Characterization of nanomaterials and nanostructures in the SEM using On-Axis TKD technique

Dr. Alice Bastos da Silva Fanta
Dr. Daniel Goran

Outline

01 Spatial resolution: On-Axis TKD vs. EBSD vs. Conventional TKD

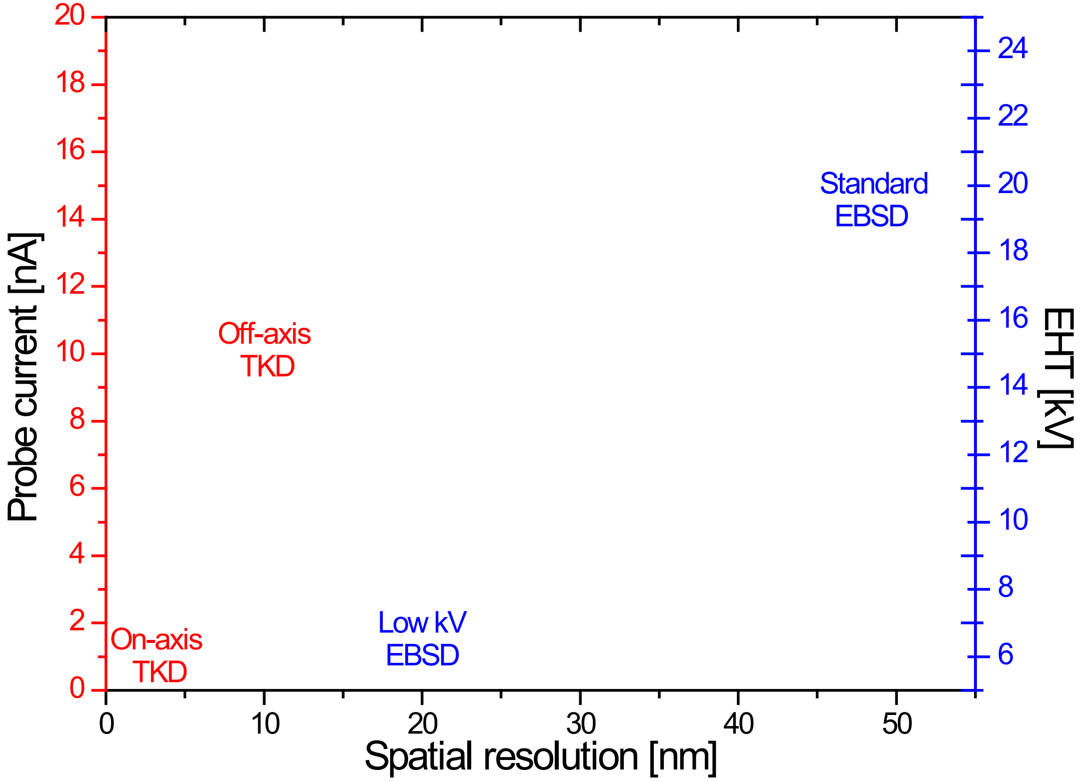
02 Augmented On-Axis TKD in SEM – capabilities and benefits

03 *Invited speaker: Dr. Alice Bastos da Silva Fanta*

04 Q&A Session

Spatial resolution
EBSD vs. conventional TKD vs. On-Axis TKD

- **Physical** Spatial Resolution (PSR) is set by the interaction volume
- **Effective** Spatial Resolution (ESR) is given by the SW ability to correctly index patterns produced by multiple crystals

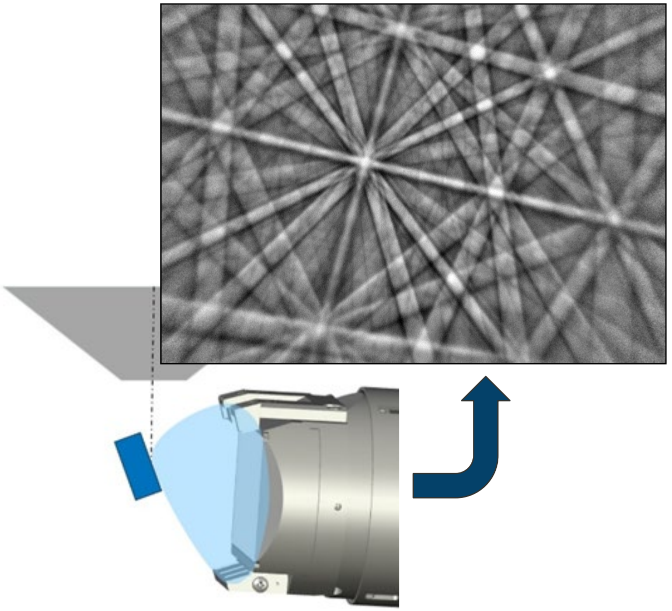


Effective spatial resolution values:

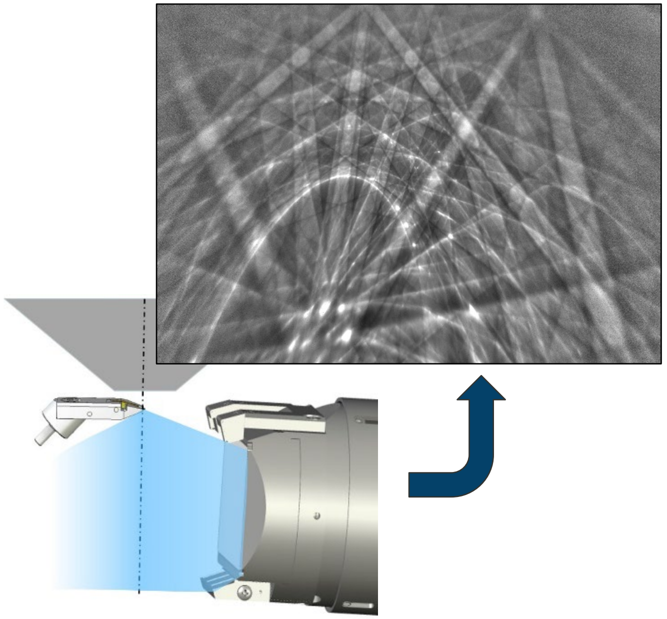
- EBSD at 20kV: down to ~50nm
- Low-kV EBSD: down to ~20nm
- Conventional TKD, a.k.a. t-EBSD: down to ~8nm
- On-Axis TKD: down to ~2nm

Experimental setup
EBSD vs. conventional TKD vs. On-Axis TKD

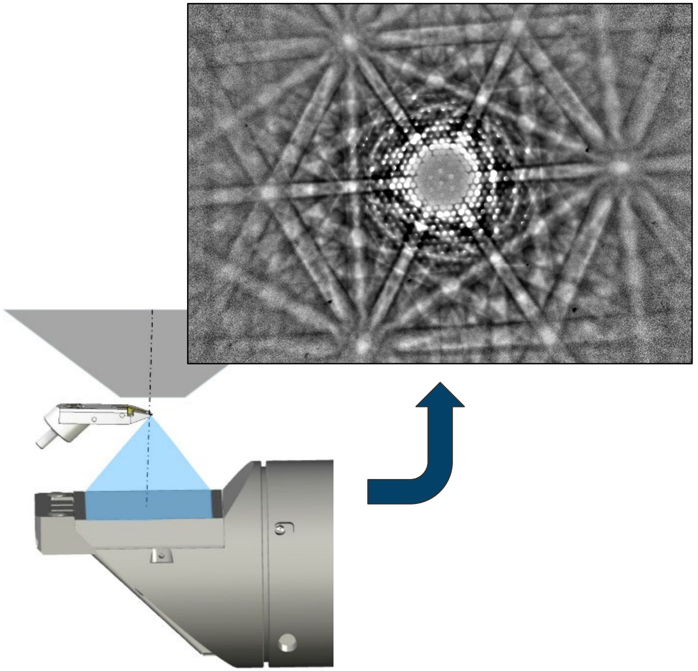
Standard EBSD



Conventional (Off-Axis) TKD



On-Axis TKD



- EBSD and standard TKD use the same hardware & software
- Non-optimum sample-detector geometry for TKD ⇔ weak signal

- Additional hardware – OPTIMUS 2
- Provides optimum sample-detector geometry for TKD ⇔ strong signal

„*Orientation mapping by transmission-SEM with an on-axis detector*" J.J. Fundenberger et all, Ultramicroscopy, 161, 17–22, 2016.

"*A systematic comparison of on-axis and off-axis transmission Kikuchi diffraction*" F. Niessen et all, Ultramicroscopy, 186, 158-170, 2018.

On-Axis TKD
“More signal, more better!” ☺…but why?

We all want to acquire orientation maps with:

1. Best spatial resolution to resolve even the finest crystals/features
2. Fastest speed possible to use lab’s resources efficiently
3. Highest indexing rate, i.e. reliable data to help us get a realistic understanding of sample’s properties

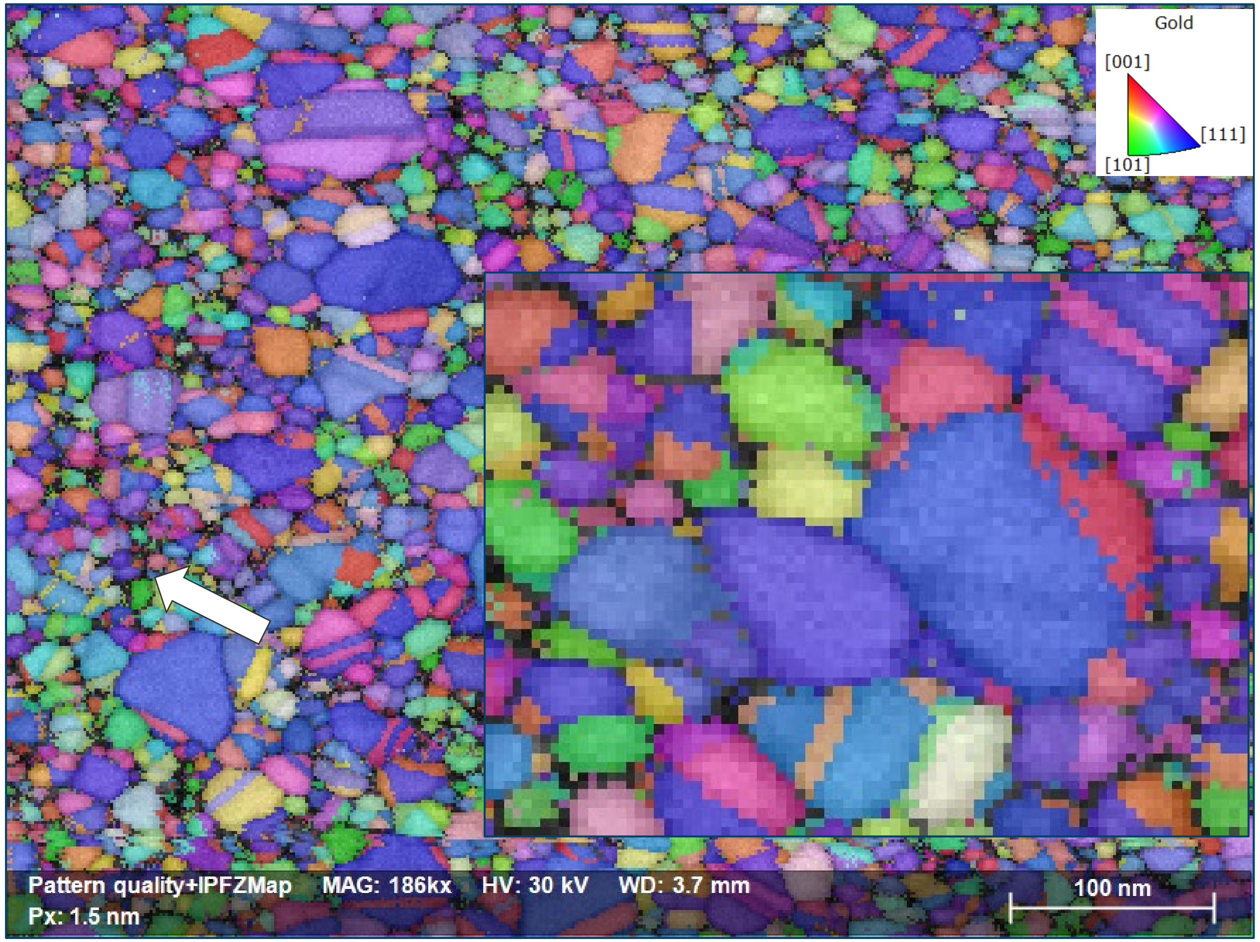
All the above is possible but it “costs” signal:

1. Signal to allow lowering the probe current (probe diameter) without damaging the pattern quality
2. Signal to compensate for the lower exposure times required by high speed pattern acquisition
3. Signal to produce low noise / high quality patterns enabling high indexing rates

