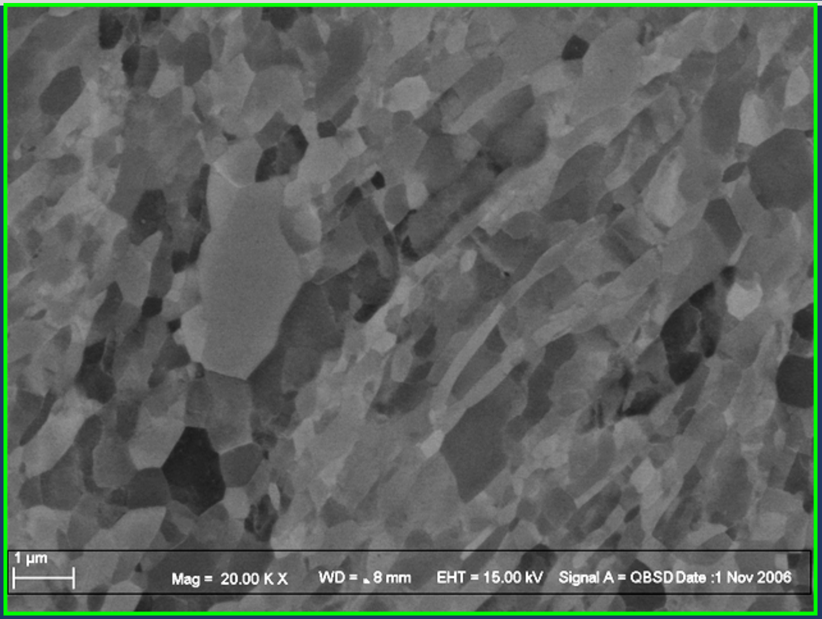
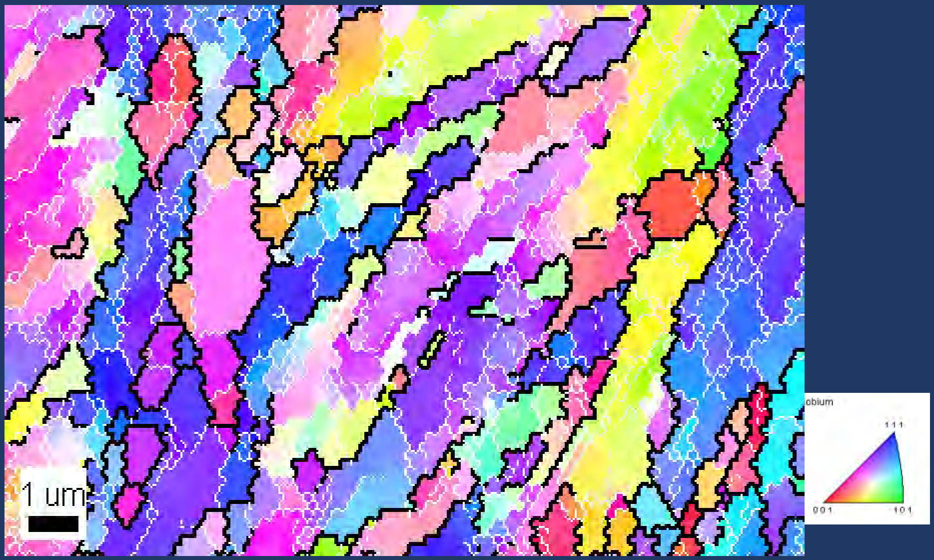
Application example -1

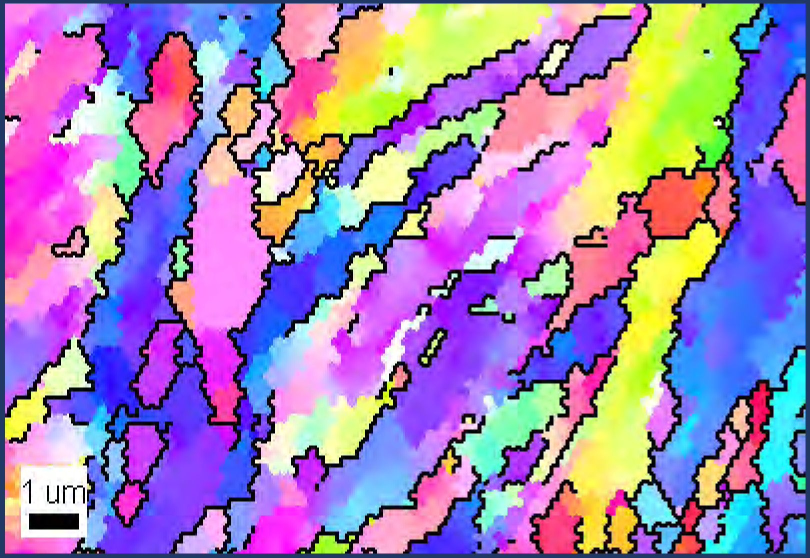




Average GS(d) =1.3µm

HAGB

LAGB



Average GS(d) =11.6µm

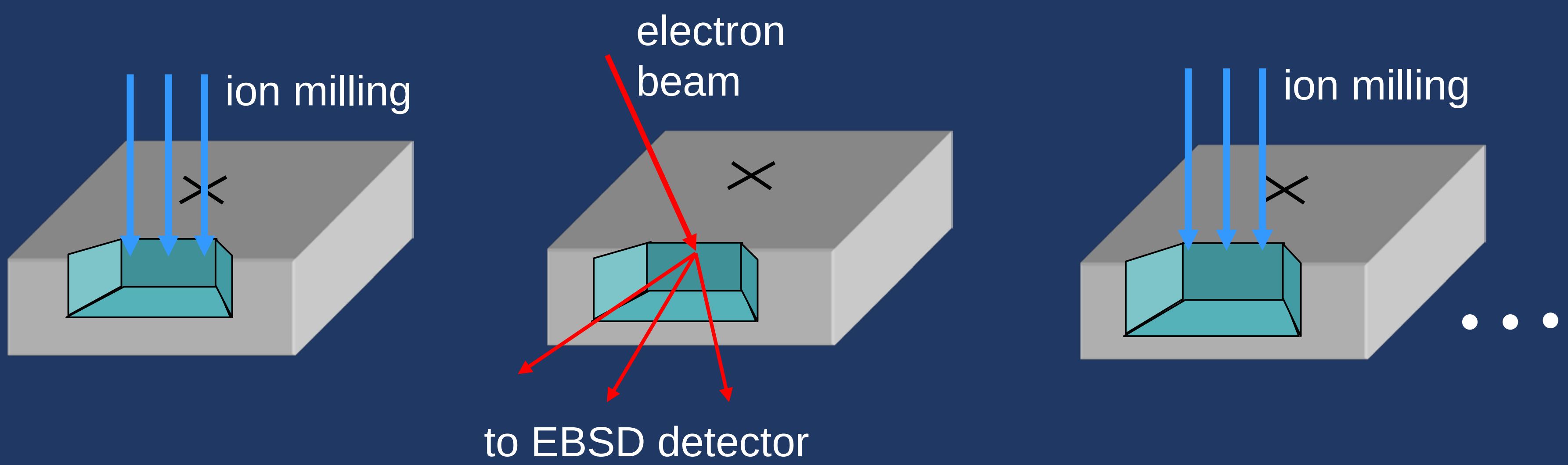
Grain boundaries have different properties depending on their rotation angle, axis and plane.

