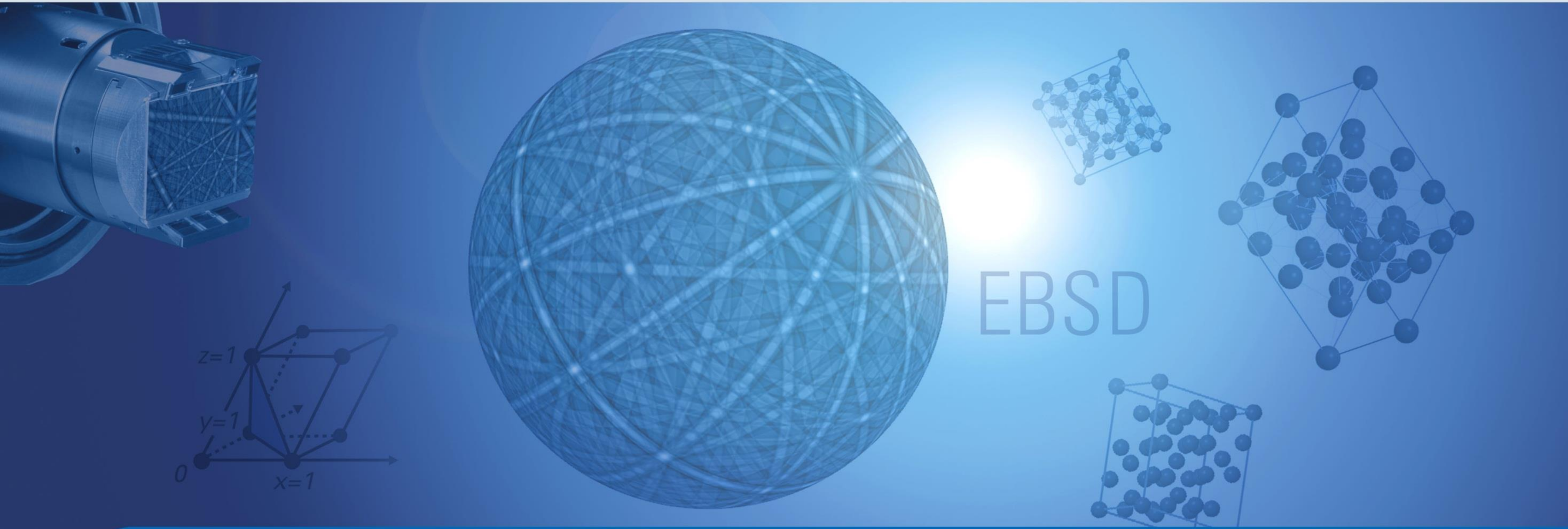
Nanostructural characterization of semiconductors with SEM

Bruker Nano Analytics webinar



Nanostructural characterization of semiconductors with SEM

Overview

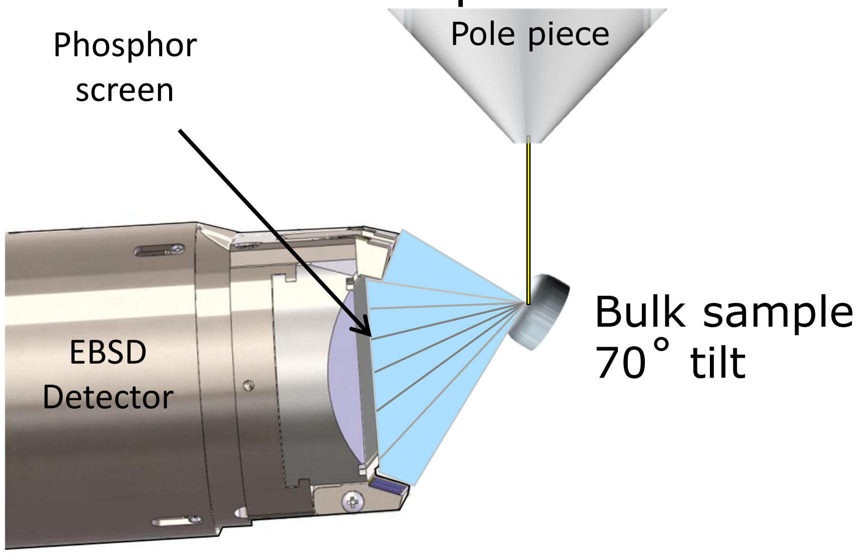
- Why TKD?
- on-axis TKD: the optimum geometry for crystal orientation mapping at the nanoscale
- Challenges with semiconducting nanostructures
- Application examples
- ARGUS™ imaging system
- Summary

Why TKD?