On-Axis TKD
Best spatial resolution, speed and data quality

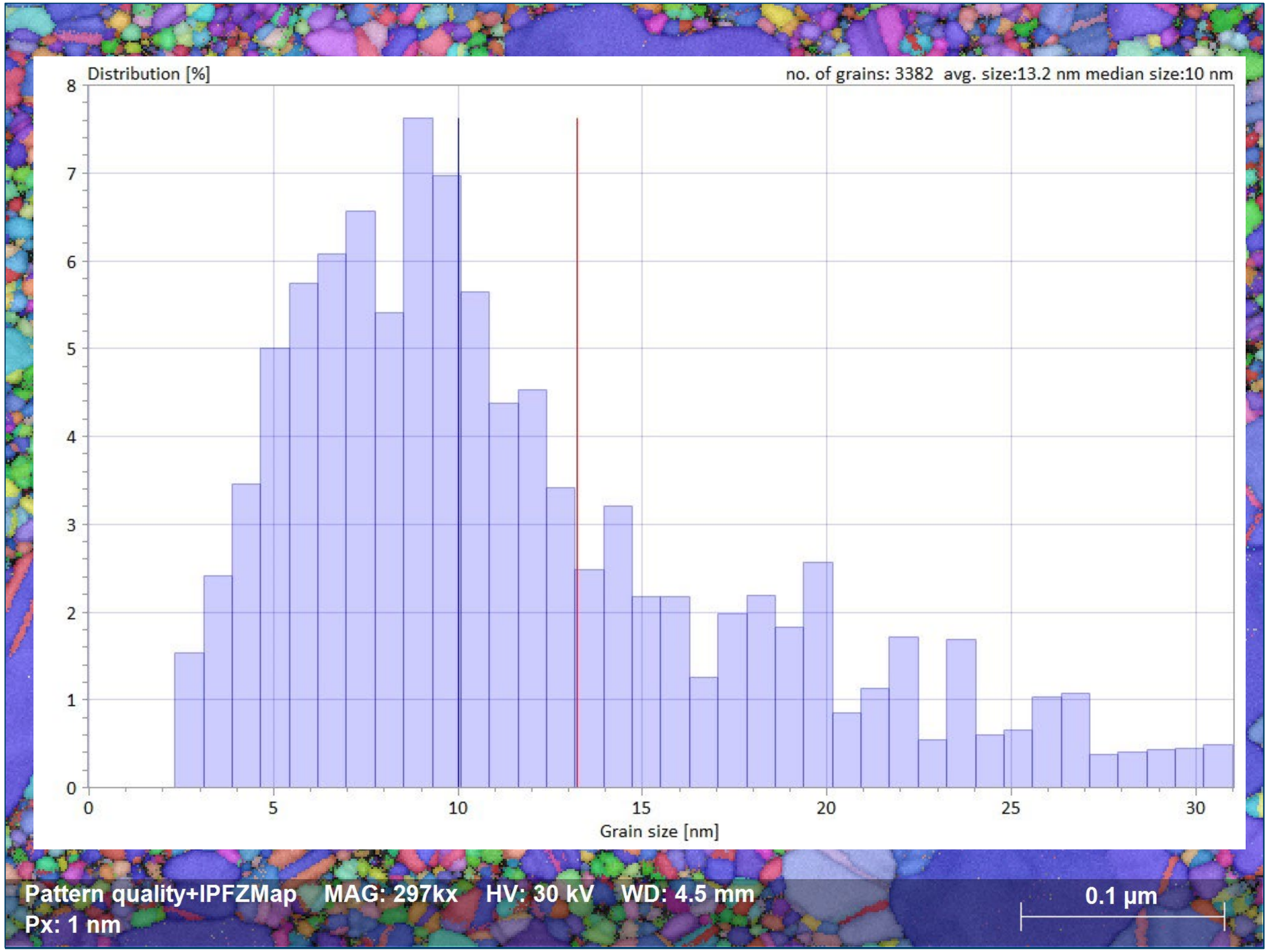
- Important parameters:
- EHT: 30kV
- Probe current: 2nA
- Step size: 1.5nm
- Mapping speed: 320fps (3ms/point)
- Total acquisition time: 6:31min
- Zero solutions: 11.5%
- Annealing twin: **~4nm wide**
- No data cleaning!

20nm Au film on 5nm Si_3N_4 membrane

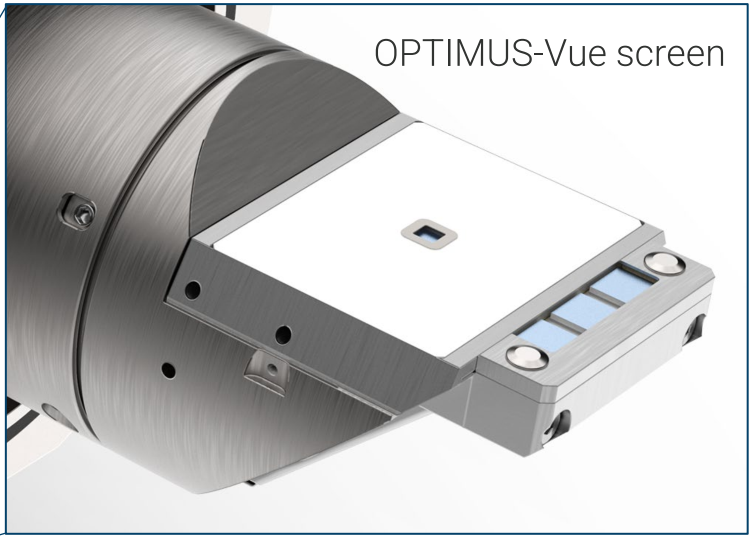


On-Axis TKD
Reliable data & great statistics

- Important parameters:
- EHT: 30kV
- Probe current: 1.5 nA
- Step size: 1nm
- Mapping speed: 243fps (4 ms/point)
- Total acquisition time: 19:15 min
- Zero solutions: 5.73 %
- No data cleaning!
- 3382 grains smaller than 33 nm with a mean equivalent diameter size of 13.2 nm

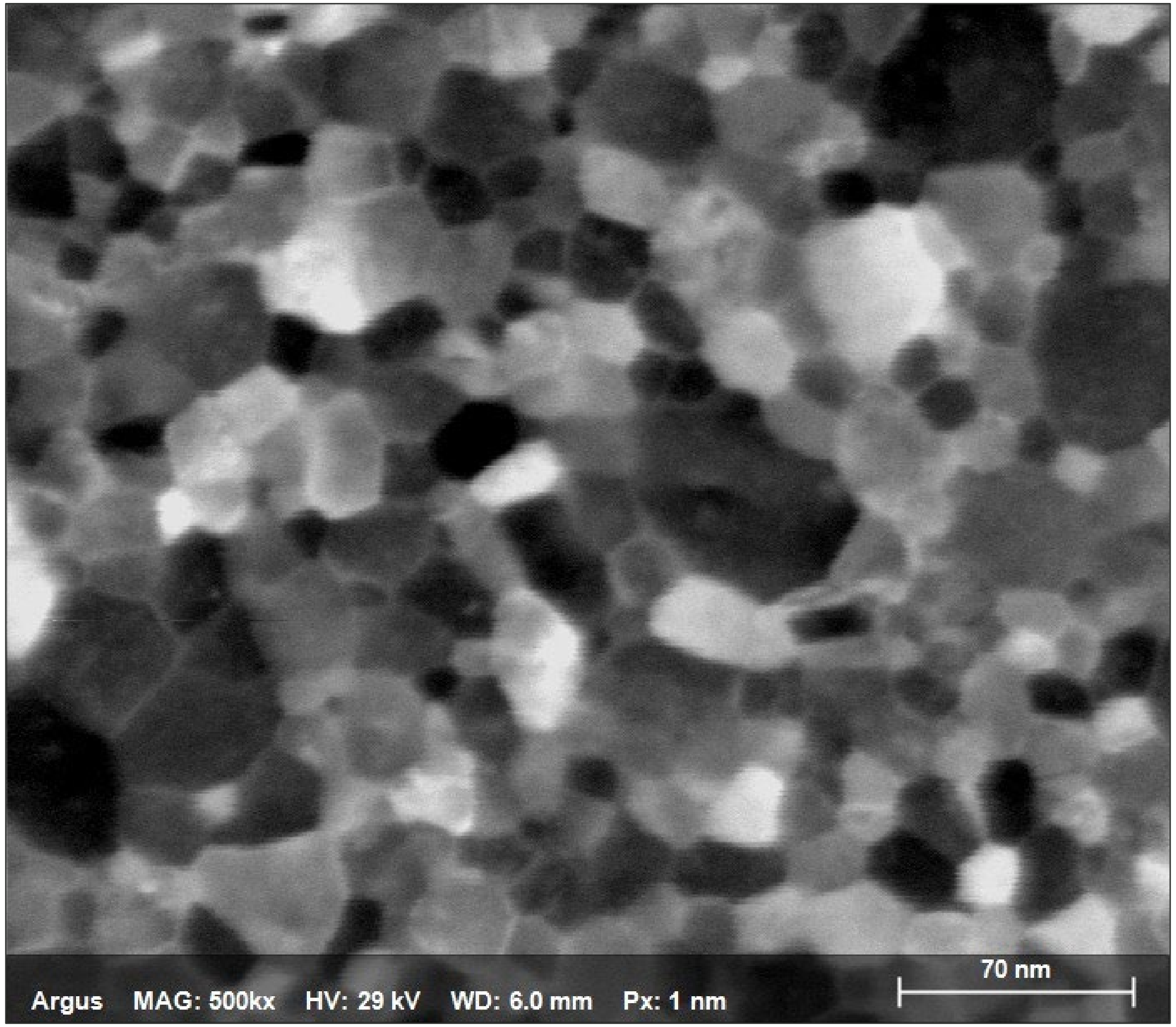


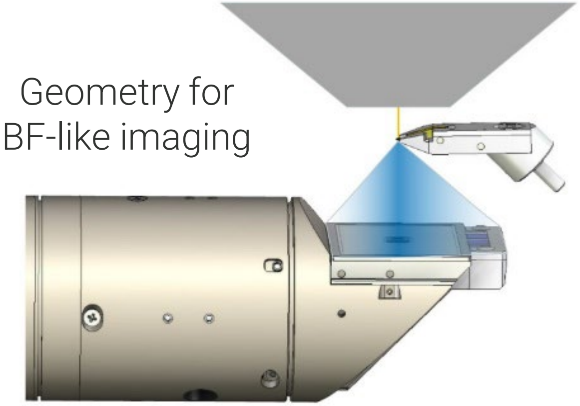
OPTIMUS 2 detector head for On-Axis TKD
Features & benefits



- **OPTIMUS-Vue screen** – BF-like imaging in mapping position (more details later)
- e- beam friendly materials – *improved spatial resolution and minimized drift*
- Additional layer in screen structure – *minimized beam interference*
- Redesigned screen frame – *easier/safer to use*

High resolution BF imaging with OPTIMUS 2
20 nm Ru film deposited on 5 nm TiN & Si wafer

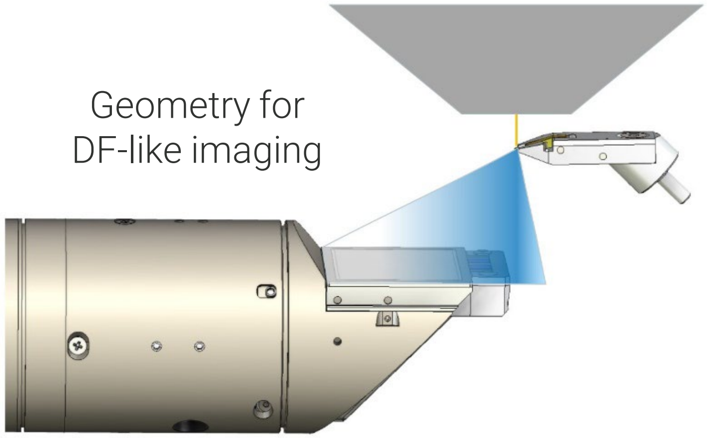




- BF image acquired with OPTIMUS on a high-end SEM equipped with immersion lens technology
- Pixel size. 0.5 nm
- Accelerating voltage: 29 kV
- Probe current: 0.4 nA
- Acquisition time: 3 seconds

High resolution DF imaging with OPTIMUS 2

15 nm Au film deposited on 10 nm Si_3N_4 membrane



High resolution DF image acquired at 30kV with 0.8nA probe current and a **pixel size of 1 nm**

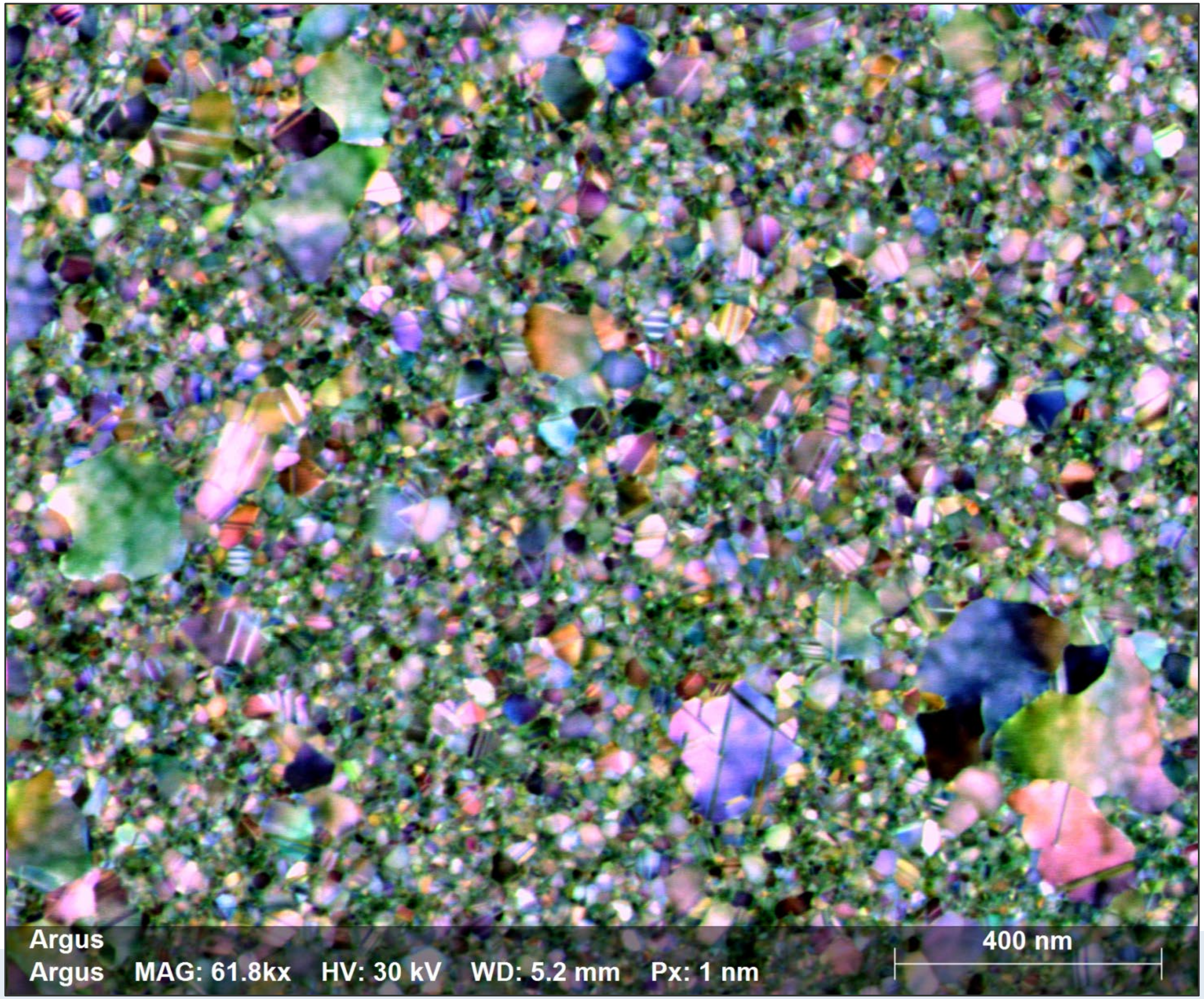
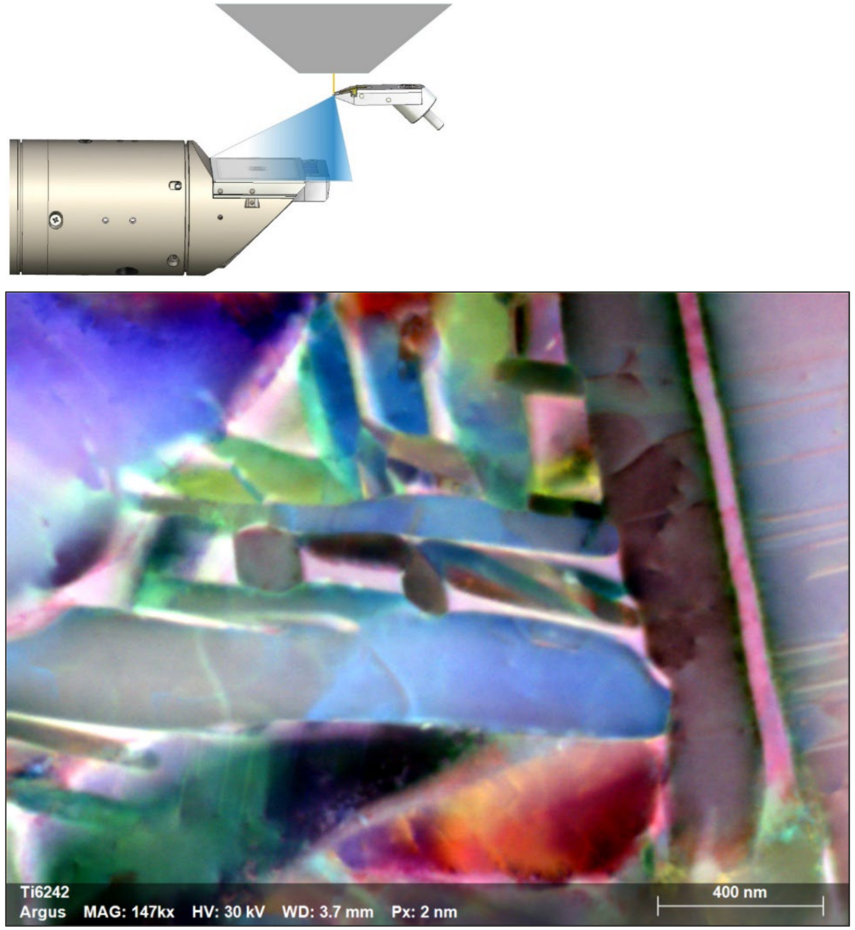