Plays a role on materials properties and performance

Common to combine EBSD with other techniques

- EBSD combine with FIB

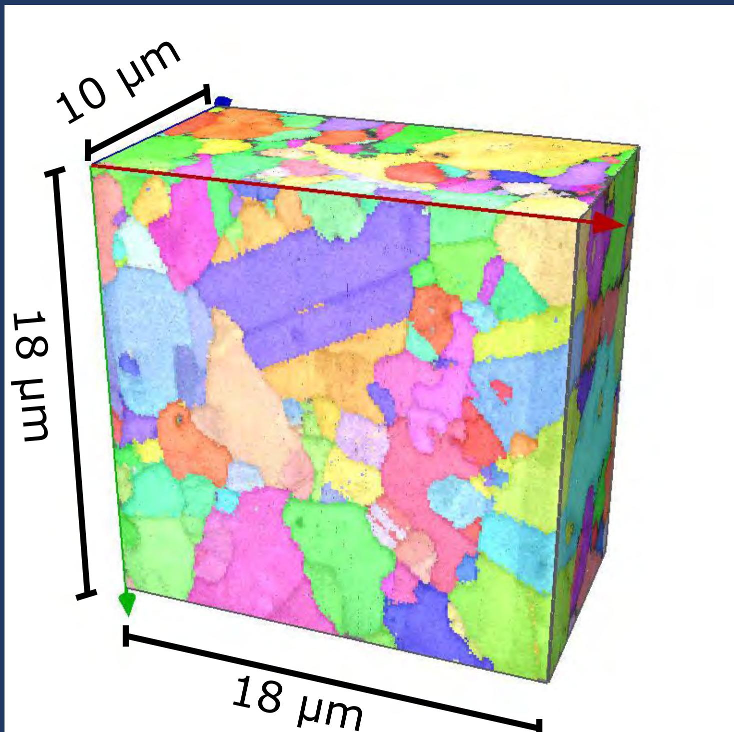
3D EBSD



Process is fully automated

Nickel super alloy

100 slices of 100 nm



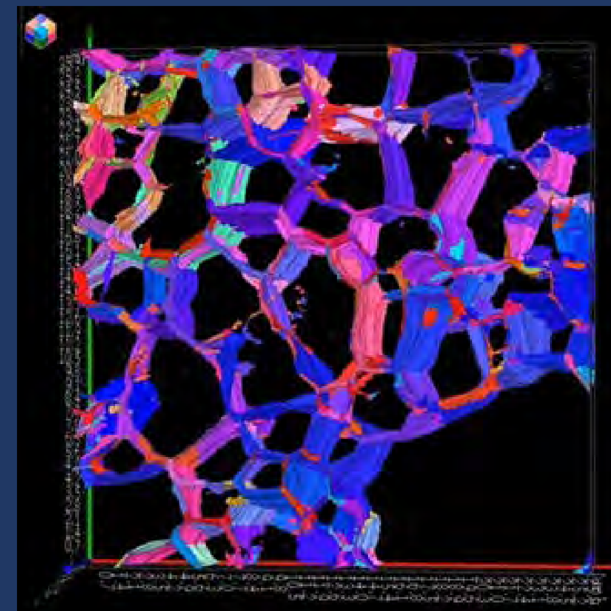
Crystal orientation // TD

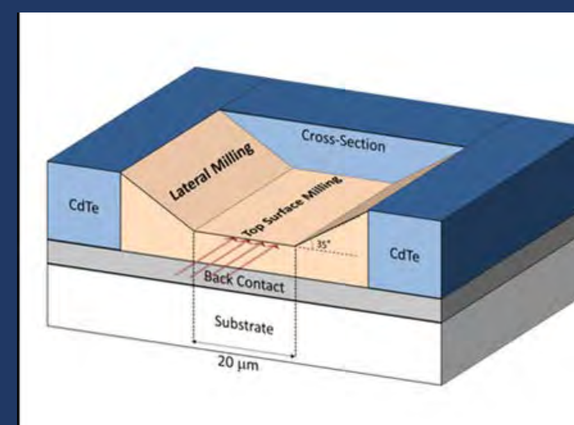
Time

EBSD map: ~ 10 min.
Slice (01): 16 min.
Slice (100): 23 min
Total of 30 hours

CdTe Solar Cells

CdTe grain boundary network reconstructed from 3D EBSD





https://www.mpie.de/3083564/Research_Project_CdTe

EBSD cannot

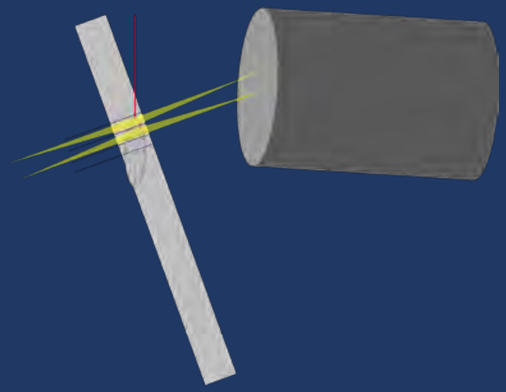
- Analyze non-crystalline (amorphous) materials
- Provide information about the crystal orientations within the volume of a sample – it is purely a surface technique
- Analyze samples with poor surface preparation or thick coats
- Discriminate between phases with similar crystallography without EDS info (e.g. Fe-FCC and Al)
- Characterize light materials like some carbon phases,
- Characterize nanocrystalline materials (GS< 50 nm)

Spatial resolution: >20-80nm, depth ~10nm (Material dependent)
Angular resolution: >0.2° (Strain mapping (cross correlation)) >0.001°

Improve spatial resolution

- Low energy EBSD (5-10KV) – challenging (low signal, long exposure time, a lot of contamination)

- Reduce the background signal by measure on thin samples (TEM samples)



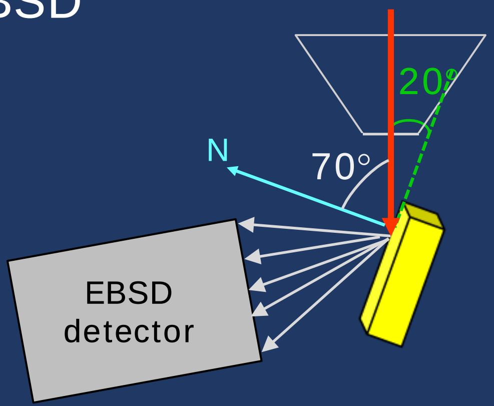
- Transmission Kikuchi Diffraction (TKD)

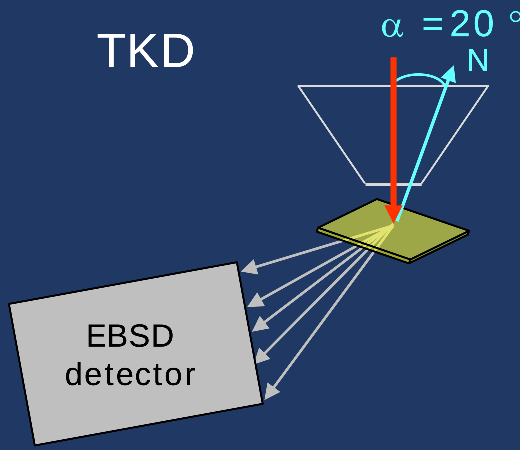
R. R. Keller & R. H. Geiss Journal of microscopy 245 in 2012

Other names:
- Transmission electron forward scatter diffraction (t-EFSD) in a SEM
- Transmission electron backscatter diffraction (t-EBSD)

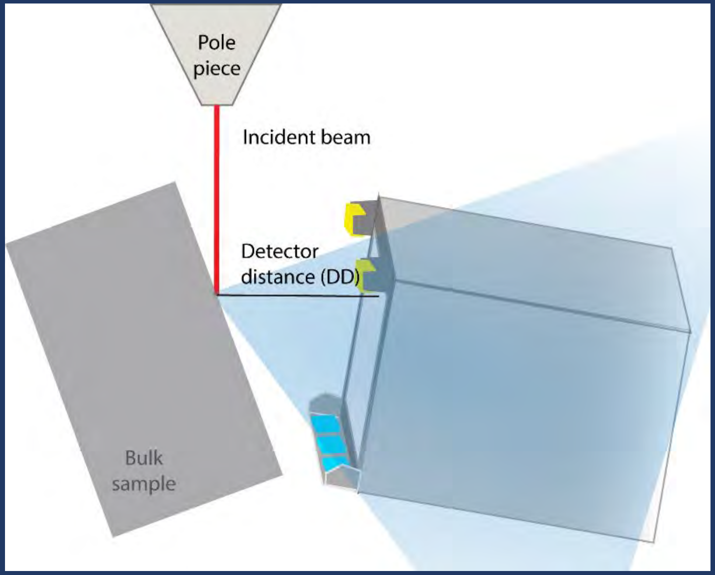
TKD
Transmission Kikuchi Diffraction

TKD - Transmission Kikuchi diffraction

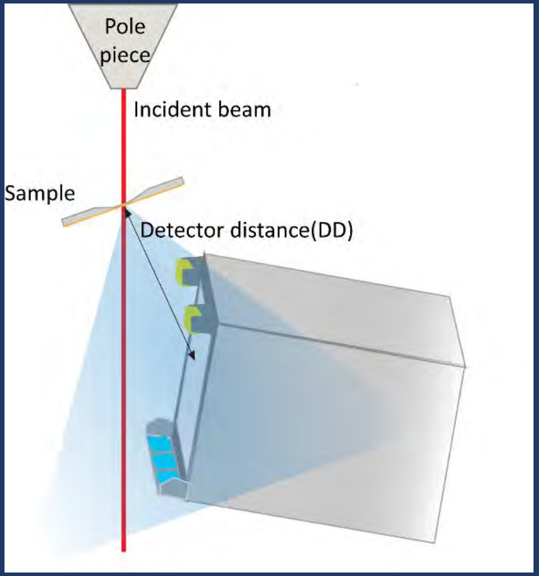




EBSD



TKD



- Tilt the sample perpendicular to the EBSD position
- Electron transparent samples