EBSD vs. off-axis TKD

Differences

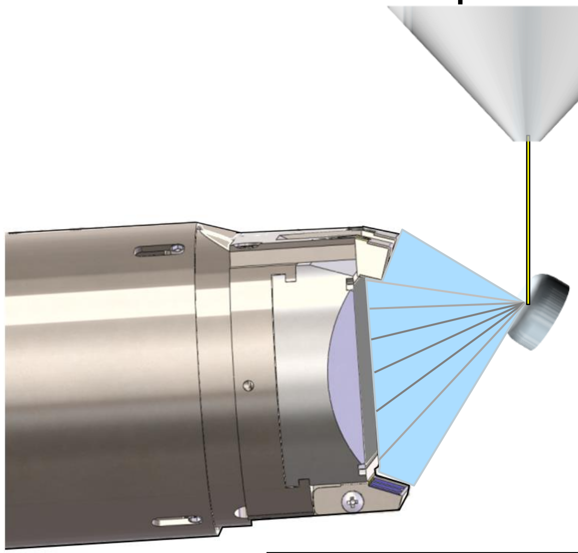
EBSD setup



EBSD vs. off-axis TKD

Why TKD?

EBSD setup



Bulk sample
70° tilt

Si bulk sample
300 nm grain size

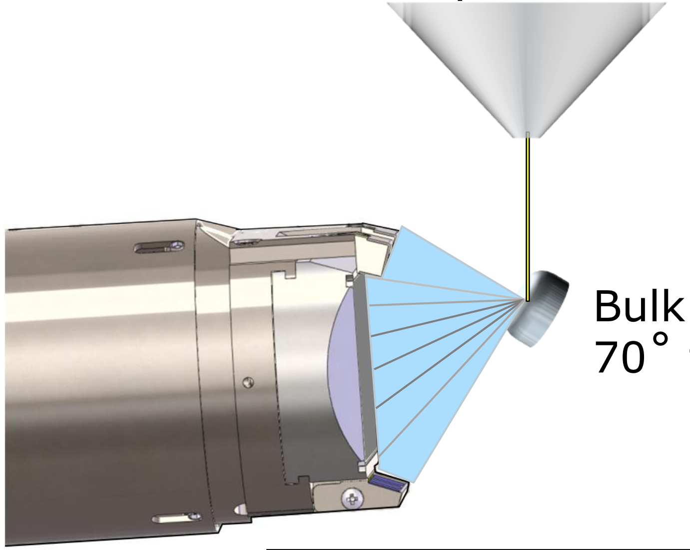
low kV EBSD
30 nm resolution

IPF map

EBSD vs. off-axis TKD

Why TKD?