Image is courtesy of Hong Zhang from Eurofins in Santa Clara CA, USA

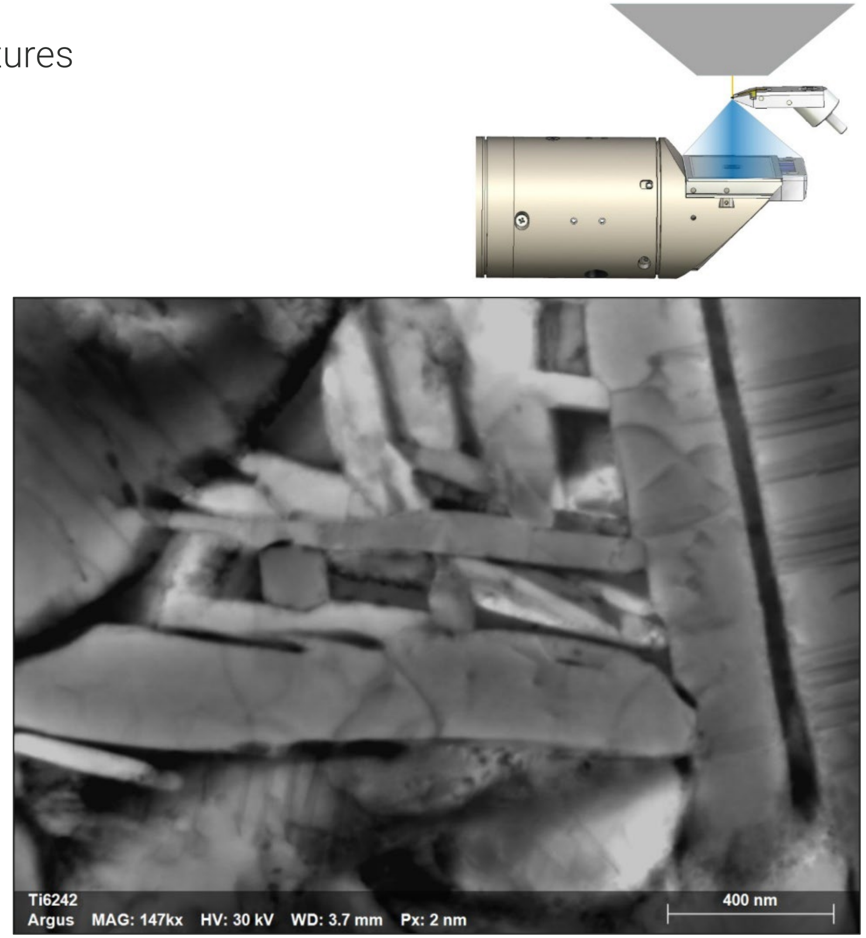
DF & BF imaging with OPTIMUS 2
Ti 6242 alloy

Applications & benefits:

- Qualitative characterization of microstructures
- Finding area/features of interest
- Refining beam focus and astigmatism
- Ideal for drift correction
- Essential for three new SW features:
    - ESPRIT FIL-TKD
    - ESPRIT TRM
    - ESPRIT MaxYield



Ti 6242 alloy

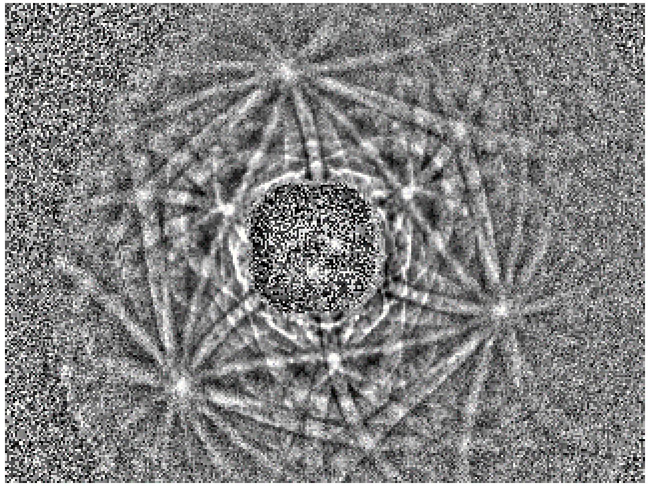


Ti 6242 alloy

Sample is courtesy of Ben Britton from The University of British Columbia, Canada (formerly with Imperial College London)

ESPRIT FIL-TKD
Enabling nano-scale TKD mapping

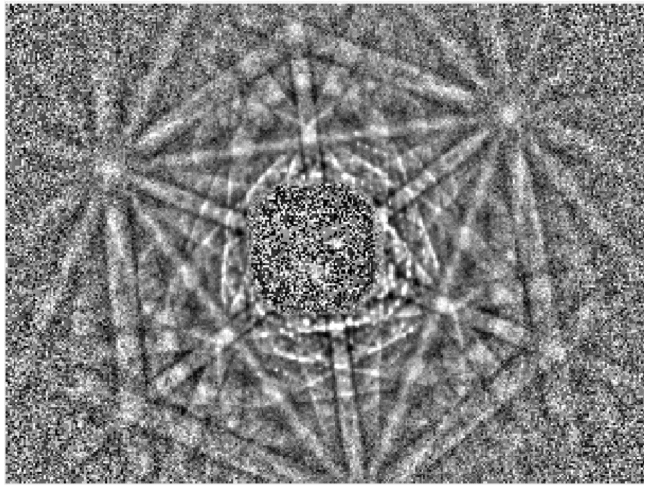
With field

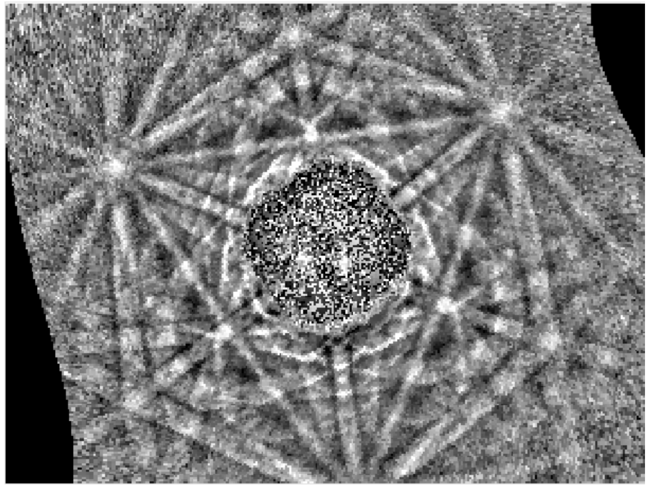


- Immersion mode ⇔ Strong Magnetic Field inside SEM chamber
  - Electrons are constrained within a narrow space around the optical axis of SEM – *limits their spread laterally*
  - Electron trajectories are affected – *distorted patterns*
- OPTIMUS 2 and ESPRIT FIL-TKD help overcome these limitations

FIL-TKD

No field



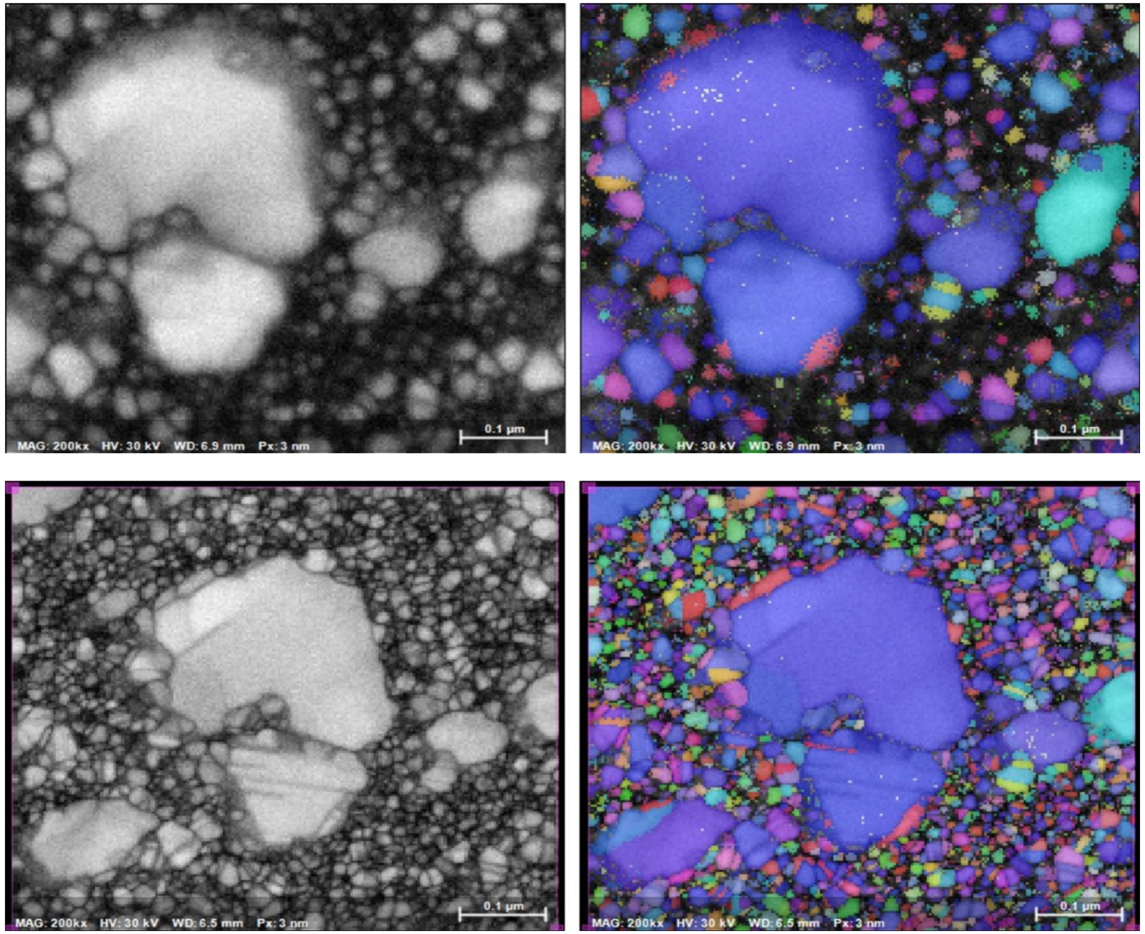


Goal: Achieve the best spatial resolution possible!