TKD - Transmission Kikuchi diffraction

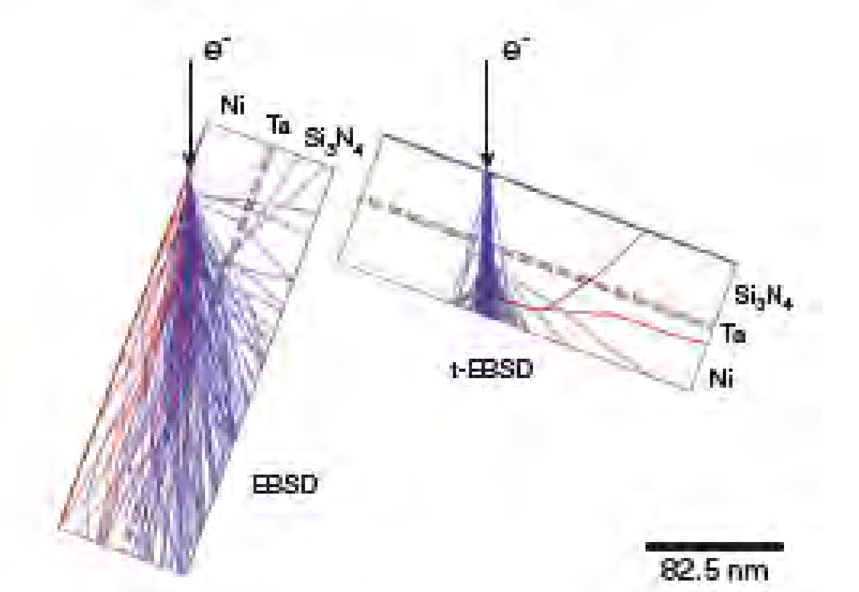


Fig. 4. Monte Carlo simulations (100 000 electrons per simulation) of scattering trajectories for 40 nm nickel/2.5 nm Ta/40 nm amorphous Si_3N_4. Beam energy = 28 keV. Incident beam direction downward in trajectory figures. Trajectories for 100 electrons each in conventional EBSD configuration (left-hand side) and t-EBSD configuration (right-hand side). Red trajectories indicate electrons backscattering out of the incident beam entrance surface. Blue trajectories indicate electrons that have either transmitted through or stopped within the specimen. Note significant difference in interaction volumes.

MC-Assumption that elastic scattering dominates

- Red trajectories indicate electron backscattered out of the incident beam entrance surface
- Blue trajectories indicate electrons that were either transmitted through or stopped within the specimen

- In EBSD - red lines forms the diffraction pattern
- In TKD - the blue line forms the diffraction pattern

↓

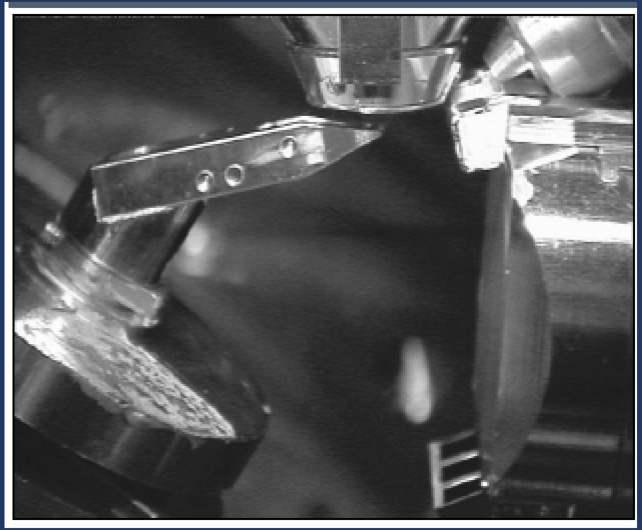
- Forward scattering is favoured→ many electron may Kikuchi scatter near exit surface – stronger signal
- Difference in interaction volume (little beam spreading) – improved resolution

Keller, Journal of Microscopy, Vol. 245, Pt 3 2012, pp. 245–251

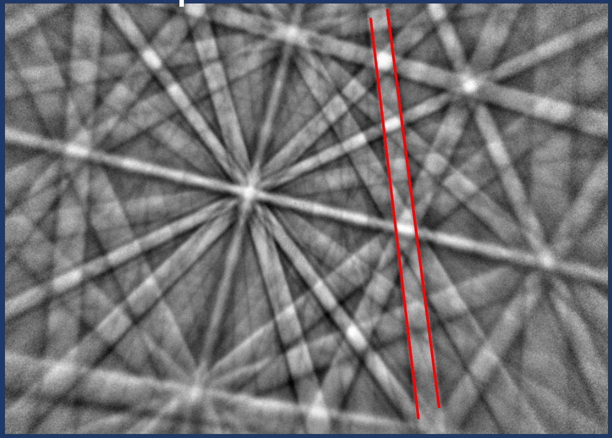
EBSD x TKD patterns

EBSD

TKD

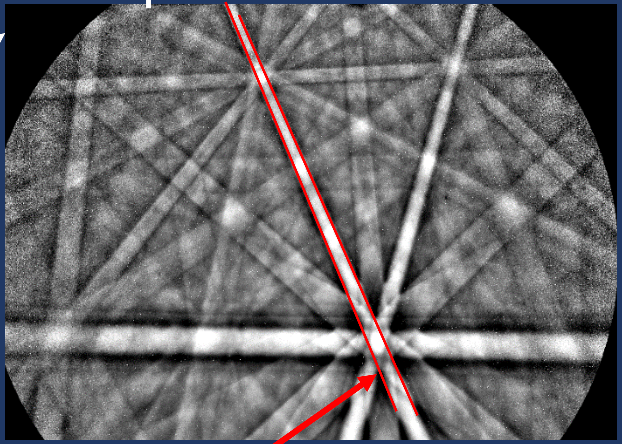


EBSD pattern



Non-symmetric intensity distribution

TKD pattern



Pattern center is sometimes outside the screen – patterns are wider at the lower part of the screen

Some differences to EBSD

EBSD	TKD
• To improve resolution - reduce electron voltage	• The higher the voltage the better the resolution – due to small beam spread (depends on material and sample thickness)
• Information from the topmost surface (10nm)	• Information from the bottommost surface
• Signal is strong dependent on surface quality	• Signal is strong dependent on sample thickness
• Spatial resolution down to **20-80** nm (material dependent)	• Spatial resolution down to **2-20** nm (material dependent)

Nanocrystalline materials can now be investigated in the SEM by OIM

In 2016 a new development in TKD – On axis TKD

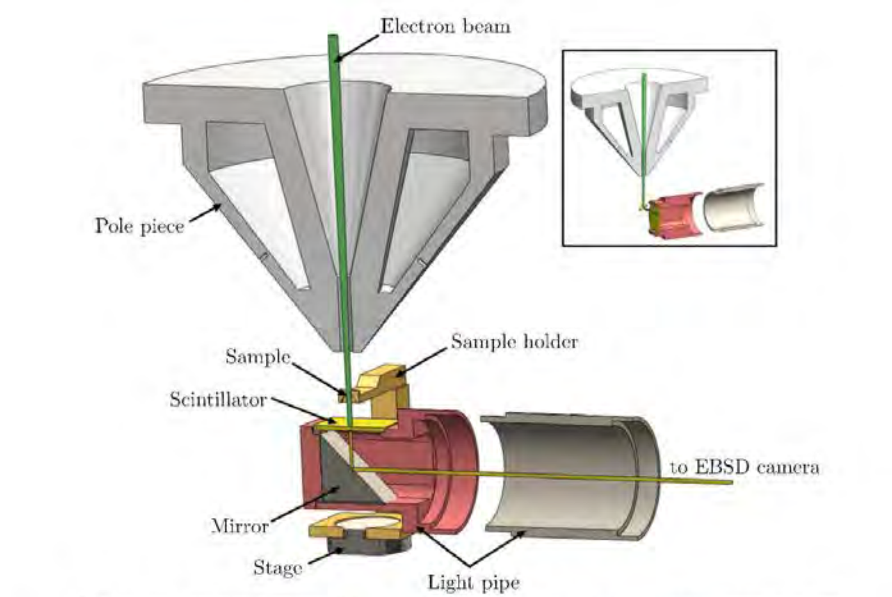
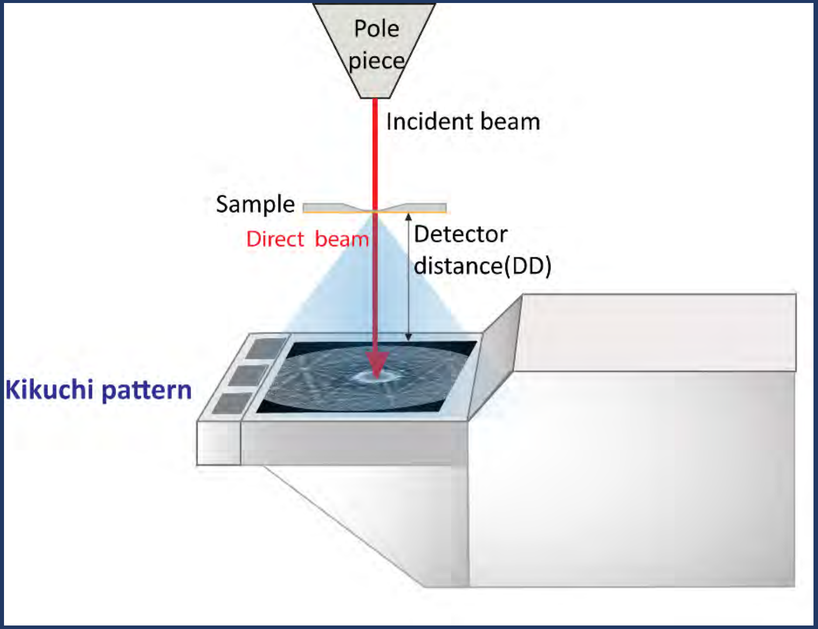