EBSD setup

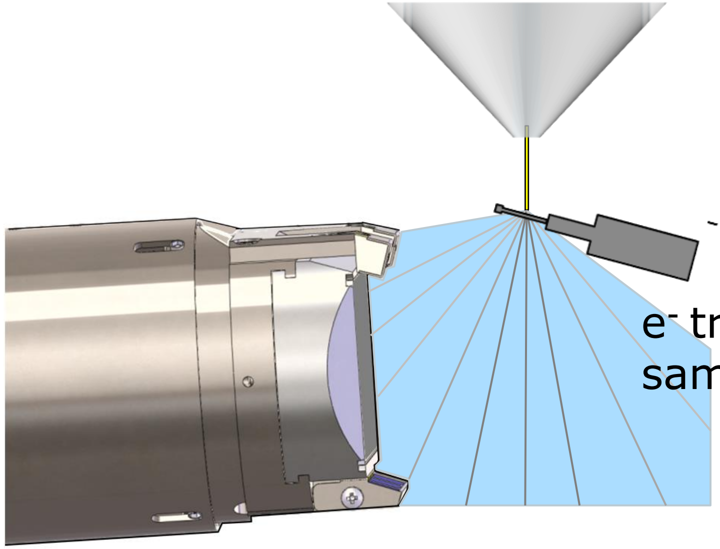


Si bulk sample
300 nm grain size

low kV EBSD
30 nm resolution

IPF map

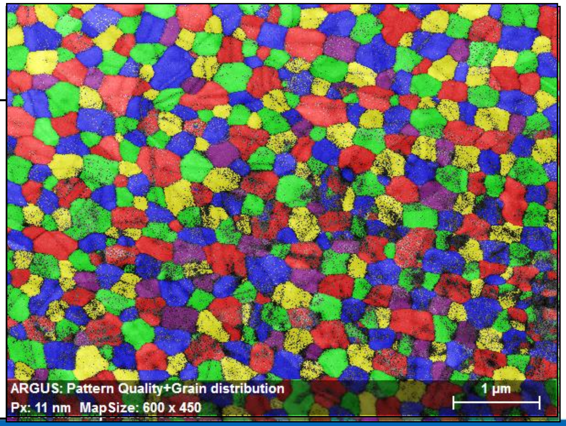
t-EBSD or off-axis TKD setup



Si lamella
300 nm grain size

off-axis TKD
11 nm resolution

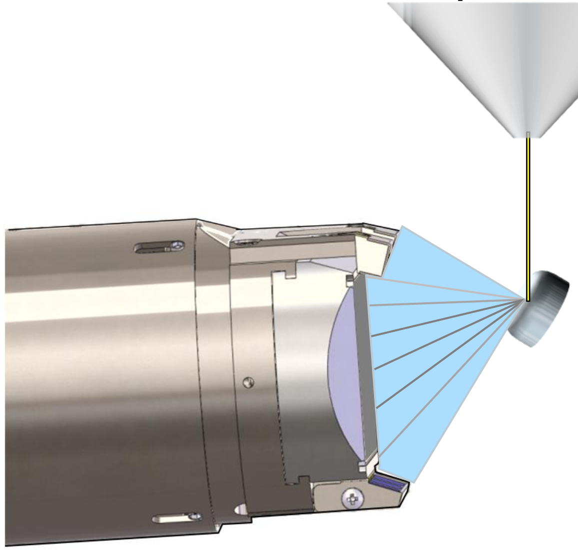
grain detection map



EBSD vs. off-axis TKD

Why TKD?

EBSD setup



Bulk sample
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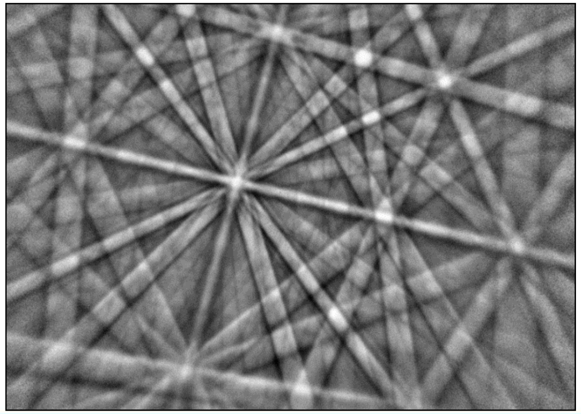
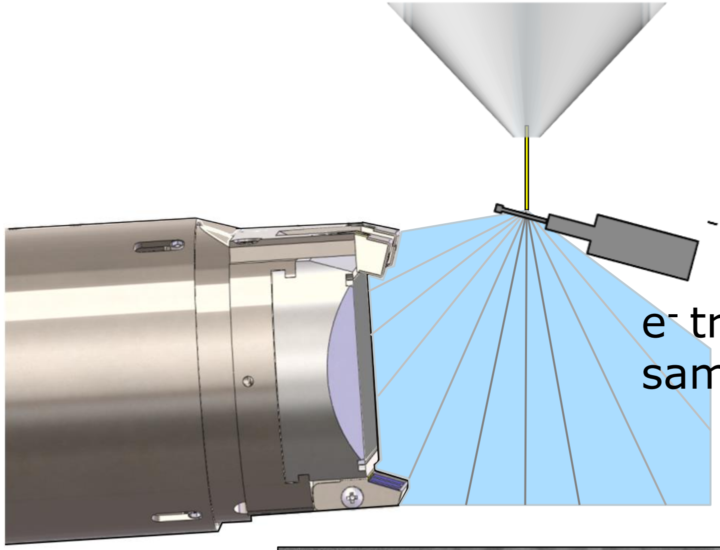
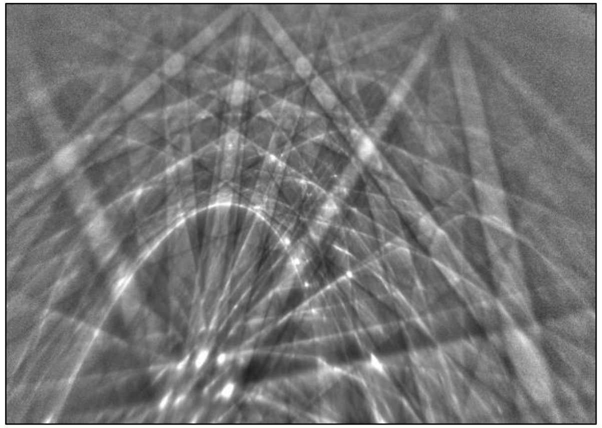