ESPRIT FIL-TKD
Enabling nano-scale TKD mapping

- Available for certain SEMs using **F**ull **I**mmersion **L**ens technology
- Fully integrated in the pattern analysis process of ESPRIT 2
- Works for all applications except residual strain analysis
- Results show here were acquired using a +10 years old NovaNano SEM
- DISCLAIMER: Resolution difference between the two functioning modes is very likely not as dramatic on latest gen. e- columns

No magnetic field ⬄ analytical mode



With magnetic field ⬄ Ultra-High Resolution (UHR) mode

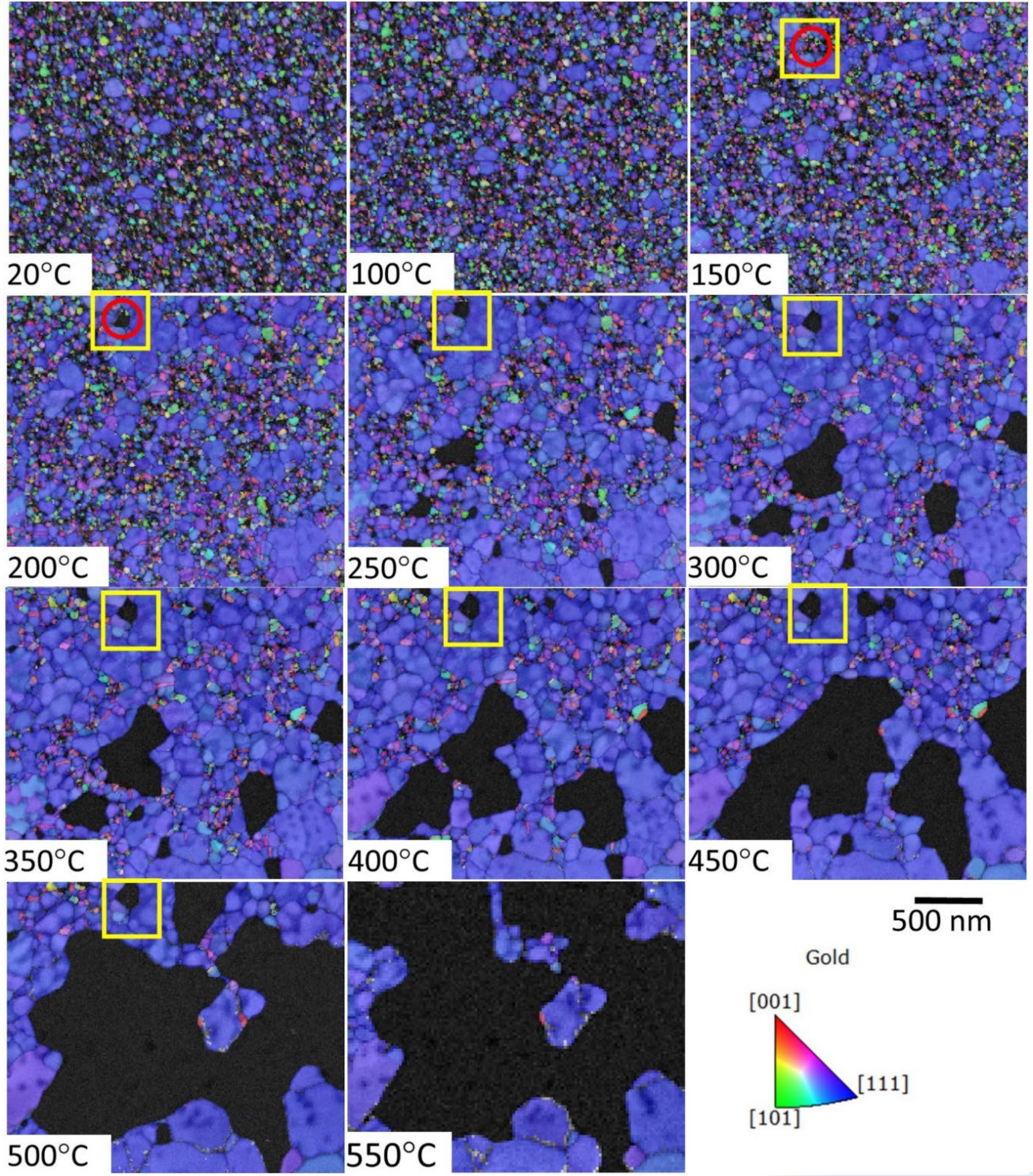
ESPRIT TRM
Time Resolved Measurements

- Automatic and repetitive acquisition of time resolved images & maps

  – *close to real time visualization of samples during in-situ experiments*

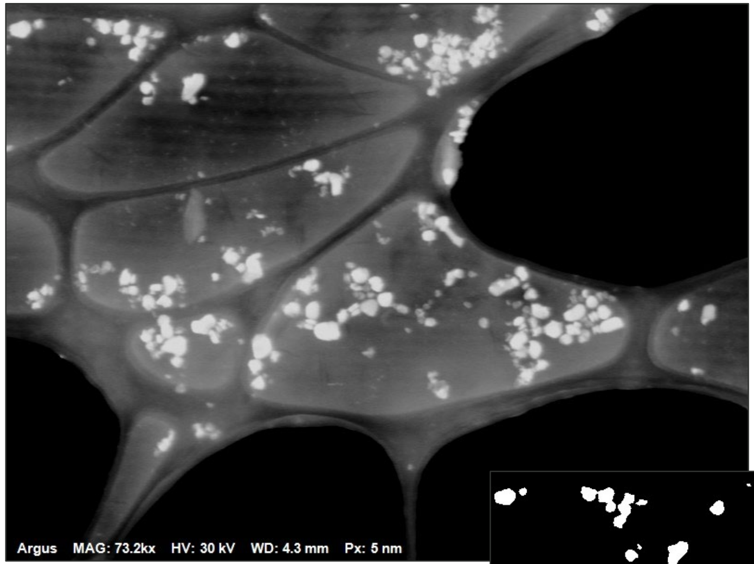
- Applicable to EDS/EBSD/TKD on SEM and EDS on TEM

- Works on same location or multiple user defined locations



“Elevated temperature transmission Kikuchi diffraction in the SEM” Fanta et all, Materials Characterization, Vol´.139, May 2018, Pages 452-462

ESPRIT MaxYield
Productivity boost



ARGUS image of nanoparticles on C-lacey support film

MaxYield

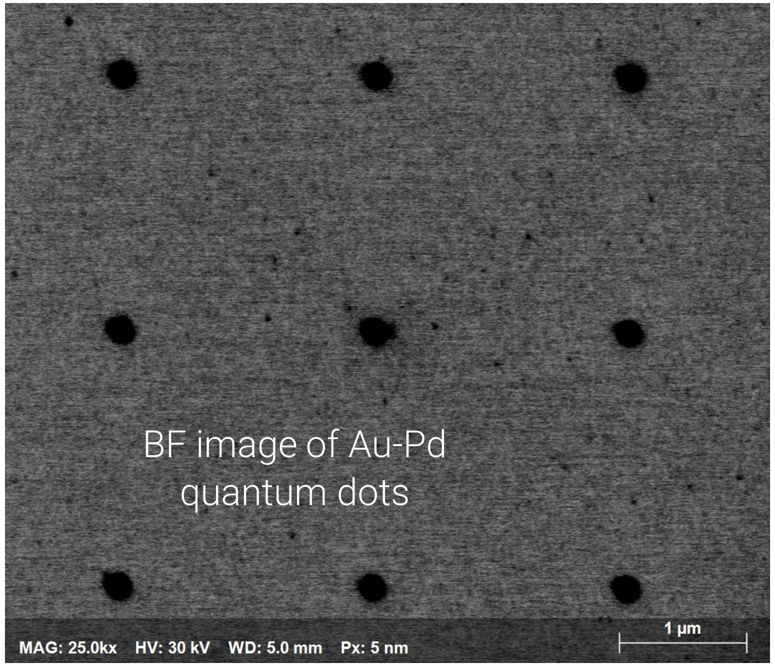
Binarized image to be used as a mask

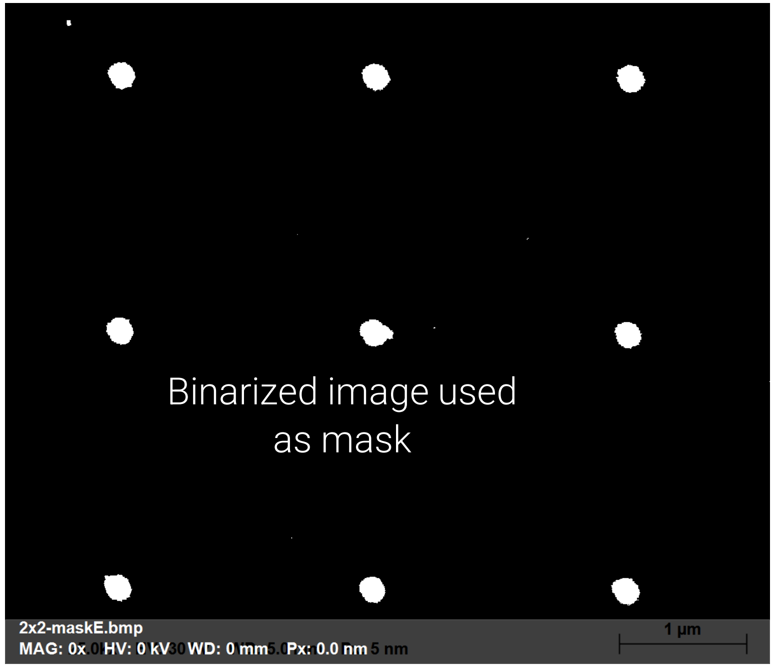
ESPRIT MaxYield => key details / *benefits*:

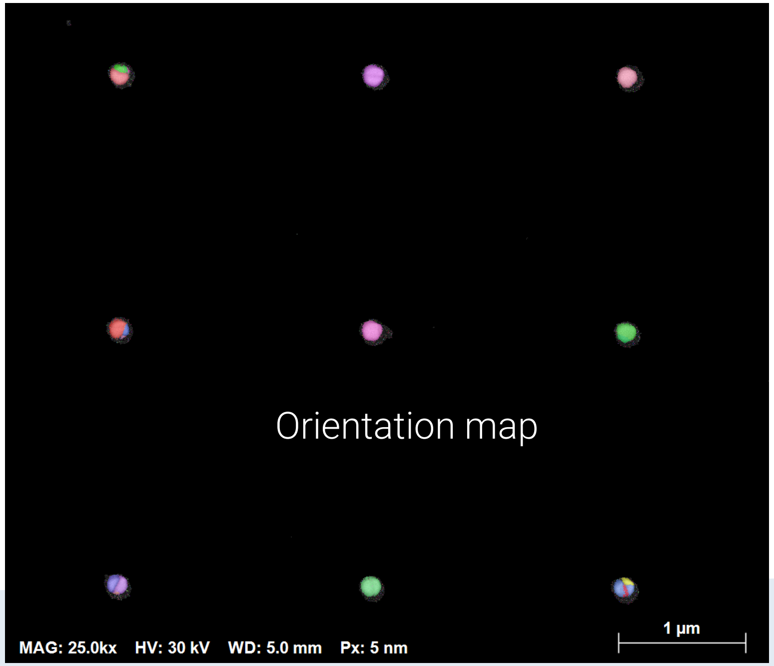
- Acquire and binarize ARGUS/SEM images
- Use such images as masks to map sparse samples, e.g. nanoparticles, nanorods, nanotubes, etc.
- Acquire data only from the area of interest:
  - Productivity boost
  - Reduced drift induced artifacts

ESPRIT MaxYield
Productivity boost

- Use masks to map sparse samples, e.g. nanoparticles, nanorods, nanotubes, etc.
- Acquire data only from the area of interest







Thank you!

Info.bna@bruker.com
www.bruker.com/bna

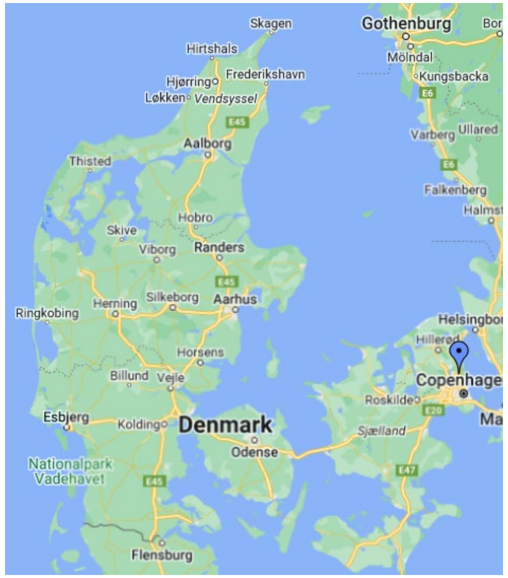
Daniel Goran
Daniel.Goran@bruker.com

BRUKER

Innovation with Integrity

On-axis TKD at DTU Nanolab

Alice Bastos da Silva Fanta



Technical University of Denmark (DTU)

Nanolab - National Centre for Nano Fabrication and Characterization

Clean room facility at DTU

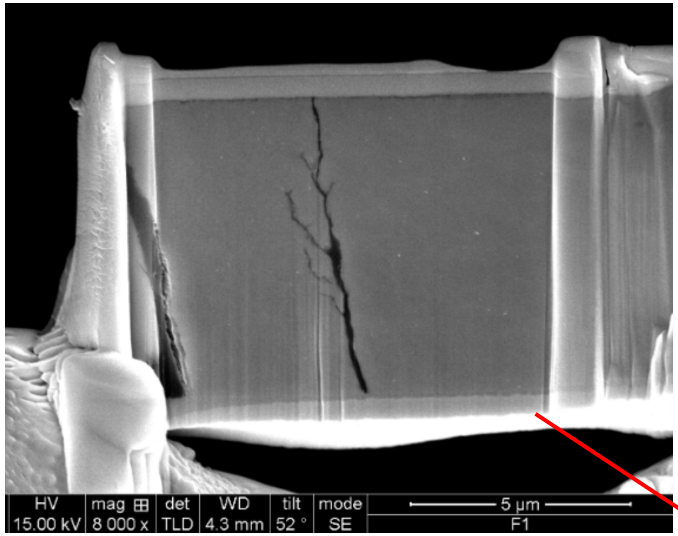
Electron microscopy facility at DTU

Outline

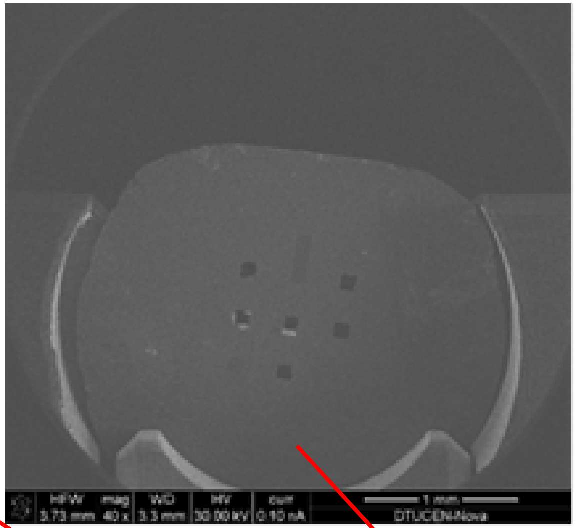
- General experience with setting up a TKD experiment
- Some examples of TKD applications
  - Characterization of nanoparticles
  - Thin film thermostability

Setup - Samples

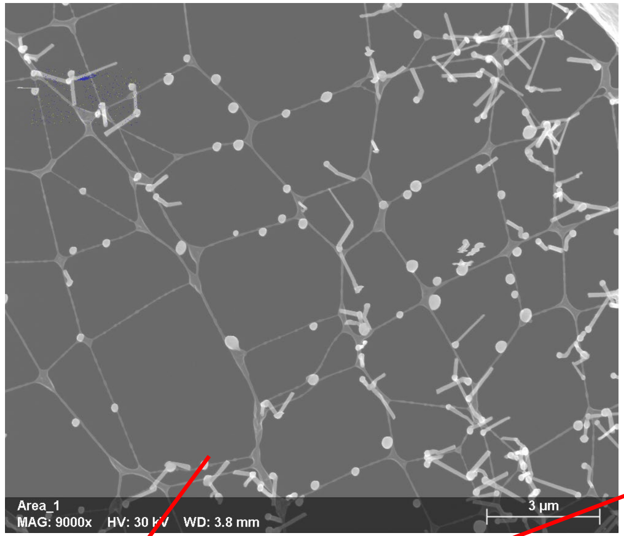
FIB lamella (steel)



