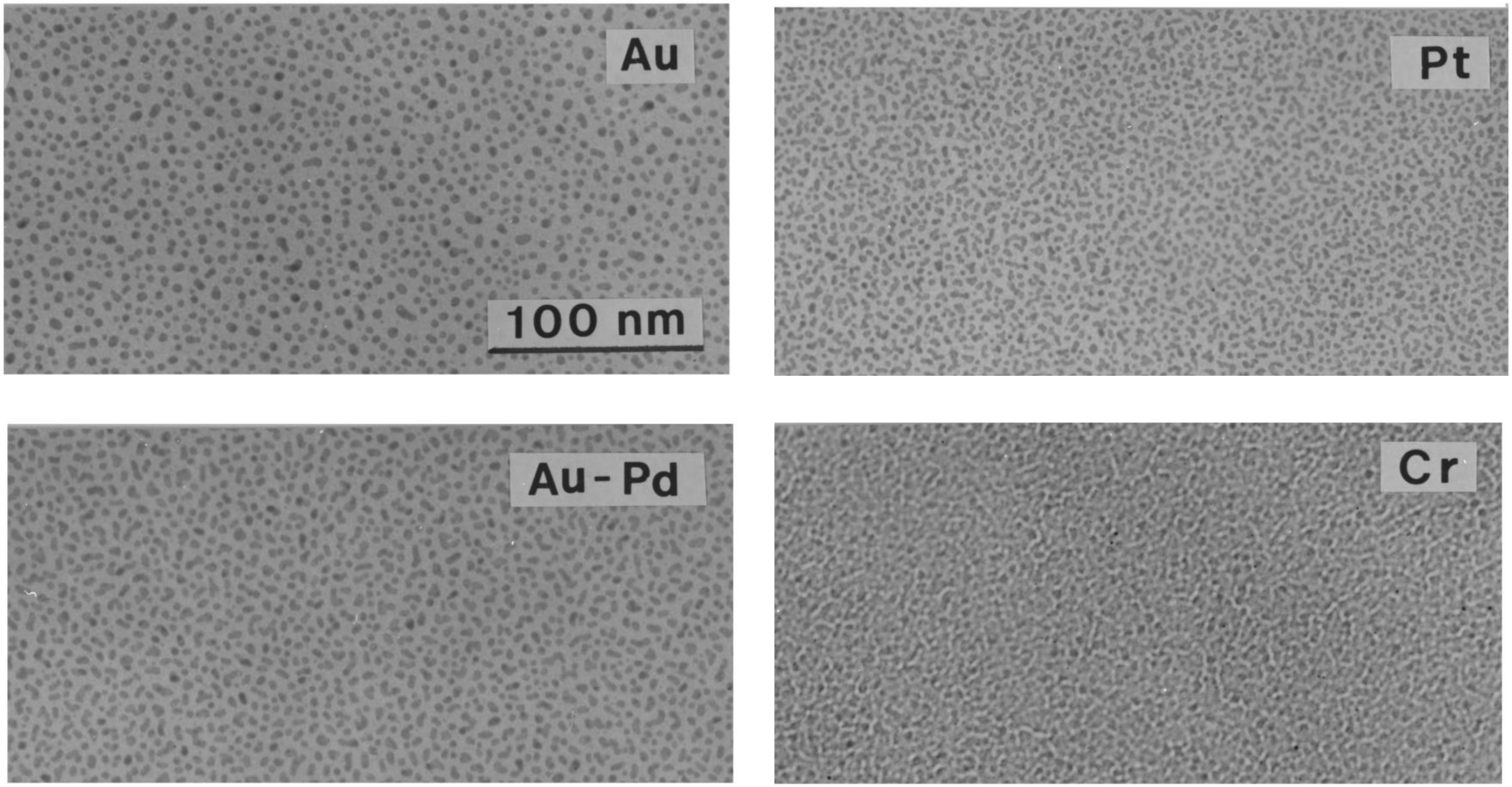
Influence of the Coating Material to the Quality



0.4 nm nominal film thickness (magnetron sputter) coatings produced at room temperature at low vacuum conditions

TEM coating

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Take home information

- Before SEM characterization
  - Check recent published papers
- Preparation of bulk (soft and hard) material
  - Think what you want to image (surface, fractures,…) and prepare the sample
- Preparation of powders
  - Make sure that there 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
  - Choose the coating depending on the imaging and sample, for BSE use carbon, for biological specimens 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

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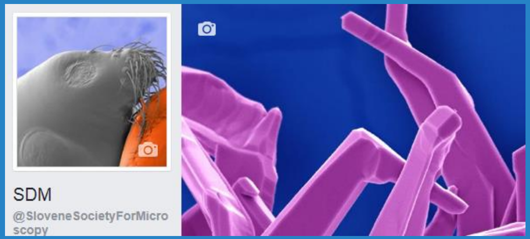

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