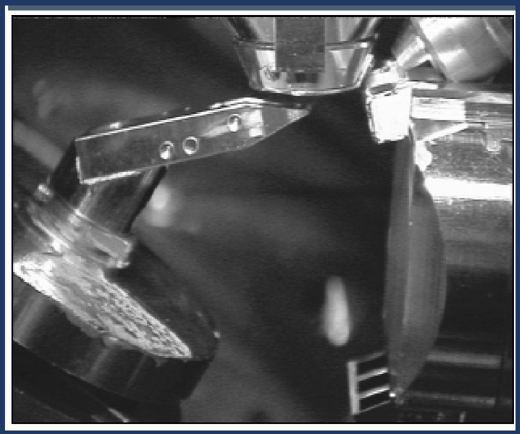
Fig. 1. Schematic illustration of the new TKD configuration. The conventional configuration of [11–13] is shown in the inset.

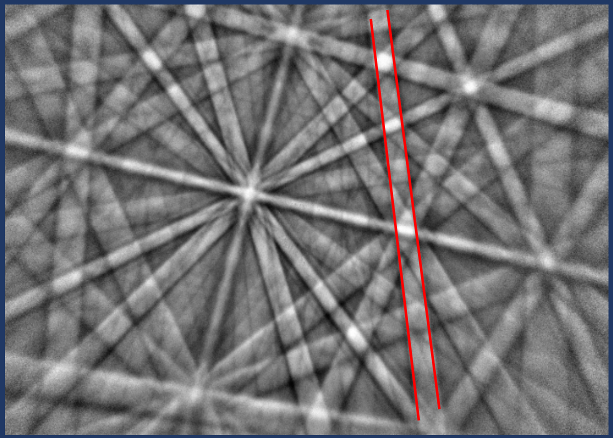


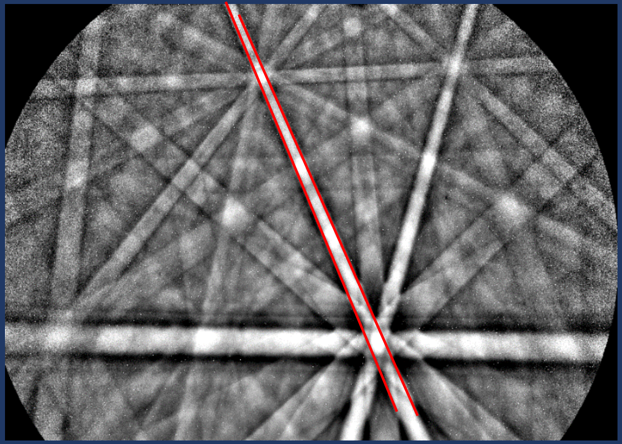
OPTIMUS Detector (Bruker GmbH)

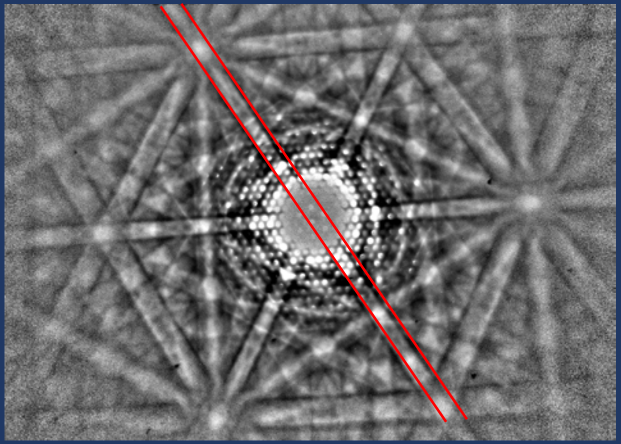
Fundenberger et al. Orientation mapping by transmission-SEM with an on-axis detector. Ultramicroscopy 2016

EBSD pattern | TKD pattern off-axis | TKD pattern on-axis









+ No distortion
+ Homogeneous intensity distribution
+ Optimized geometry

Optimized geometry – on-axis TKD

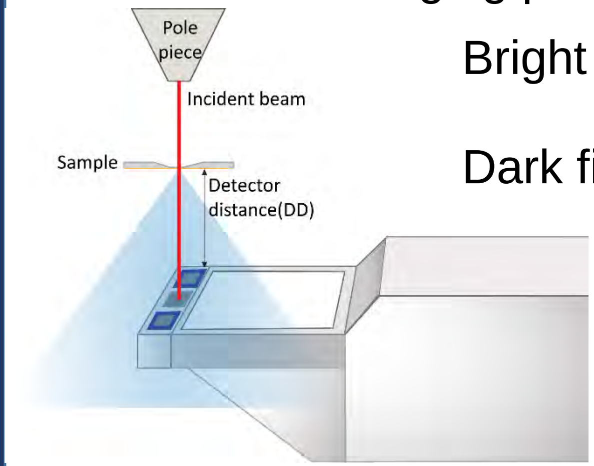
- More signal
  - Faster measurement – less problems (charging, drift, contamination, instrument availability …)
  - Lower beam current – better spatial resolution
  - Better for beam sensitive materials
- Combination of TKD measurement with STEM images (scanning transmission electron images)

Measuring position

Imaging position

Bright field (pseudo bright field) image -BF

Dark field image - DF



~20 mm

Direction of movement between positions

F. Niessen, A, Ultramicroscopy. 186 (2018) 158–170.

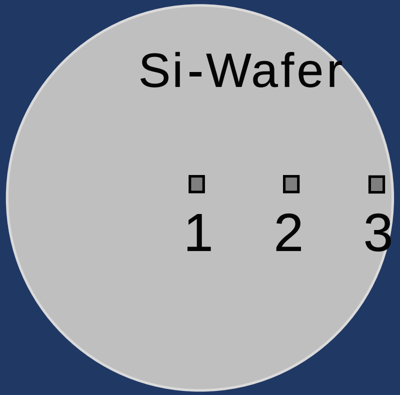
Application examples -1

Collaboration with DTU Photonic

- Develop a catalyst-free method to growth InP nanowires with controlled crystal structure by MOVPE (Metal-organic vapour epitaxy)
- Traditionally crystal structure is investigated by TEM (some few NW).

Statistical characterization of III/V nanowires grown on Si by TKD

Investigated nanowires from 3 positions.