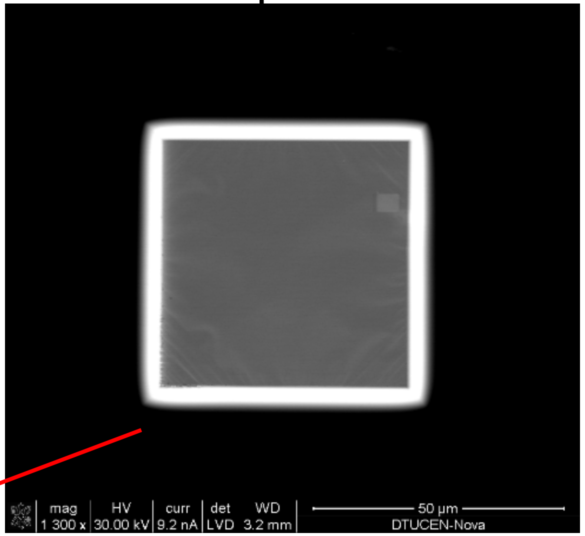
Thin films on TEM windows



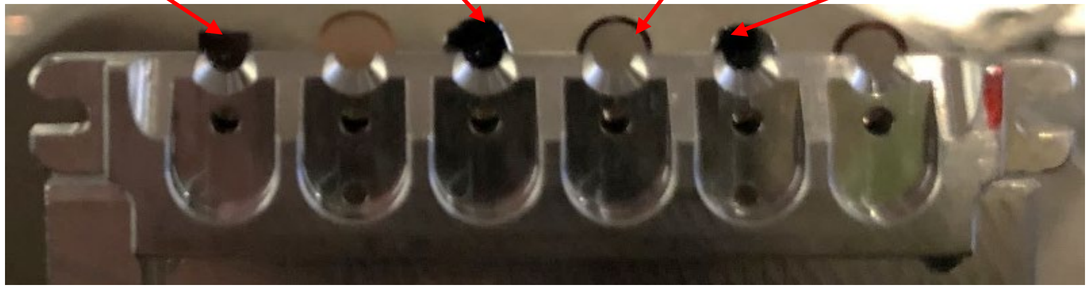
Lacey Carbon TEM grids with nanowires or nanoparticles



In-house designed TEM windows with arrays of nanoparticles



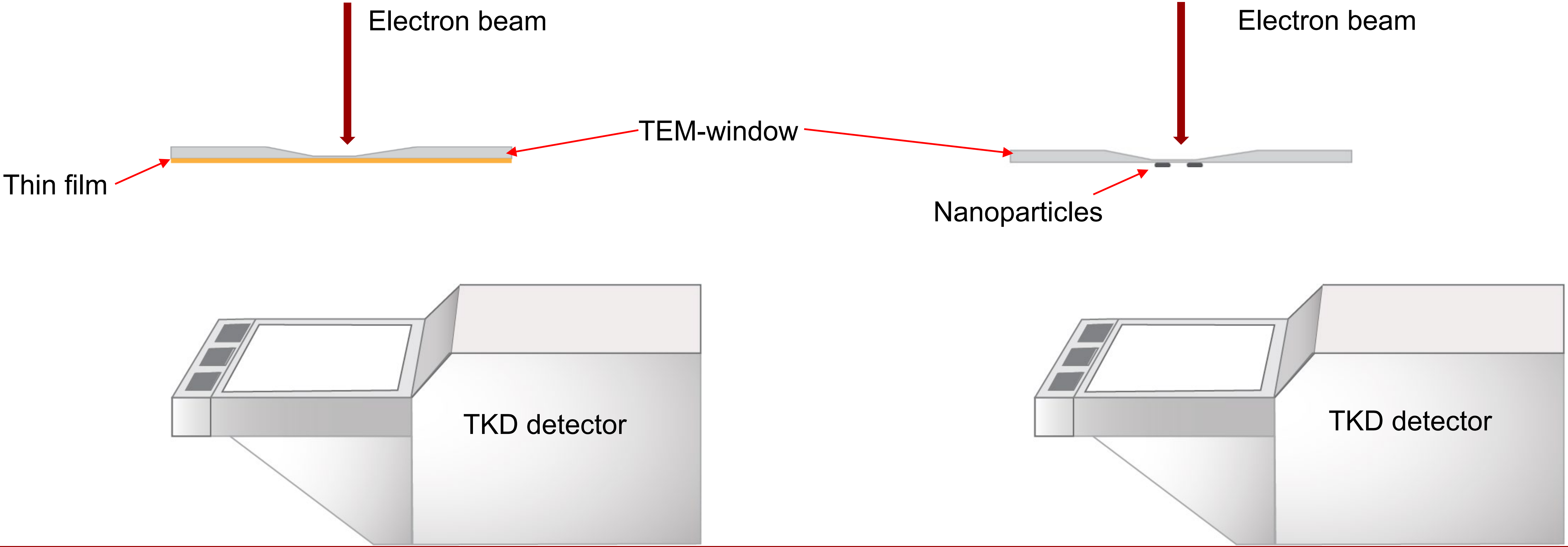
6 samples in one holder



Setup - Samples

Because most of the signal arriving at the detector comes from the **last diffraction event**

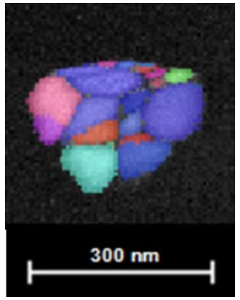
The samples are always facing the detector, not the electro beam!

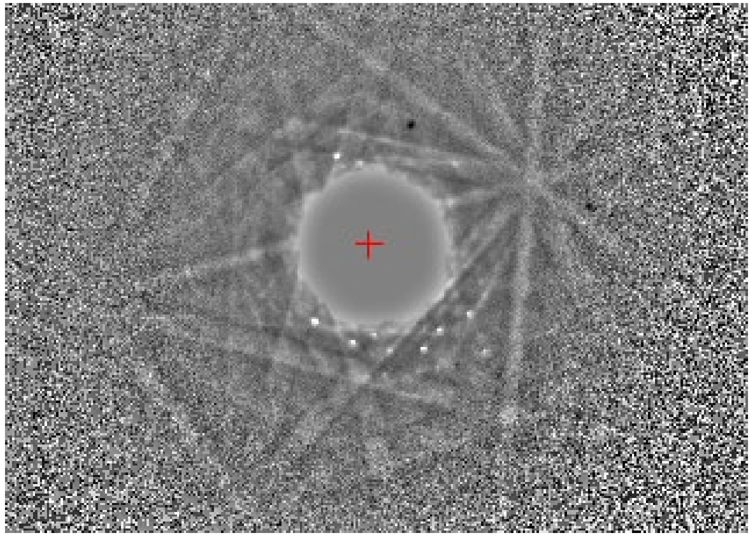


Setup – Dealing with charging effects

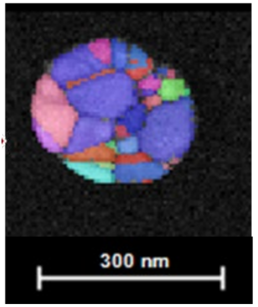
- 1. Tip: Let everything stabilize before start mapping
- 2. Tip: Measure as fast as possible
- 3. Tip: If possible, measure in low vacuum condition (water vapor)

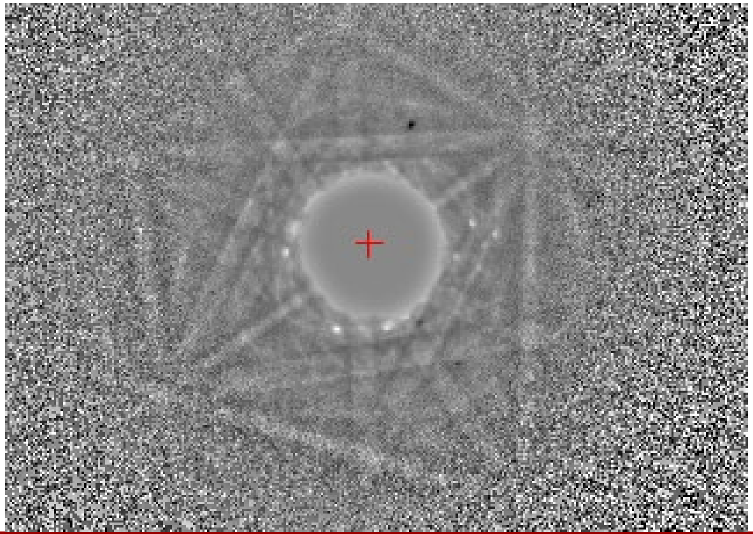
High vacuum





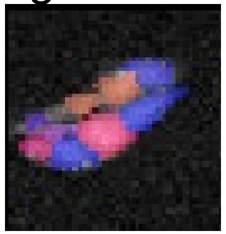
Low vacuum (50Pa)





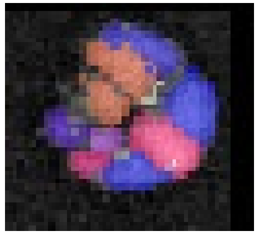
Setup – Dealing with charging effects

High Vacuum



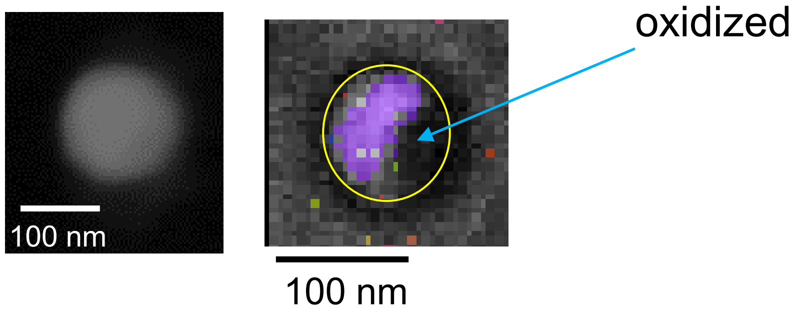
100nm

Low Vacuum

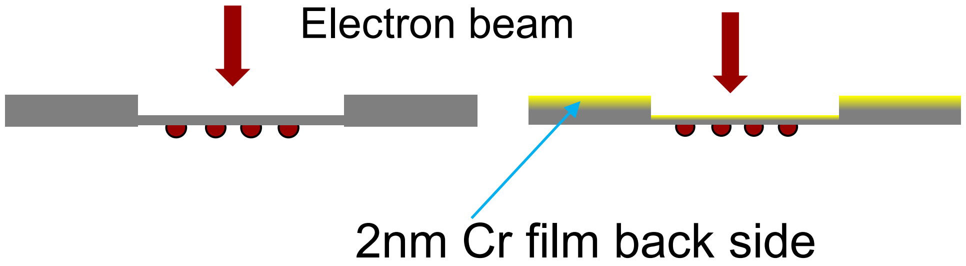


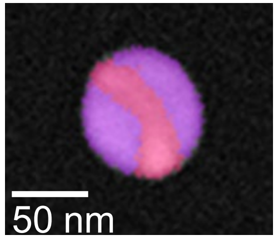
100nm

Cu-particles oxidizes faster in low vacuum



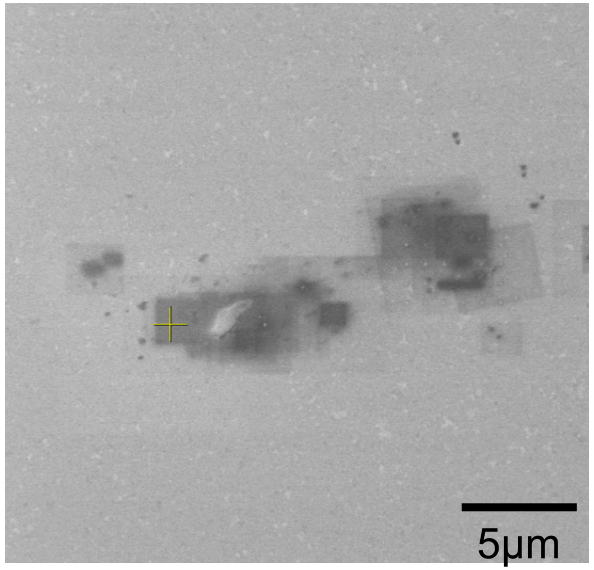
4. Tip: Coating





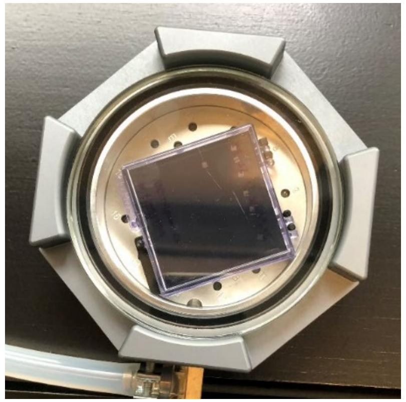
Setup – Dealing contamination

CARBON CONTAMINATION

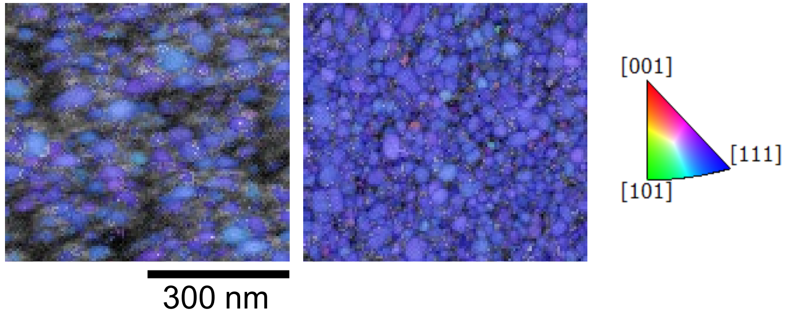


Always a challenge!

- We have introduced a Cryo-can and Cryo finder,
- We do our best to keep the sample and microscope clean.



- We always plasma clean the sample in the SEM chamber previous to any experiment.