t-EBSD or off-axis TKD setup



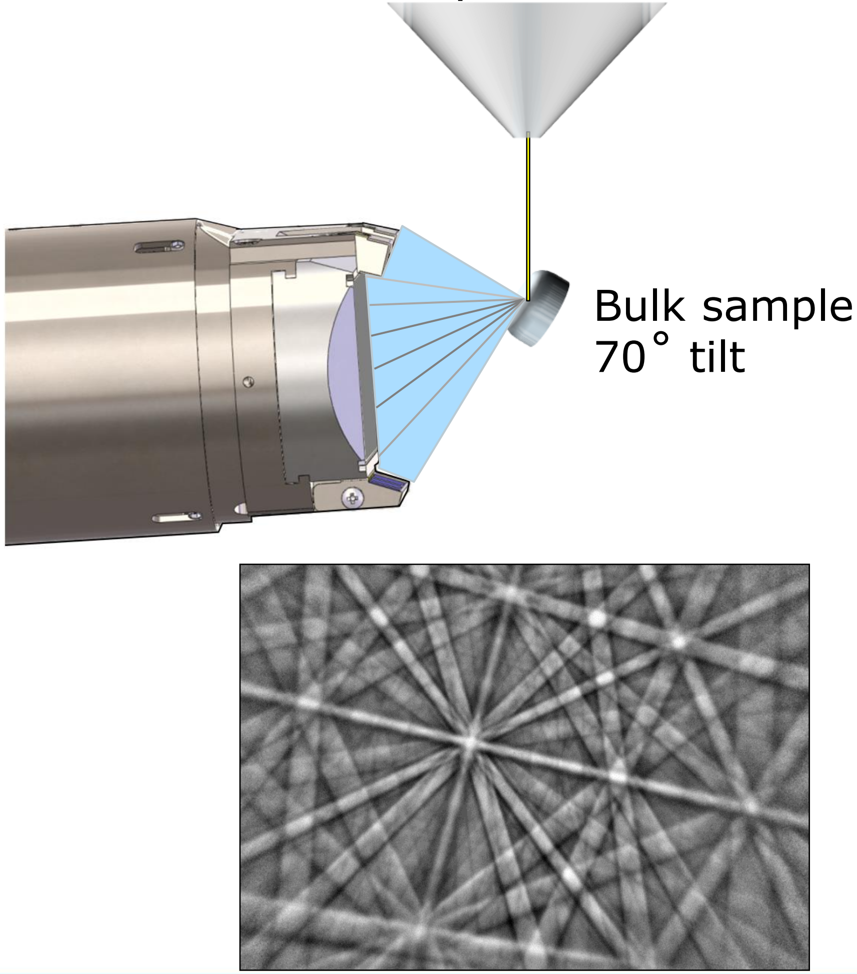
e⁻ transparent
sample @ -15° tilt



EBSD vs. off-axis TKD

Why TKD?