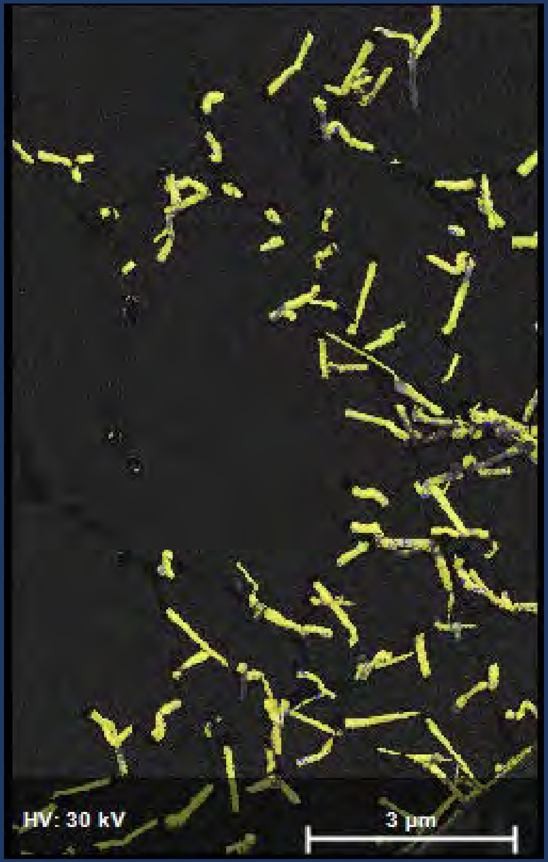


2 possible phases:

- Zinkblende (cubic)
- Wurtizite (hexagonal)

Fast crystal structure control for nanowires growth

Step size 15 nm 8hrs



Step size 100 nm 19 min

More than 500 NW were investigated

Position	Fraction of Cubic NW
1	97%
2	98%
3	98%

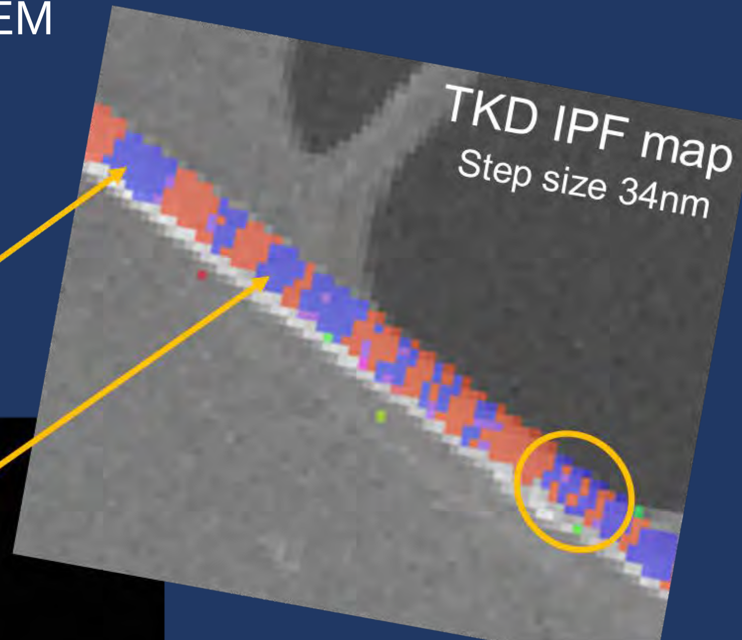
Cubic phase
Hexagonal phase

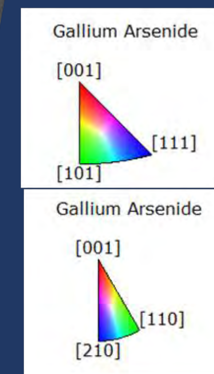
CONCLUSION: Growth process optimization and structure control of nanowires is fast and easily performed with TKD.

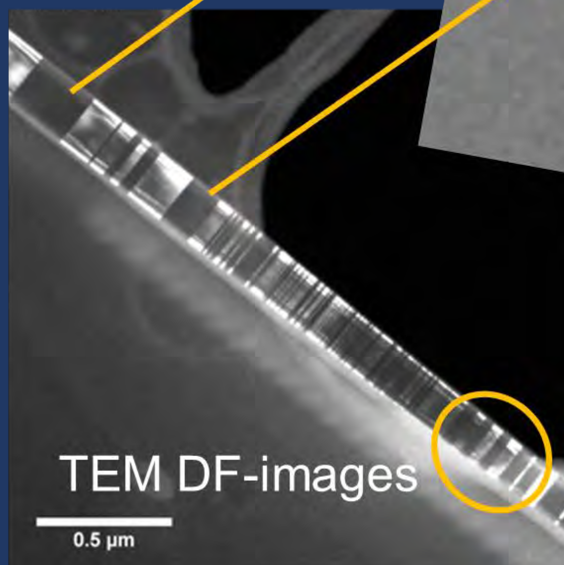
However software developments to allow feature identification are required to further optimize the characterization.

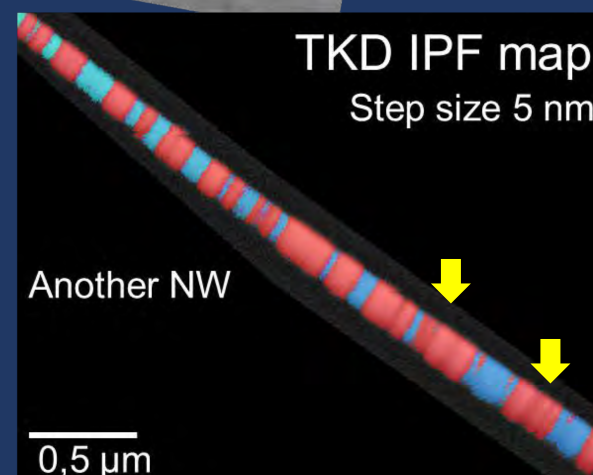
Further microstructure details

Resolution is not as good as in TEM



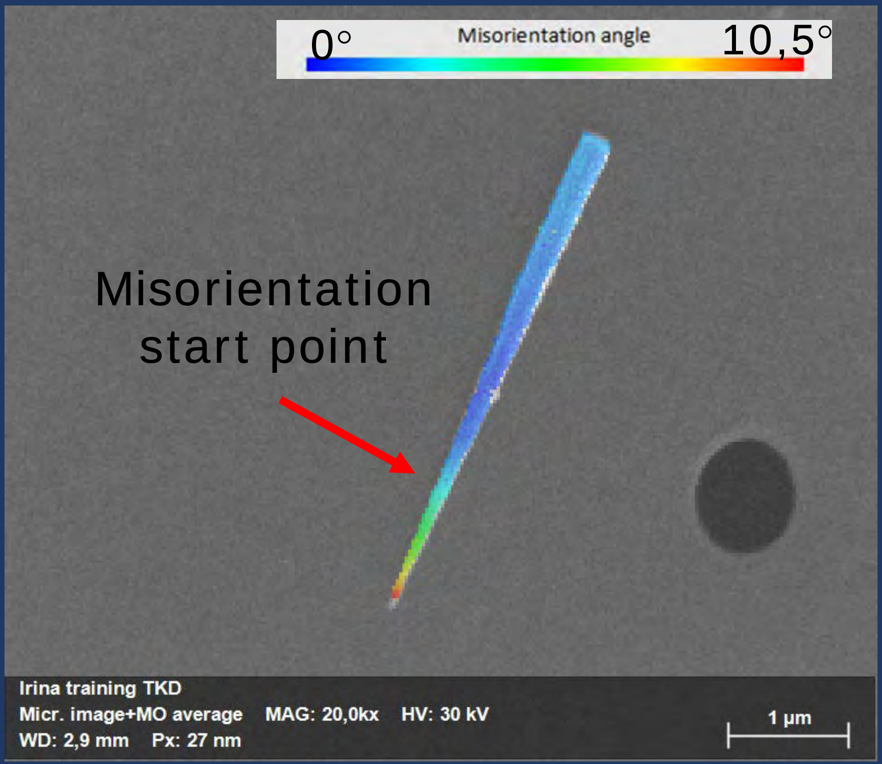


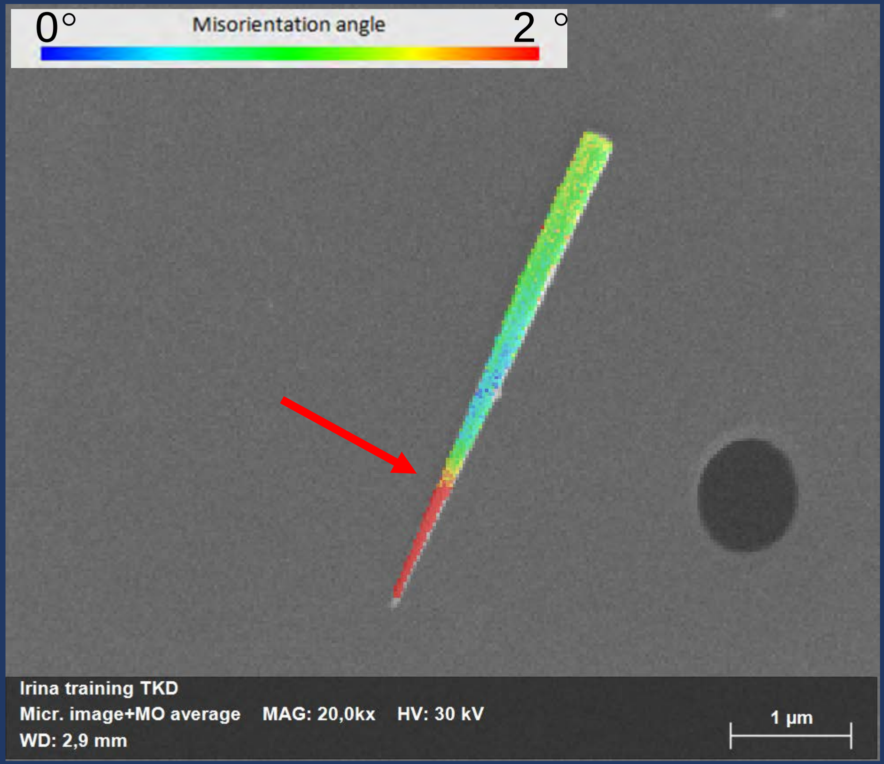




Doped GaAs-NW: Single hexagonal phase

Misorientation within a doped GaAs-NW (WZ structured)