Index-rate improves significantly

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

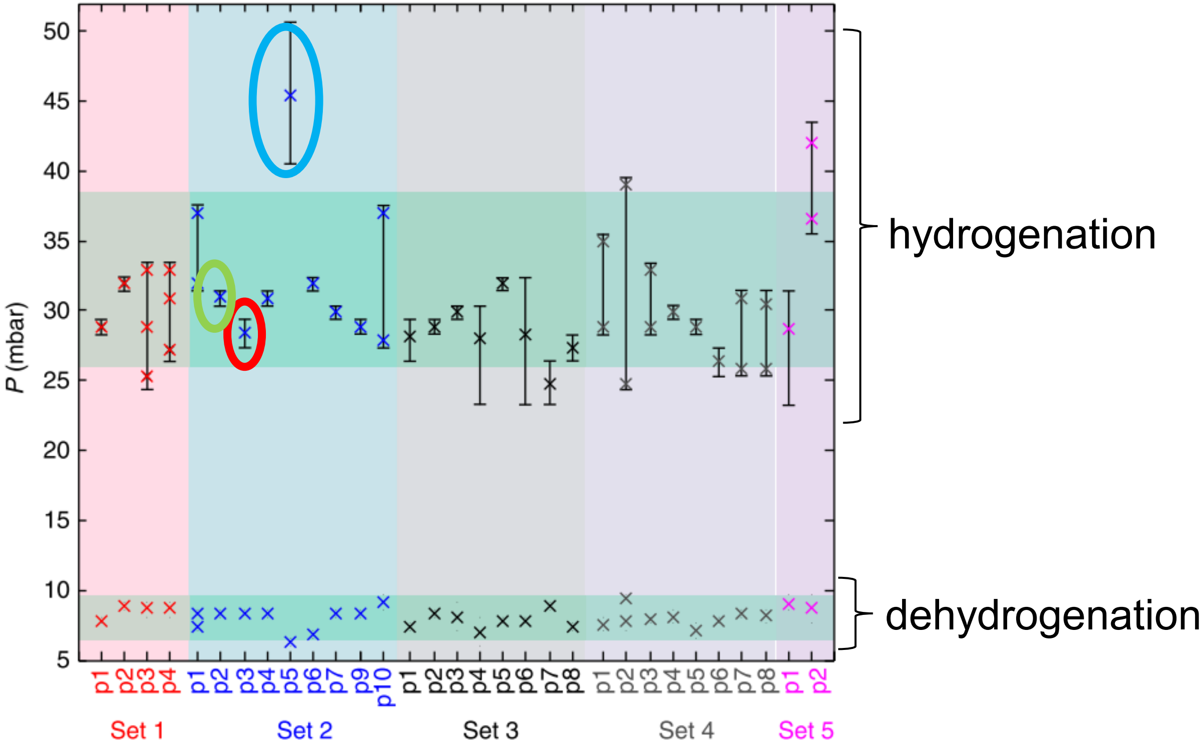
Application: Optical hydrogen sensors

In collaboration with: Christoph Langhammer and Svetlana Alekseeva (Chalmers University of Technology)

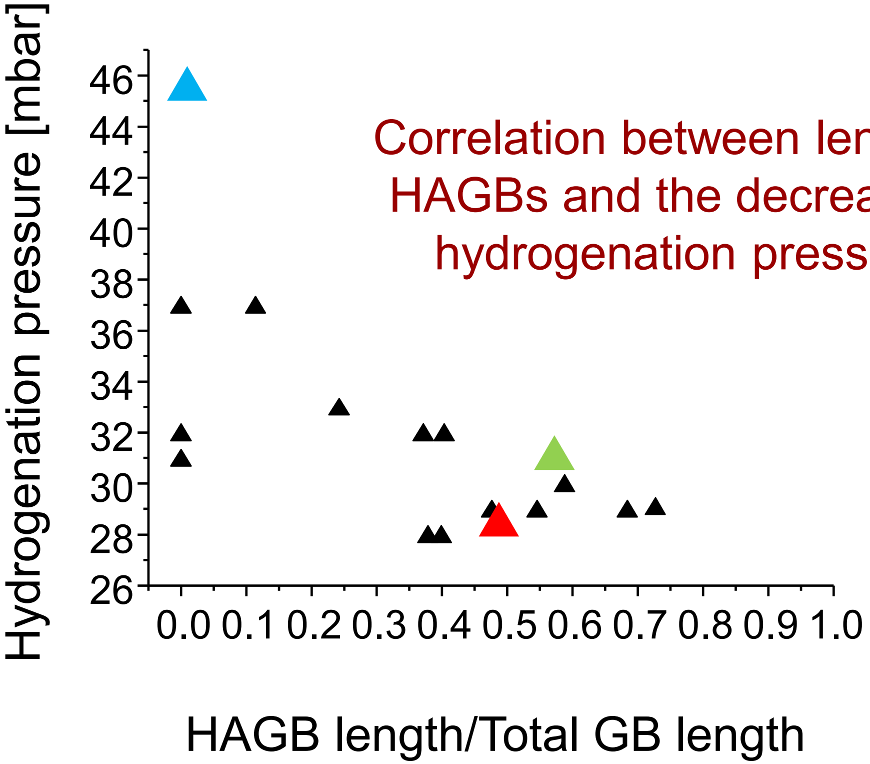
Particle-specific hydride formation

26 particles

(De) hydrogenation pressures of single Pd nanoparticles



Study the role of the microstructure with TKD on hydrogenation of individual nanoparticle.



Correlation between length of HAGBs and the decrease in hydrogenation pressure

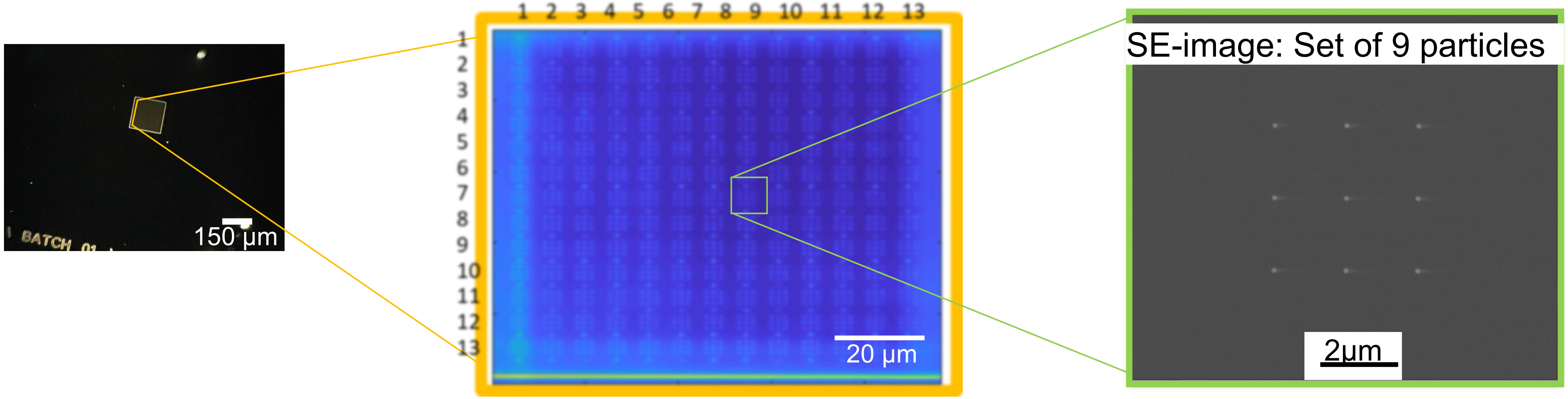
Ref. : S. Alekseeva et al., Nat. Commun. 8 (2017) and Nugroho et al., Nat. Materials (2019).

Application: Optical hydrogen sensors

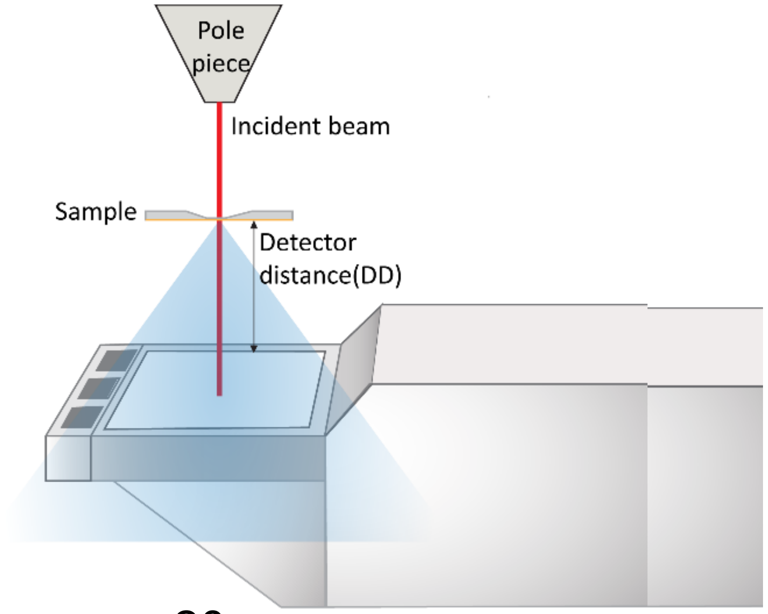
Extend it to 1000 particles

TEM Window

Particles arrangement on TEM window



Challenge: STEM and TKD



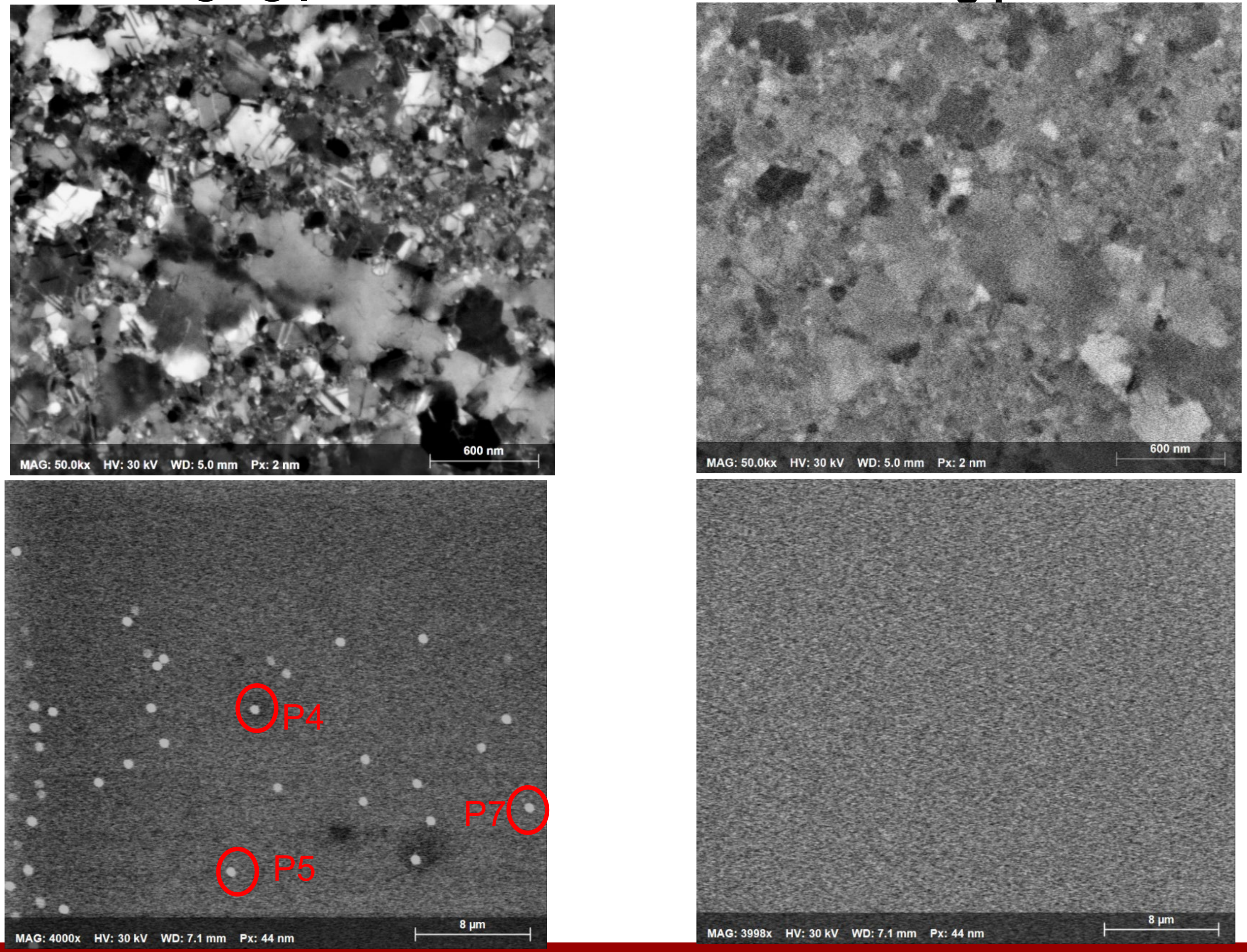
~20 mm

Direction of movement between positions

Image displacement of up to 1 µm

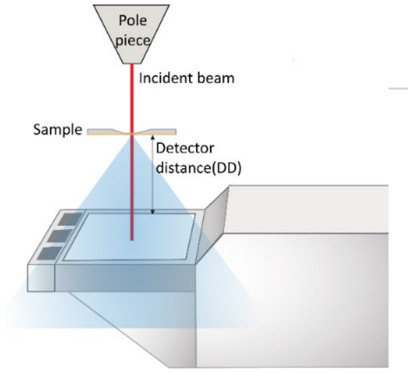
Poor image quality in measurement position

Imaging position STEM image **Measuring position**



Ref.: Fanta, A. B. S., et al. (2019). Ultramicroscopy 206 112812. https://doi.org/10.1016/J.ULTRAMIC.2019.112812

Challenge: STEM and TKD



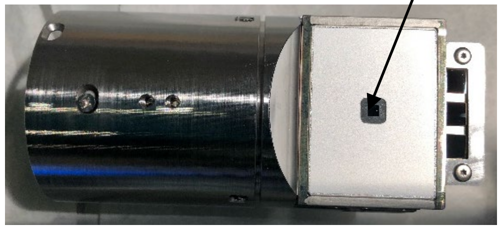
How to deal with this?