EBSD setup



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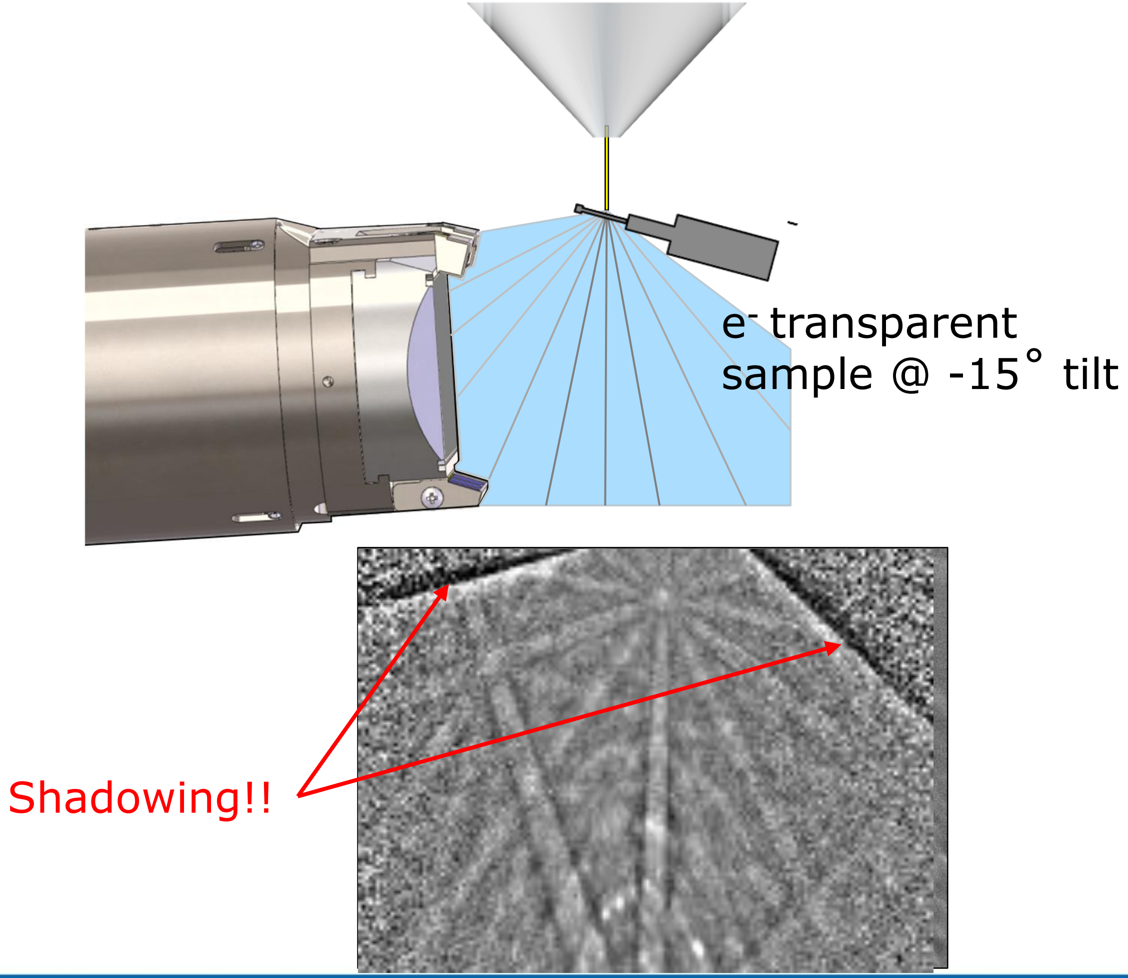
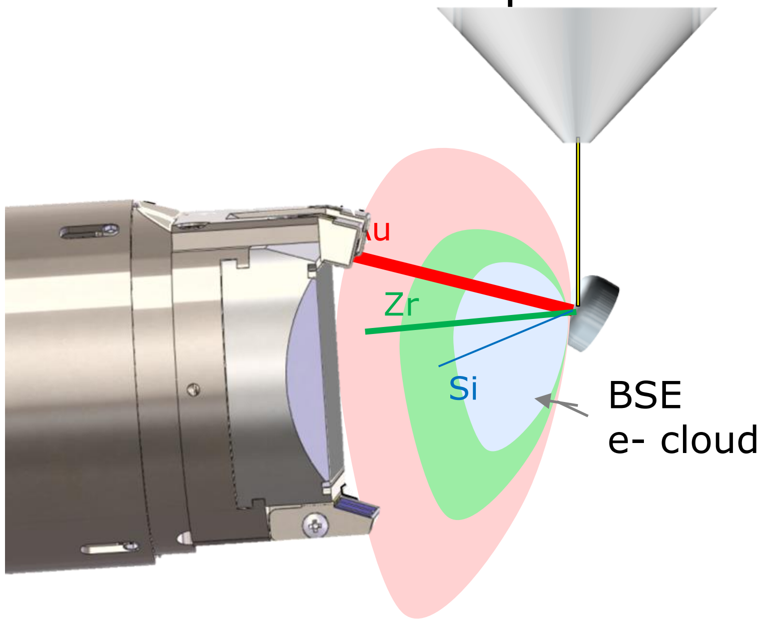
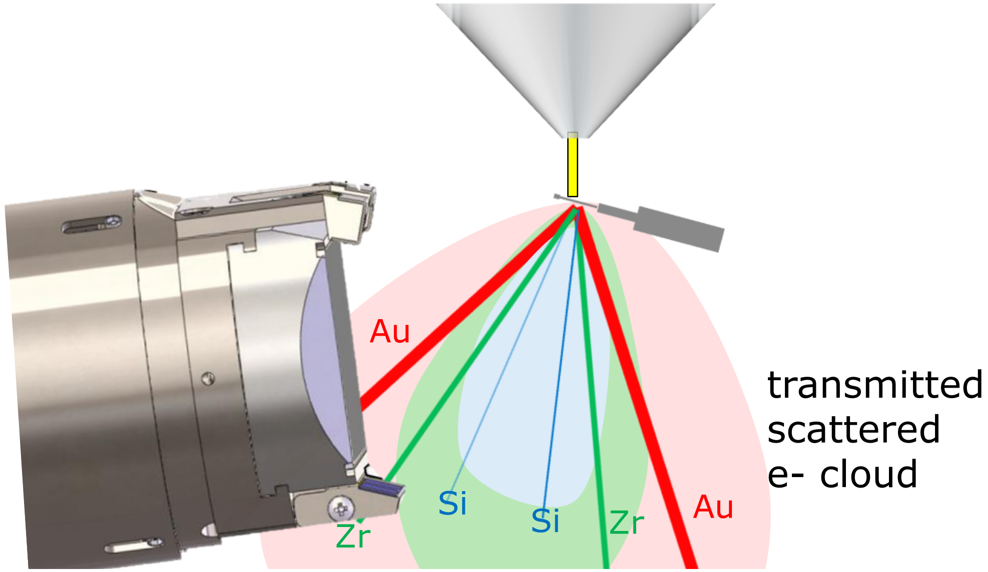