An Easy way to verify presence of dopant

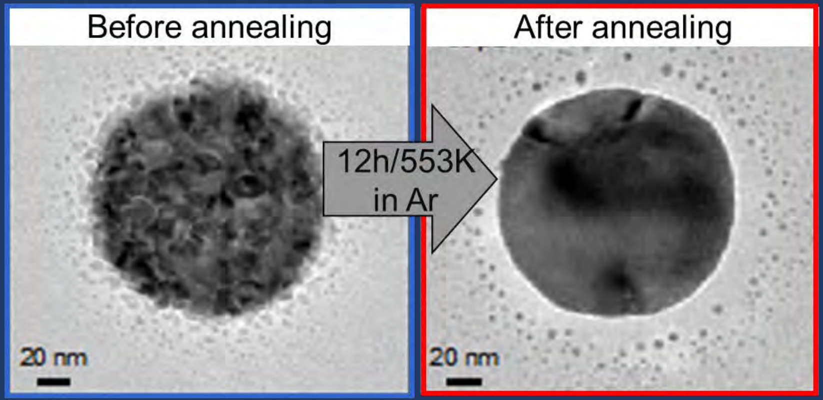
Application examples - 2

Collaboration with Chalmers University of technology

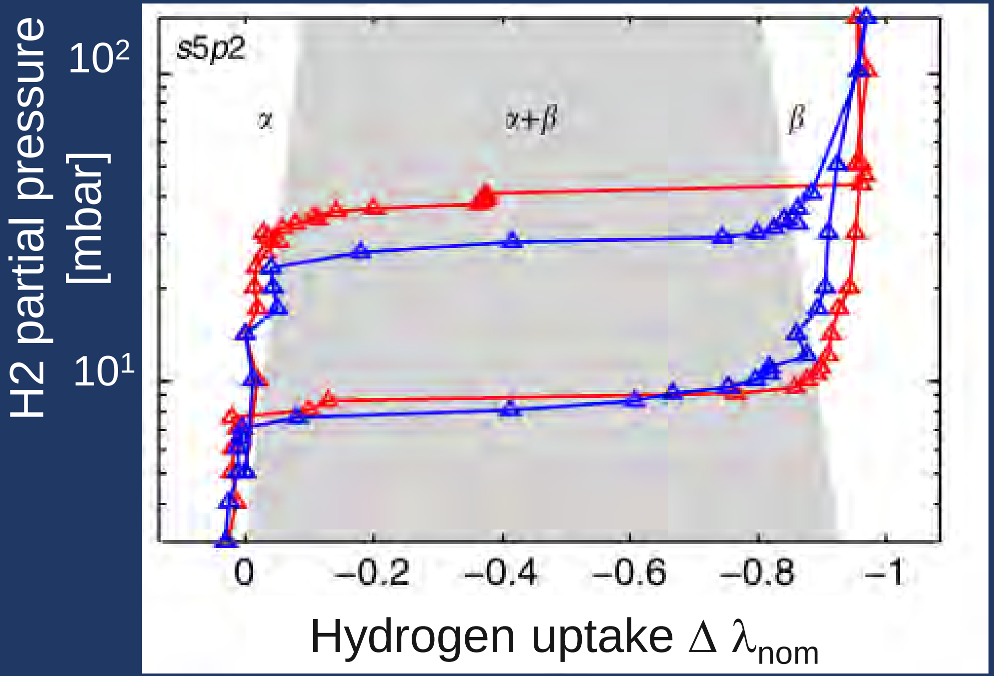
MOTIVATION:

- Hydride formation pressure is microstructure sensitive.
- Understand the role of microstructure on hydrogenation in individual nanoparticles

TEM – LSPR- Pd nanoparticle



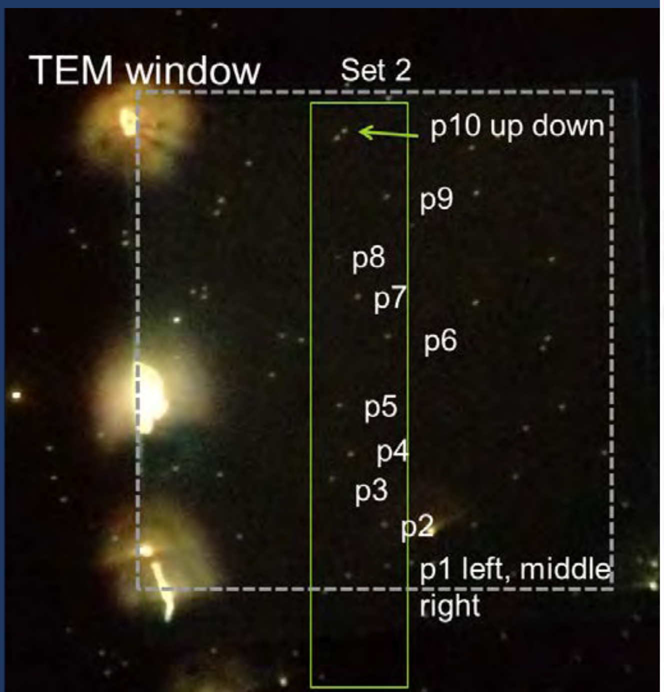
Before annealing After annealing



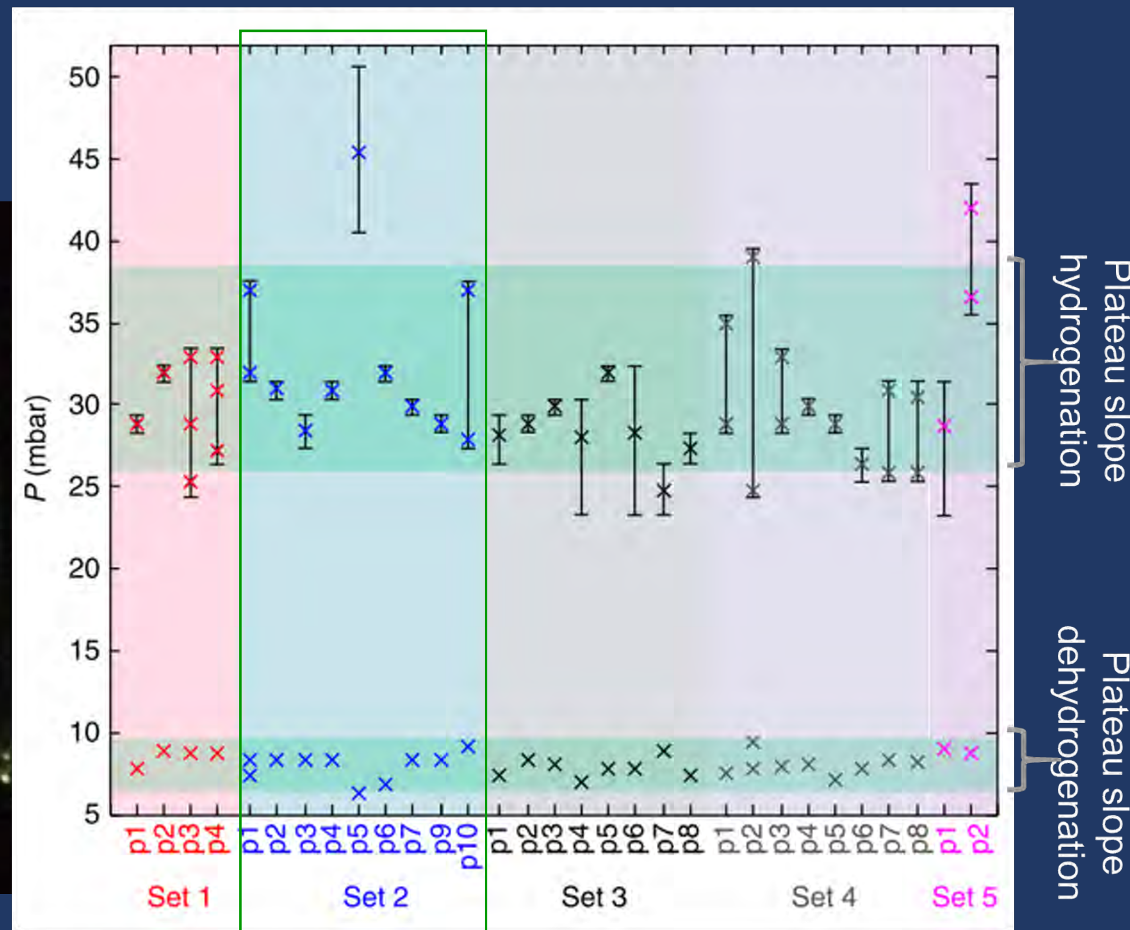
S. Alekseeva, Nat. Commun. 8 (2017).