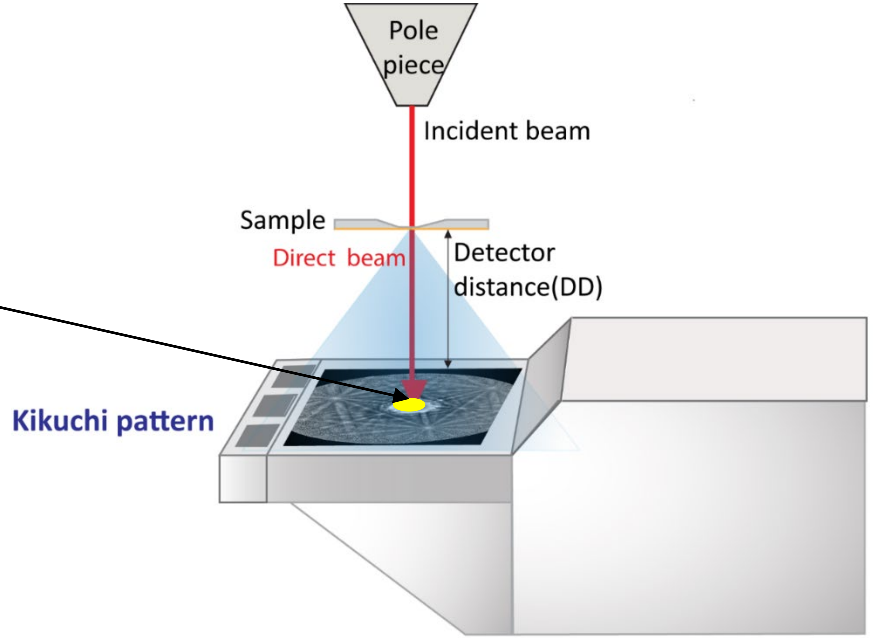
- Tilt the detector down
- Move the detector slowly following the image shift on the SE image
- Take advantage of the carbon contamination to re-locate the position (Sometime useful ☺)

STEM image and TKD measurement without detector movement

OPTIMUS 2

Bright field image





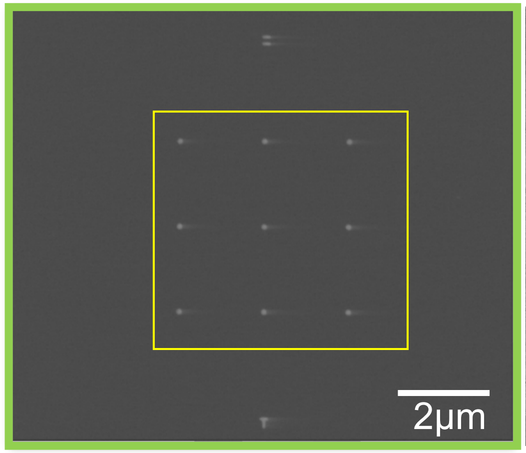
https://www.bruker.com

Ref.: Fanta, A. B. S., et al. (2019). Ultramicroscopy 206 112812. https://doi.org/10.1016/J.ULTRAMIC.2019.112812

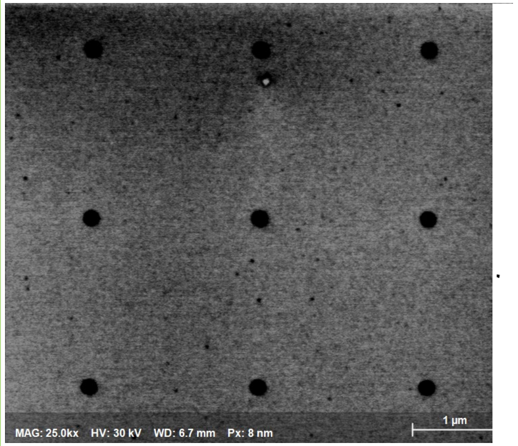
Application: Back to the goal of analyzing 1000 particles

Optimize measurement time by using a feature recognition mask – map only where there are particles

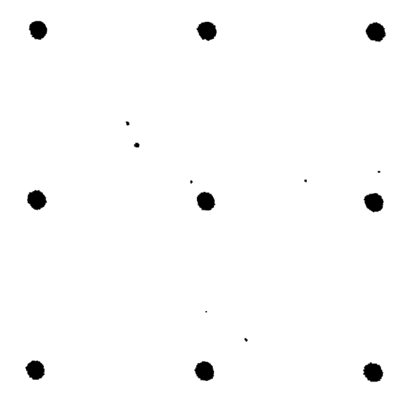
SE-image



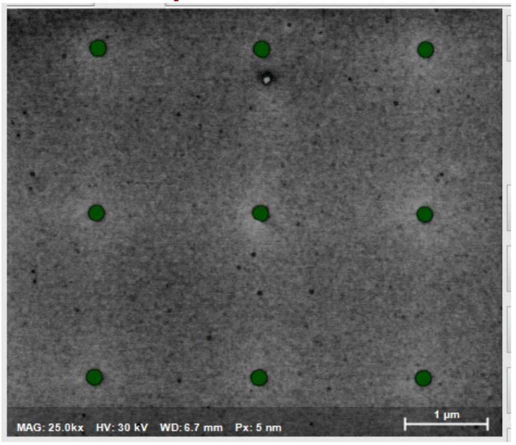
STEM image with center diode



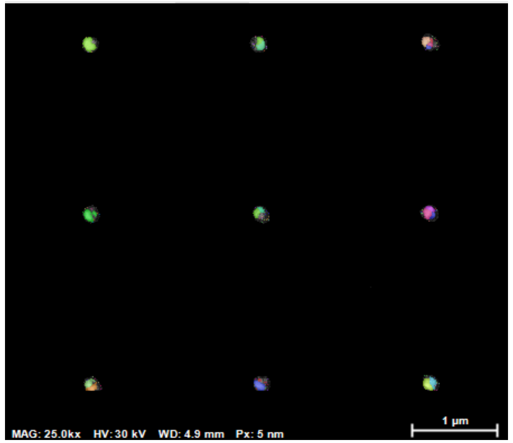
Mask



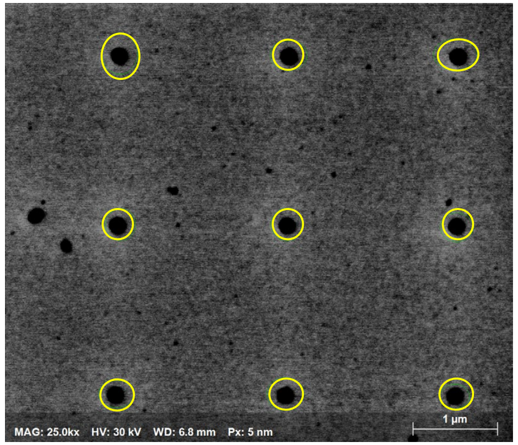
Map only over the particles



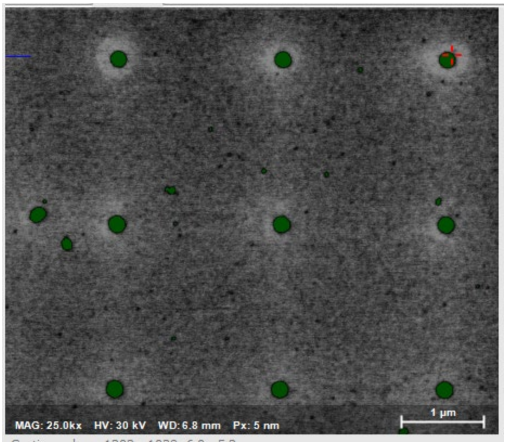
TKD map



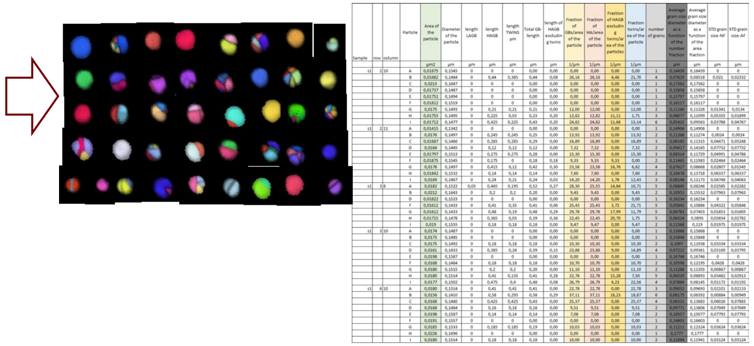
16 min



7 min



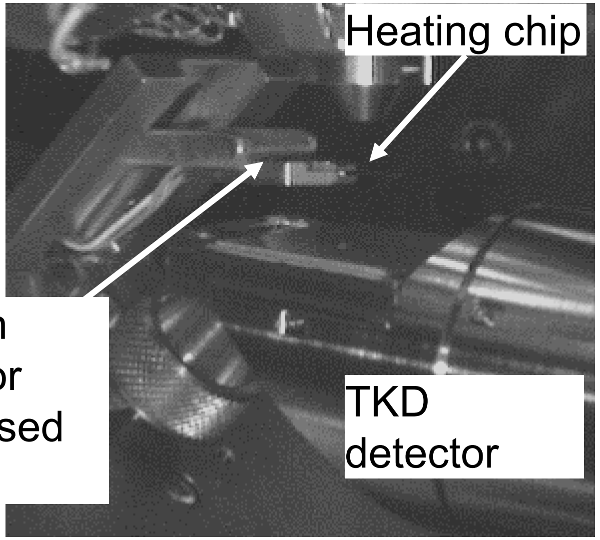
A lot of particles and data to analyze



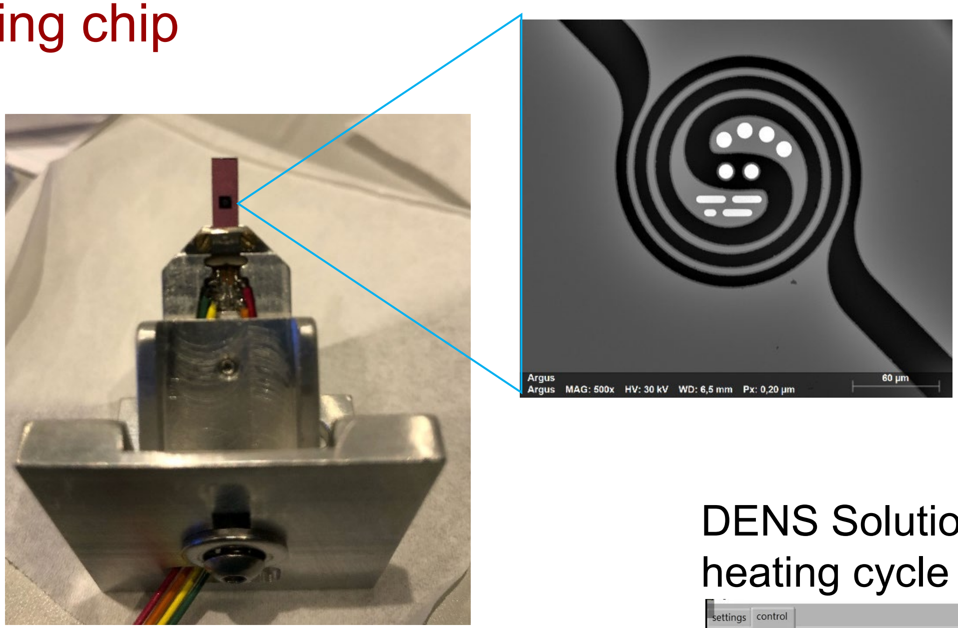
It is a powerful platform to correlate microstructure-function at the **individual nanoparticle level**

In-situ experiments

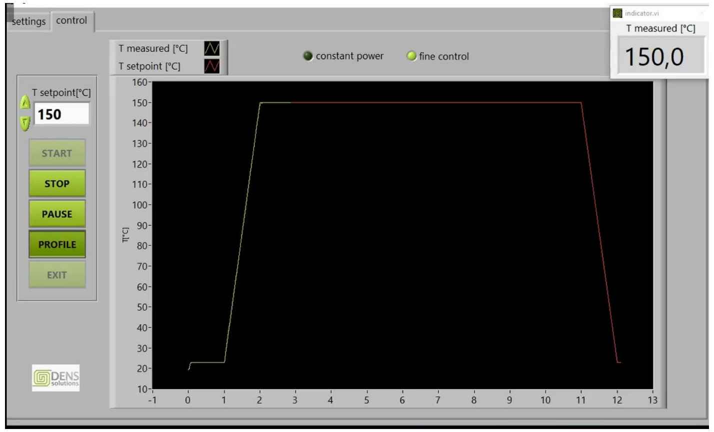
Build an SEM stage for the TEM heating chip



In-house design heating stage for TEM MEMS-based heating chip



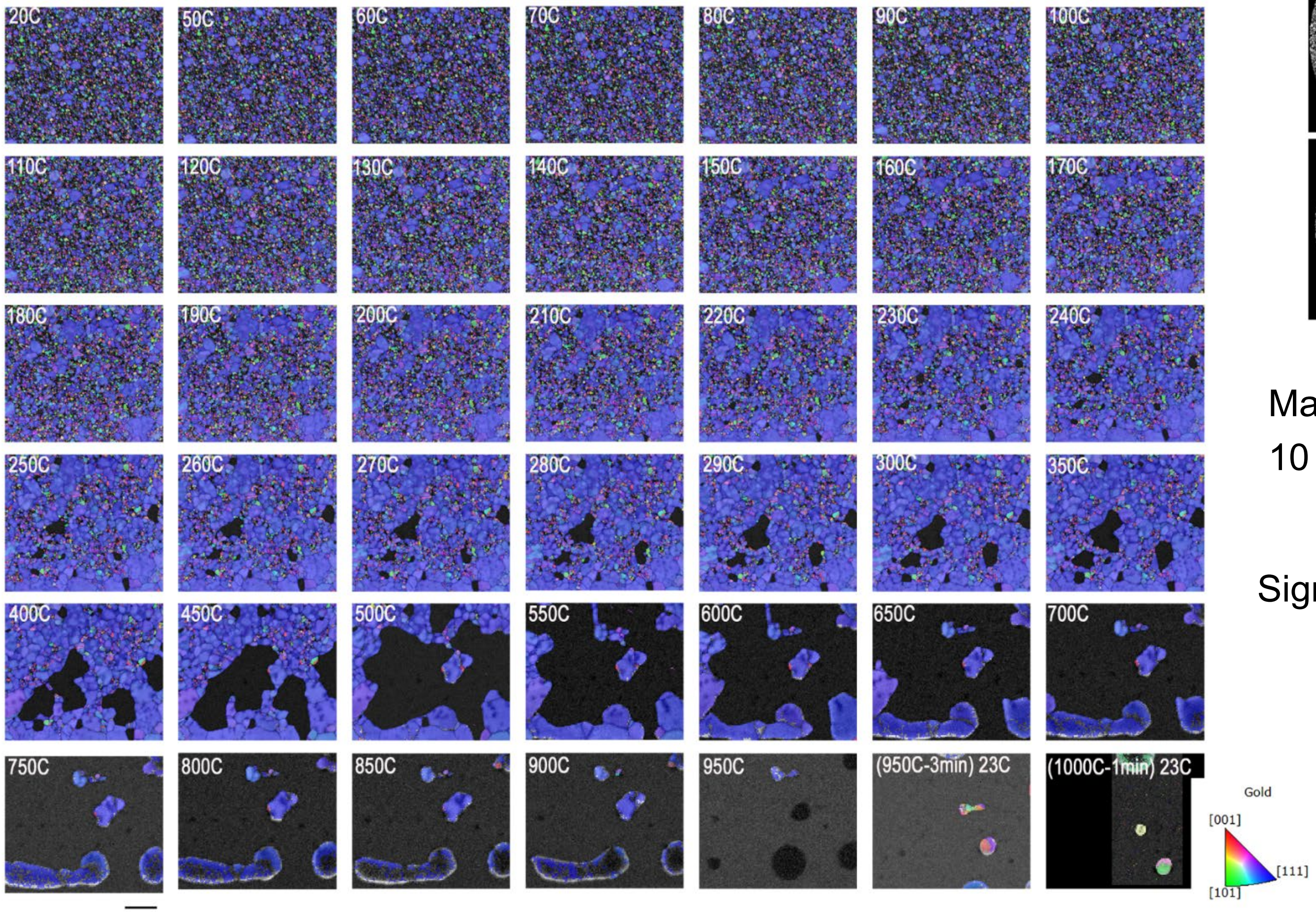
DENS Solution software to control heating cycle

