Transmission electron forward scatter diffraction (t-EFSD)

- Is based on the forward-dominance of low energy-los scattering.
- Detect and analyse transmitted electrons that have been coherently forward scattered
- Electrons that diffract near the top surface cannot maintain coherence for a significant distance in the specimen
- The most important Kikuchi scattering occurs near the exit surface

The **quality of the pattern** depends on both specimen thickness and Acc. Voltage

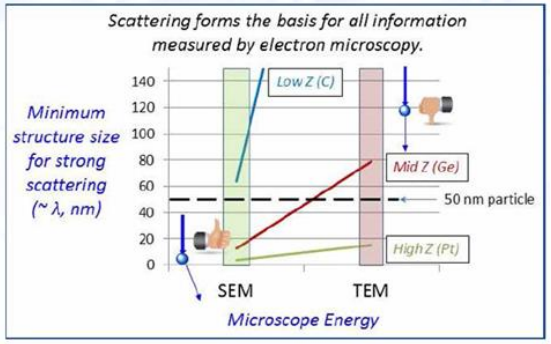
- Thicker samples pattern may also overlapping (Optimum between 75-200nm)
- ↓ KV - improve pattern in thinner areas, (maybe difficulties in indexing due to broader bands)

Angular resolution

- Standard conditions 0.5-1° but it can be as good 0.003° (strain measurements)
- In thin samples one must keep in mind the artefacts originating from the bending of the thin foil samples
- Because EBSD covers a large enough solid angle including a large number of zone axes, the angular resolution is independent of the crystal orientation.

Scattering mean free path (λ)= f(Z,E)

λ↑ with E^2 and ↓ with Z^2



When the size of the sample ↓ the λ must be decreases ↓ → go to lower energy will be beneficial (most relevant for mid and high atomic number nanoparticles).

Physical resolution = 6-8 nm
Effective resolution 2 nm (Nickel), ~10 nm (Al)

Specimen tilt

↓ tilt → weaker band contrast in the upper part of the detector → difficult for automated indexing → Solution move the detector up

↑ tilt → electron beam path in the specimen becomes longer → resulting in increasing beam spreading and poor resolution

Resolution depends on:

- Probe size – spatial resolution is in the order of the probe size
- Sample thickness – there is a range that works better (Ni:75-200nm; t<400/ ρ)
- Acceleration voltage – will depend on sample thickness and on Z
- Tilt angle – resolution in x and y direction and beam spread (also related to the pattern centre ↔ quality of indexing)
- Detector distance – compromise between large field of view and pattern shift accuracy (closer detector = zoom out)
- Working distance – shorter are better, but pattern centre outside the screen can cause indexing problems
- Atomic number Z – Contrary to s-EBSD, increasing Z increases beam broadening for a constant thickness and KV, and decrease resolution
- Number of grain through thickness– Some indications that does not affect much (???)

Better resolution

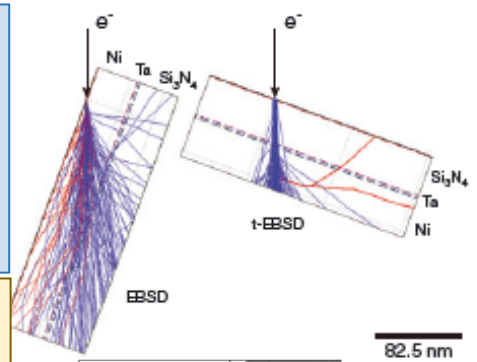
- Forward scattering is favoured→ many electron my Kikuchi scatter near exit surface
- Forward scattered beam scatter through small angles.
  - Little beam spreading in thin specimens
  - Many high energy electrons reach the exit surface
  - Interaction volume is smaller

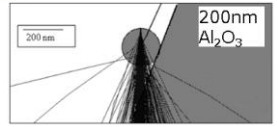
Parameters:

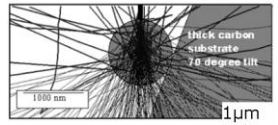
- Shorter detector sample distance (but not too short otherwise the camera resolution is not enough)
- Minimum tilt to avoid difference resolution in x and y direction
- Higher Acc. V is recommended to get good quality orientation maps.
- In case of very think samples, lowering Acc.V will give better results

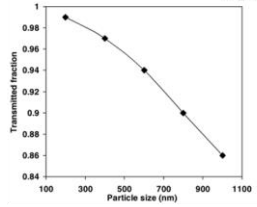
Typical parameters

- Tilt up to 30 degrees away from EBSD detector
- Short WD (pattern center on the screen) improve the lateral resolution
- Beam energy – 15-30KV
- Probe current – similar to EBSD (No study on this)
- Dwell time – similar to EBSD (our experience was a bit different)









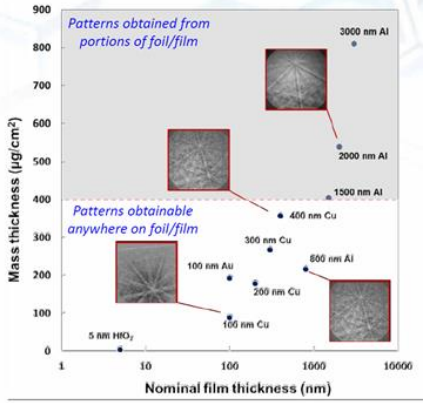
Plot of the fraction of transmitted electrons vs. Al_2O_3 particle diameter.

Mass-Thickness is a Key Factor for TKD/t-EBSD

mass-thickness ≅ density × thickness

- Describes effective scattering power of a specimen.
- Plot shows range of mass-thickness that we have so far successfully probed.
  - Note that beam spreading will degrade spatial resolution for extremely thick specimens!
- Microstructure important!
  - Multiple grains through thickness must be considered.

t<400/ρ



T-EFSD general guideline

- Use the TEM clamp holder and put your sample in one of the extremities
- Fix the clamp holder to a cross-section stub in a way that your sample is far away from the stub
- Attach the cross-section stub to the pre-tilted EBSD stage on the longest side (the one that is tilted to 54°)
- Put the stage into the microscope and pump
- Bring your sample to a working distance of 3 mm (maybe 2.5 mm)
- Tilt stage to 36° to get the sample horizontaly
- To improve orientation determination you can tilt the sample to -20 ° (stage tilt to 16 °) – we have found that this was the optimum position.
- Change in OIM data colection software under setting/super users/ enviroment the enviroment setting to a specimen tilt of -20 ° (or whatever tilt you are using)
- Use the correct calibrated WD which are:

WD OIM	WD/ tilt Stage	WD/tilt sample
21	3mm/36°	3mm/0°
22	3mm/26°	3mm/-10°
23	3mm/16°	3mm/-20°

- Further step are just like regular EBSD, but better pattern are at 30KV
- The background is very crytical for TKD, make sure you colect a background exactly from the area you are measuaring or use dynamical background substraction (take longer time)
- Remember to set the Enviroment Settings back to 70°