off-axis TKD
Limitations

EBSD setup



t-EBSD or off-axis TKD setup



EBSD

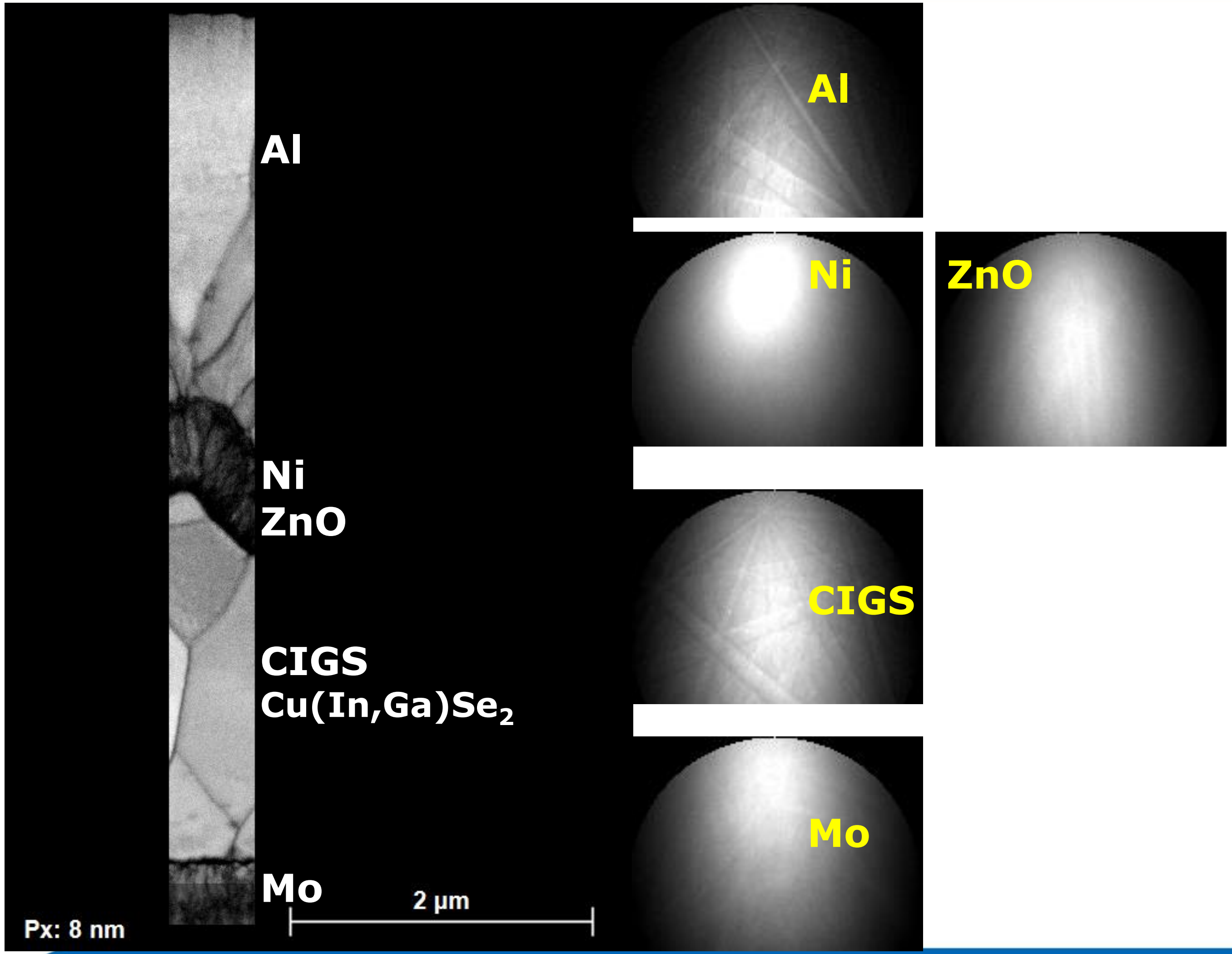
- Backscattered electrons
- Lateral resolution > 40 nm
- !! Interaction volume!!

Off-axis TKD

- Transmitted scattered electrons
- Lateral resolution > 10 nm
- !! thickness & Z !!