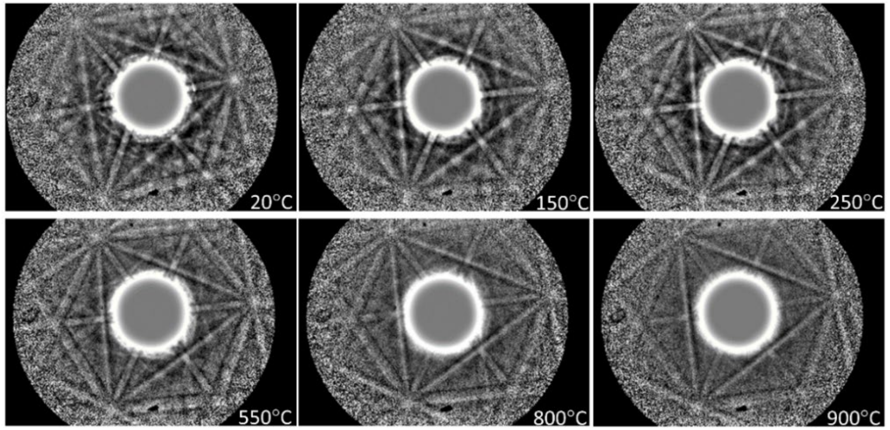


Ref. : A.B. Fanta et al. Mater. Charact. 139 (2018) 452–462.

Thin film dewetting– Previous to Optimus 2

In-situ dewetting of Au thin film





Map time 10min

10 nm step size

Significant microstructure changes during the map

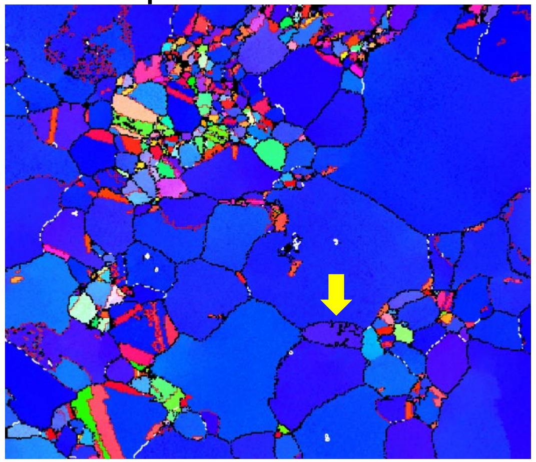
Ref. : A.B. Fanta et al. Mater. Charact. 139 (2018) 452–462.

Thermostability of thin films – Optimus 2

OPTIMUS 2

- Possibility to improve time and spatial resolution by automatically capture STEM images during heating experiments

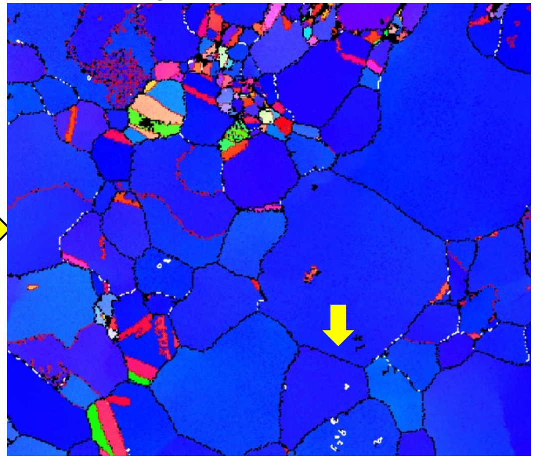
TKD map after 30 min 200° C



30 min

At 250° C

TKD map after 30 min 250° C



RT for 40 min

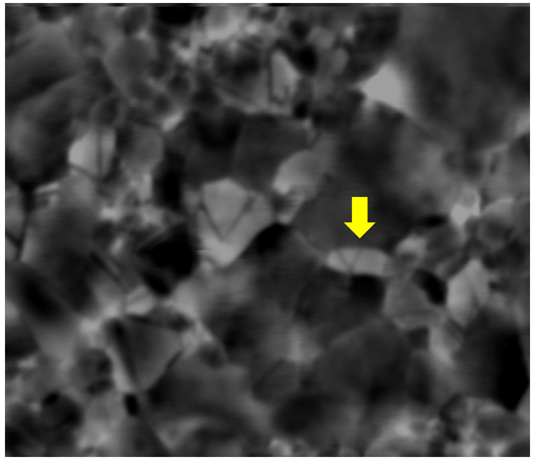
Map with 2 nm step size

200 nm

Ref.: Fanta, A. B. S., et al. (2019). Ultramicroscopy 206 112812. https://doi.org/10.1016/J.ULTRAMIC.2019.112812

Thermostability of thin films

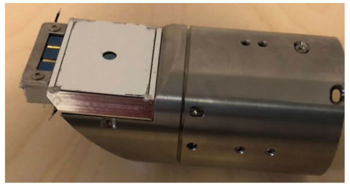
200° C



- 30 min STEM images

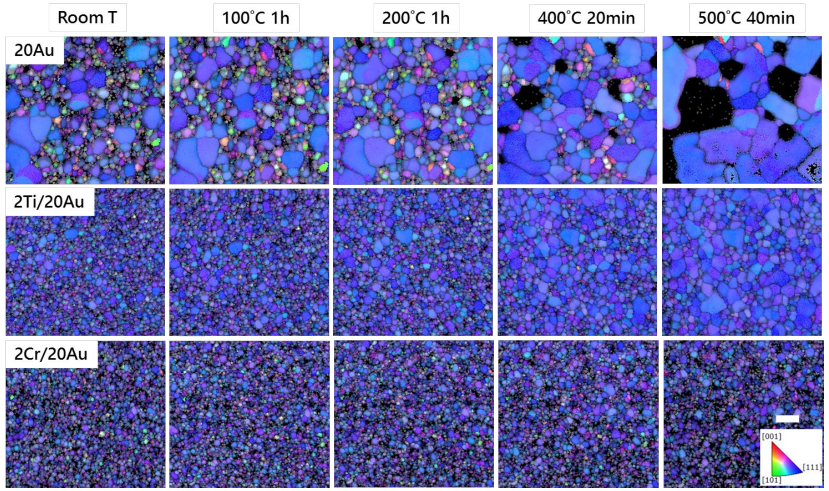
(manually saved every 45 sec)

- 40 min map at RT

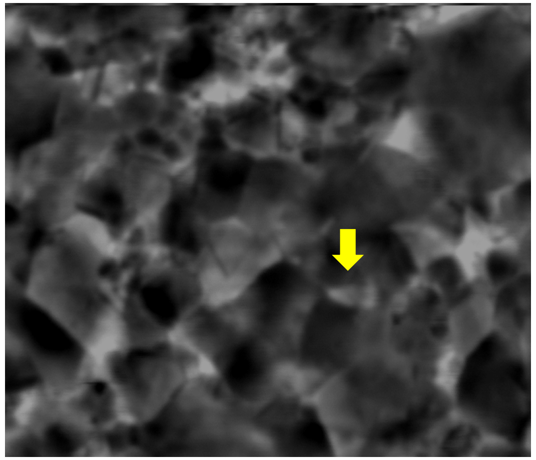


Prototype

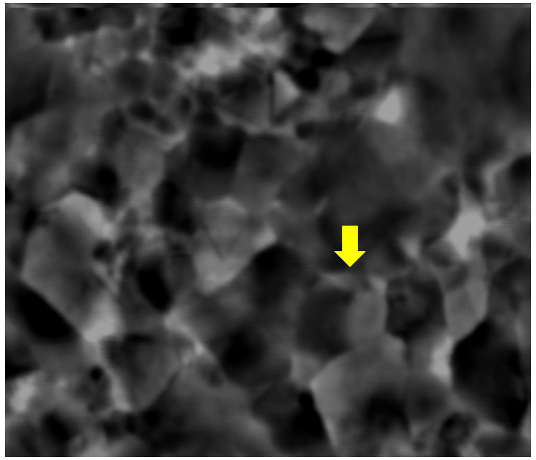
Adhesion layer influence



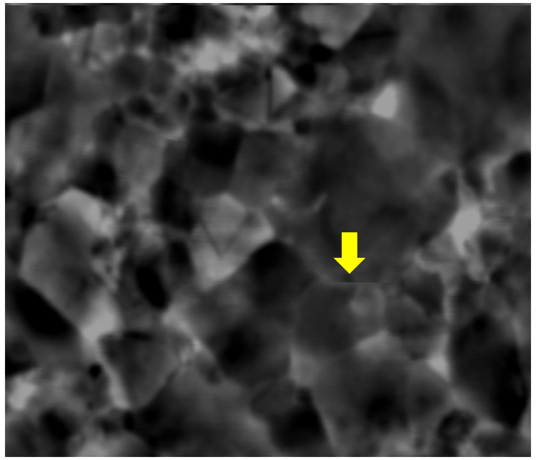
250° C



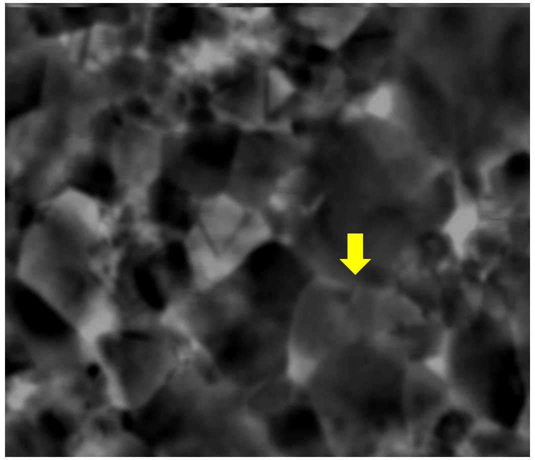
250° C (45s)



250° C (1.5 min)



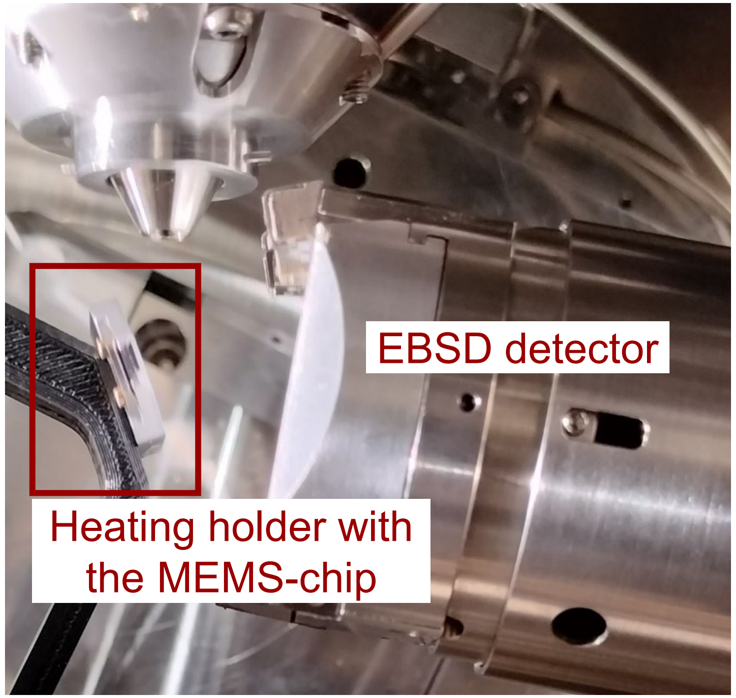
250° C (2.3 min)

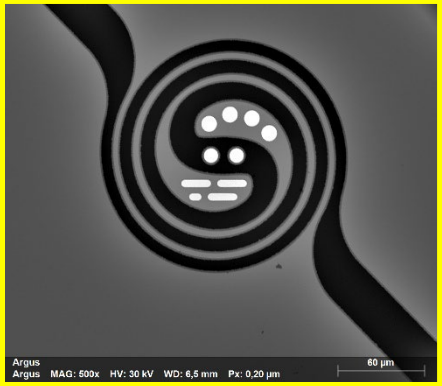


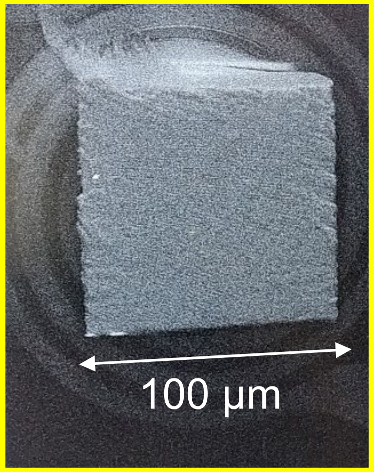
200 nm

Ref.: *Fanta, A. B. S., et al. (2019). Ultramicroscopy 206 112812. https://doi.org/10.1016/J.ULTRAMIC.2019.112812*

Next-step: In-situ EBSD with MEMS based heating chips







Acknowledgments:

Thanks to the people who contributed to the work:

DTU: Christian Damsgaard, Christina König

Former DTU: Adam Fuller, Matteo Todeschini, Mario Heinig, Andrew Burrows, Hossein Alimadadi, Mohammad Ahmed and Mamadou Mandie Kone

Chalmers: Sara Nilsson, Svetlana Alekseeva, Ferry Nugroho, David Albinsson, Henrik Klein Moberg and Christoph Langhammer.

Screen recording during heating of Au thin films 40 x faster

200nm

Thank you for your attention