Multichannel single particle plasmonic nanospectroscopy

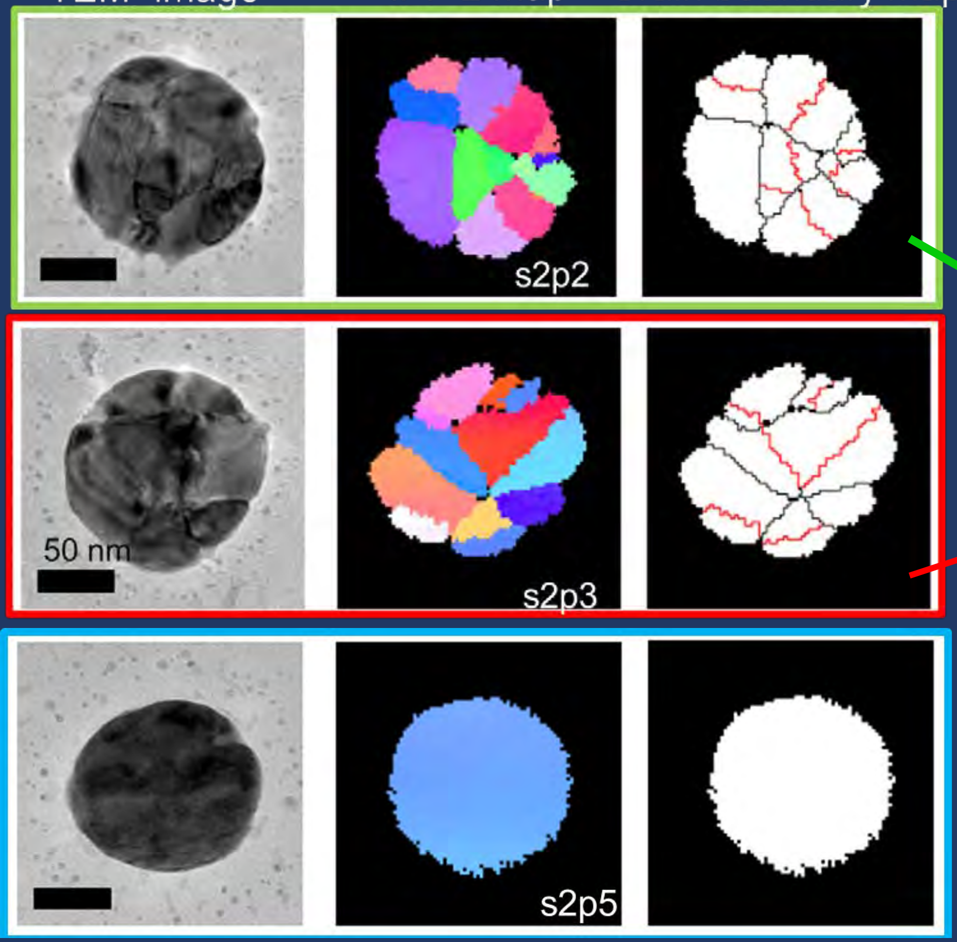
- Measurement of individual response from up to 10 particles simultaneously.

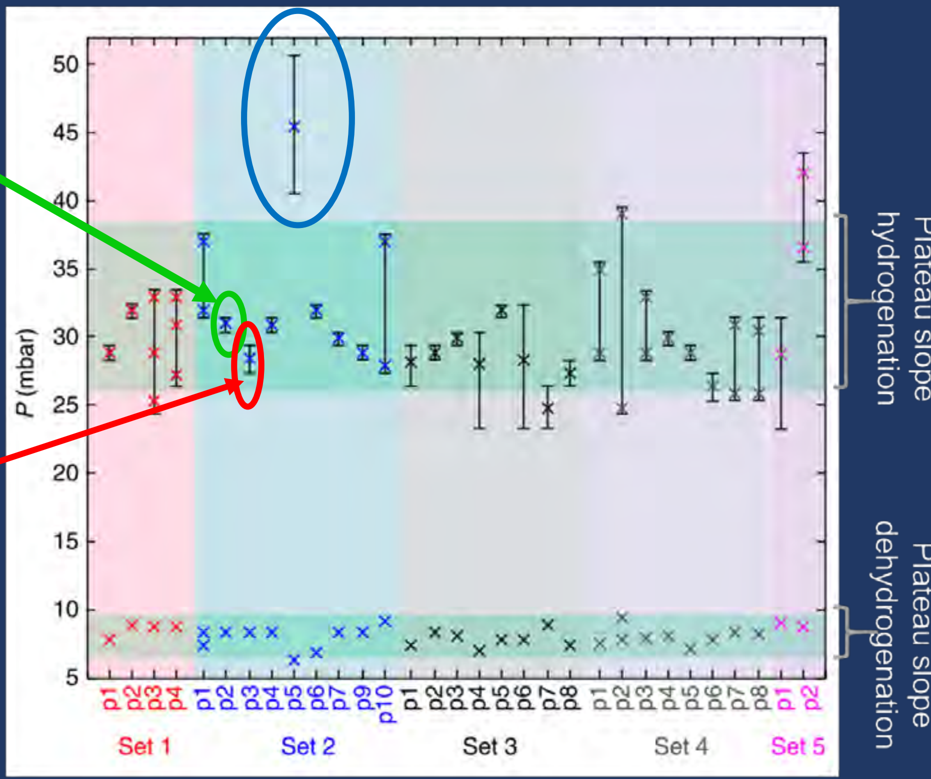


(De) hydrogenation pressures of single Pd nanoparticlesc



TKD characterization of individual nanoparticles





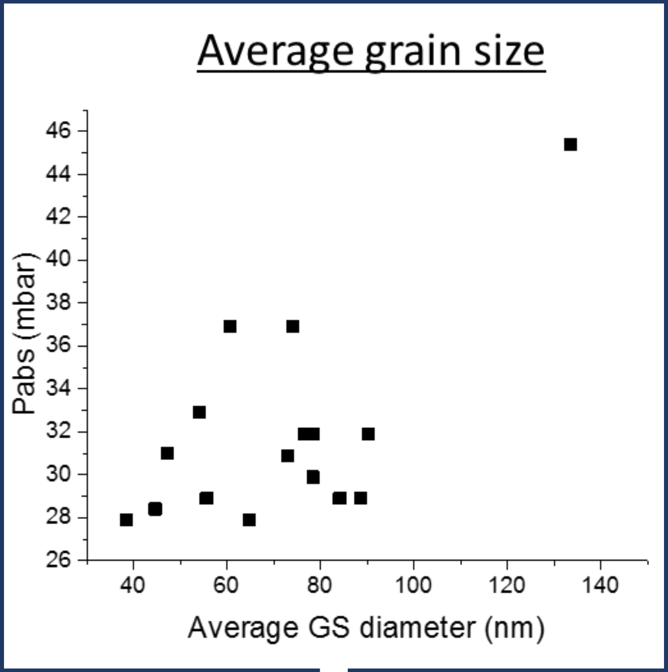
HAGB

Twin Boundaries

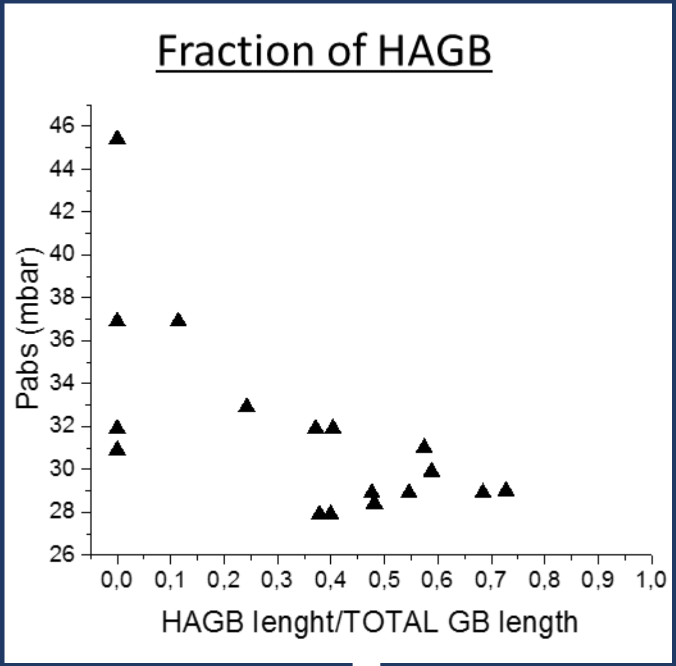
Measurement time/particle ~ 1 min (step size of 3nm)

TKD characterization of individual nanoparticles

Extracting quantitative descriptors of the microstructure and correlating these with the information obtained from the plasmonic nanospectroscopy, shows a clear correlation between grain size and grain boundary length with hydrogenation pressure.