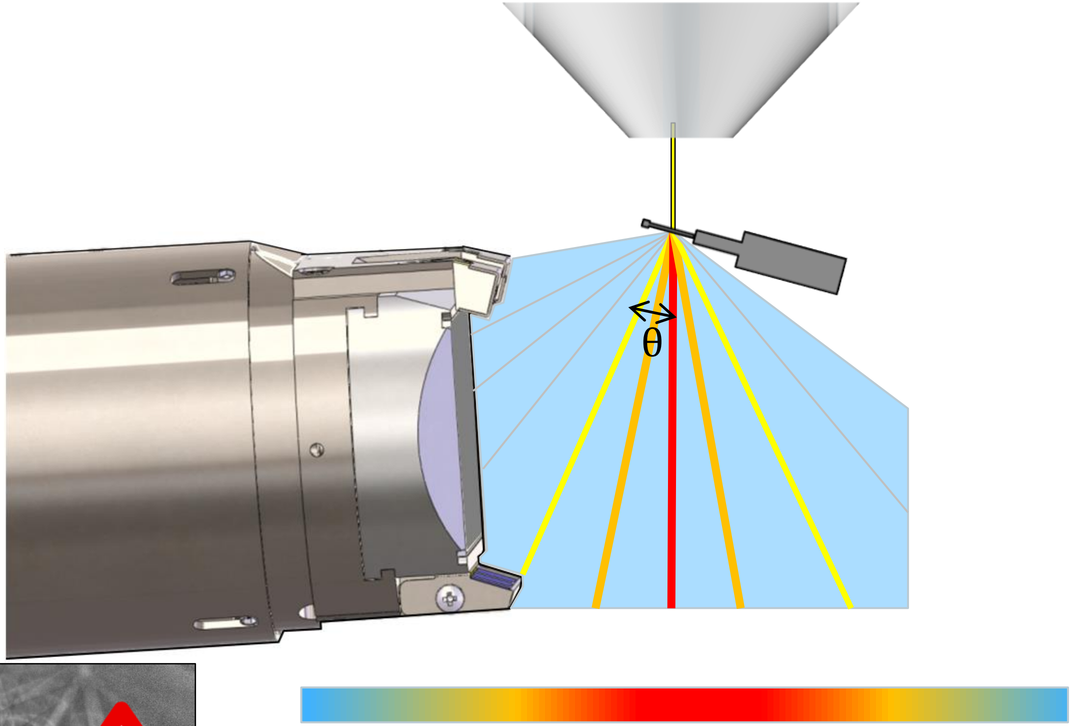
off-axis TKD

Limitations – nanomaterials with different atomic weight

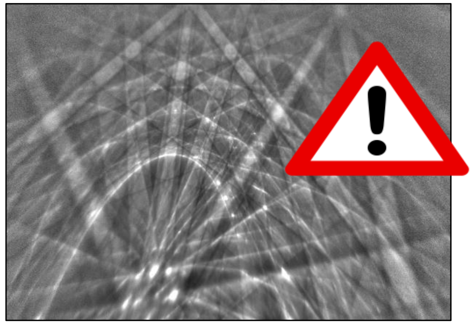


off-axis vs. on-axis TKD

signal strength



Signal strength



Gnomonic projection distortions

- Low signal yield where it is most intense
- Gnomonic projection distortions – indexing quality/band detection affected
- “low” spatial resolution

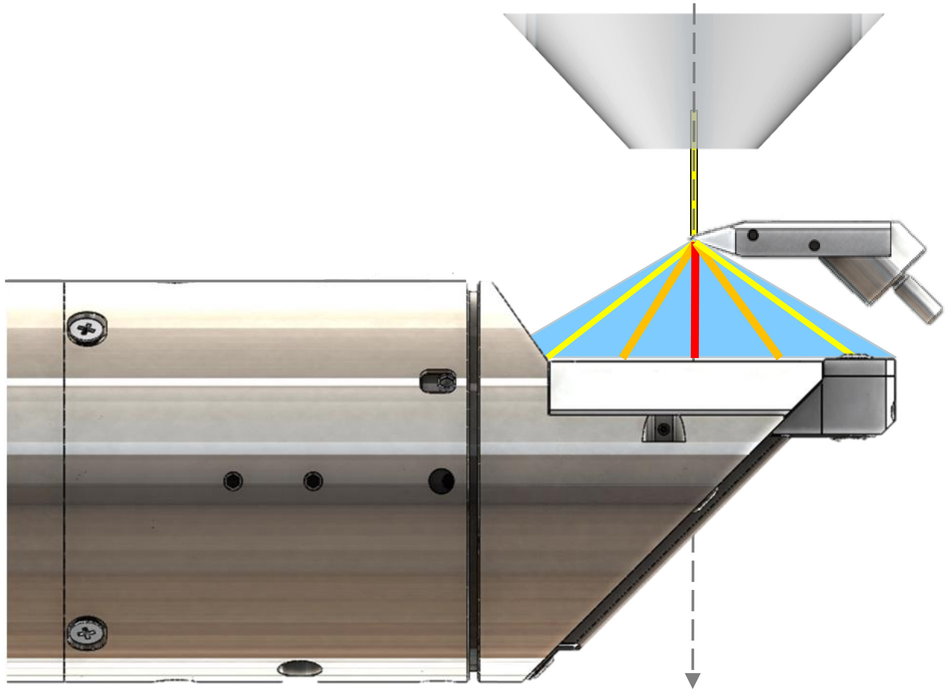
Weak signal is compensated by:

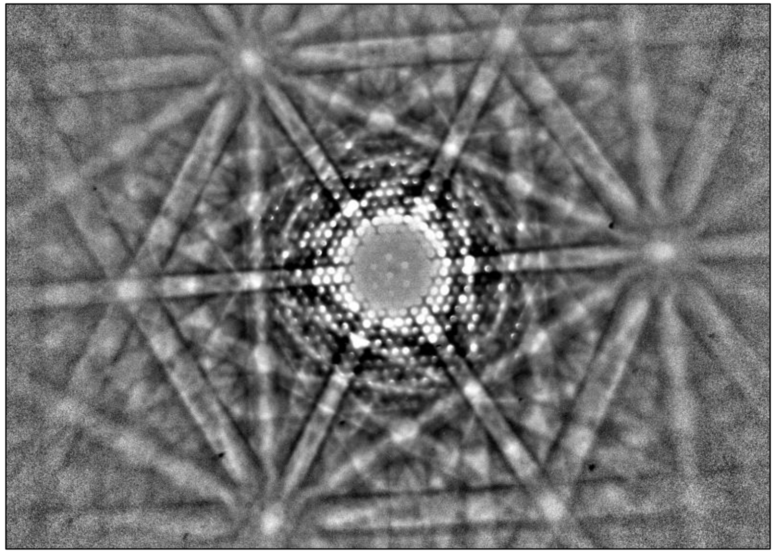
- High electron dose/beam current >> resolution loss
- Sample charging/contamination
- Sensitive to beam instabilities