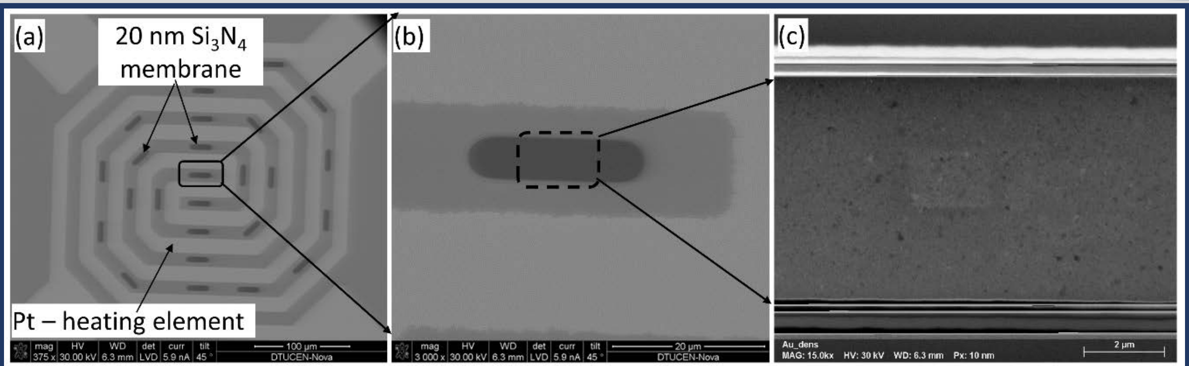
Smallest the grain size the lower the hydrogenation pressure



By distinguishing between the grain boundary types, it becomes evident that HAGBs are the main contributor to the decrease in hydrogenation pressure.

S. Alekseeva, Nat. Commun. 8 (2017).

In situ TKD



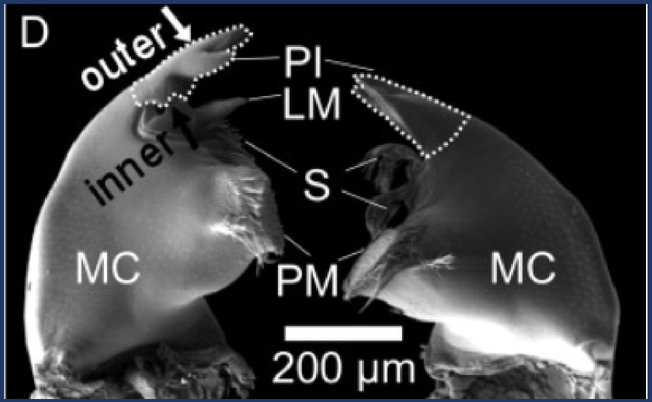
Solid state dewetting of 20nm Au film

A.B. Fanta et al. Mater. Charact. 139 (2018) 452–462.

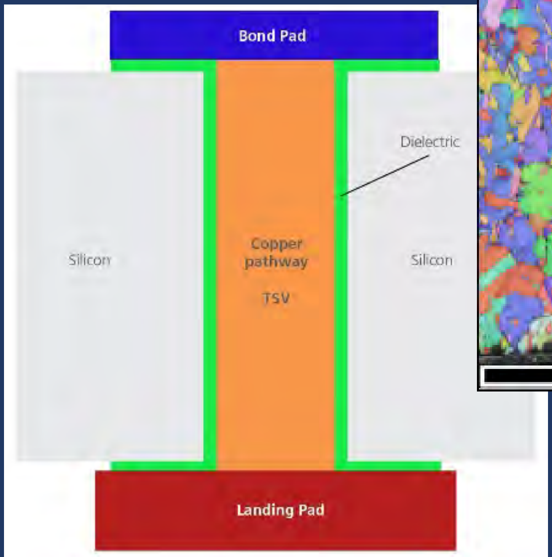
Many other areas of applications:

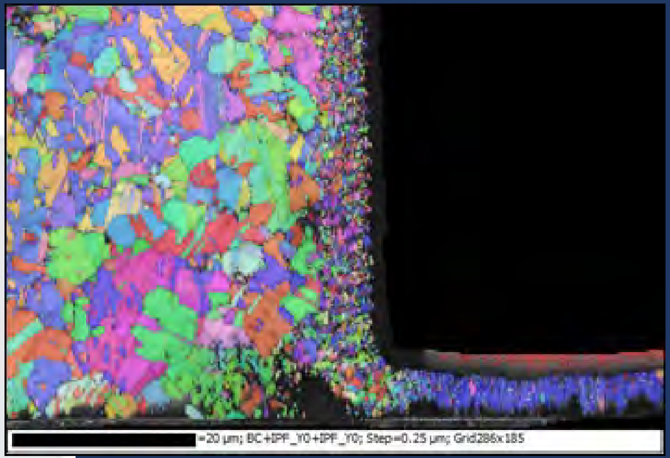
brachiopod shells

crustacean cuticle

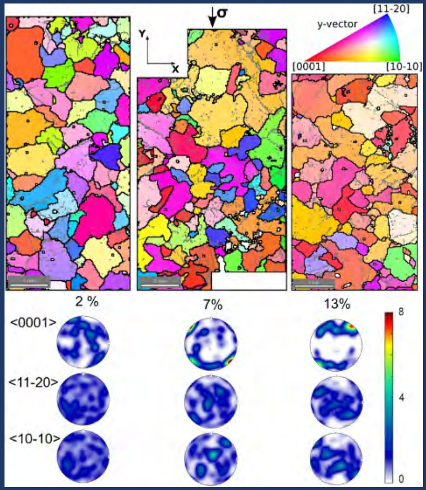


Through-Silicon Vias





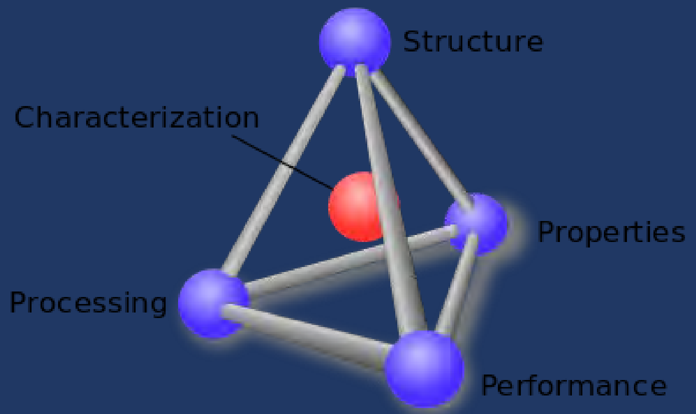
Ice



Front. Earth Sci., 17 December 2015

Final take home message

- Both EBSD and TKD are very powerful technique to characterize crystalline material in the SEM.
- Getting good pattern is not always easy (material, sample, thickness and surface quality dependent)
- Once you can get good pattern from your sample, the measurement runs fast and automatically.
- You get full crystallographic orientation from every point in the scanning area
- The quantitative data can be investigated in different ways to understand the microstructure and correlate it to:
  - the properties of the material
  - or to processes used to produce this material
- It becomes even more powerful when combined with other techniques, like:



Wikipedia

+ EDX or WDX
+ EBIC
+ CL (**C**athode **L**uminescence)
+ FIB (**F**ocused **I**on **B**eam)
+ In-situ heating, deformation, etc

Thank you for your attention

STEM images of dewetting Au film acquire with the new on-axis TKD detector developed at DTU-Nanolab in collaboration with Bruker GmbH.