off-axis vs on-axis TKD

on-axis is the optimum geometry

on-axis TKD



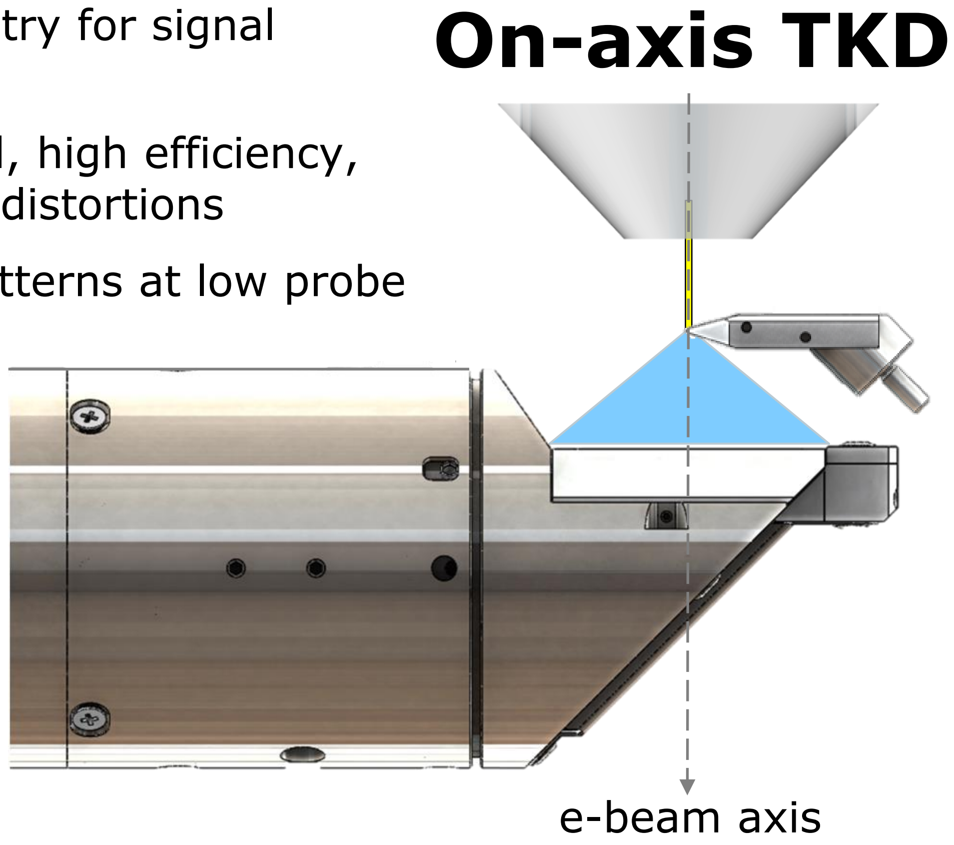


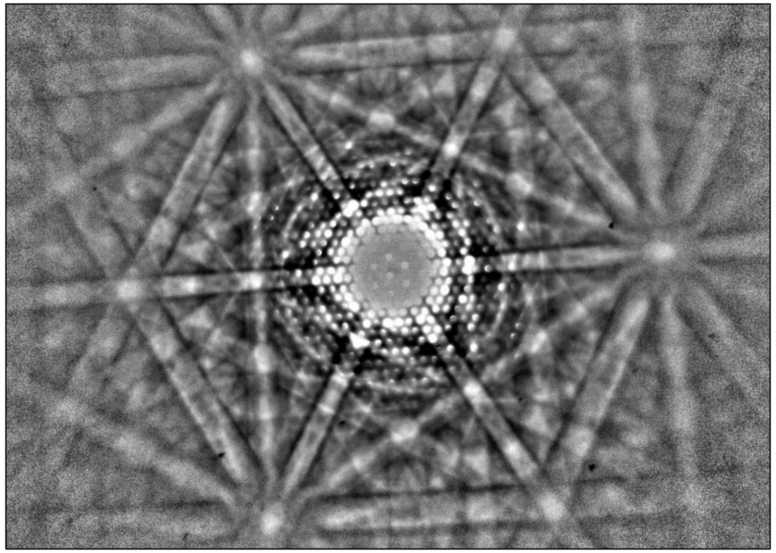
Au Pattern *courtesy of Alice da Silva Fanta, DTU, Denmark*

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

OPTIMUS TKD™
The optimum geometry

- Optimum geometry for signal collection
- 10x better signal, high efficiency, minimal pattern distortions
- High contrast patterns at low probe currents
- High quality data with improved band detection and fast measurement speeds



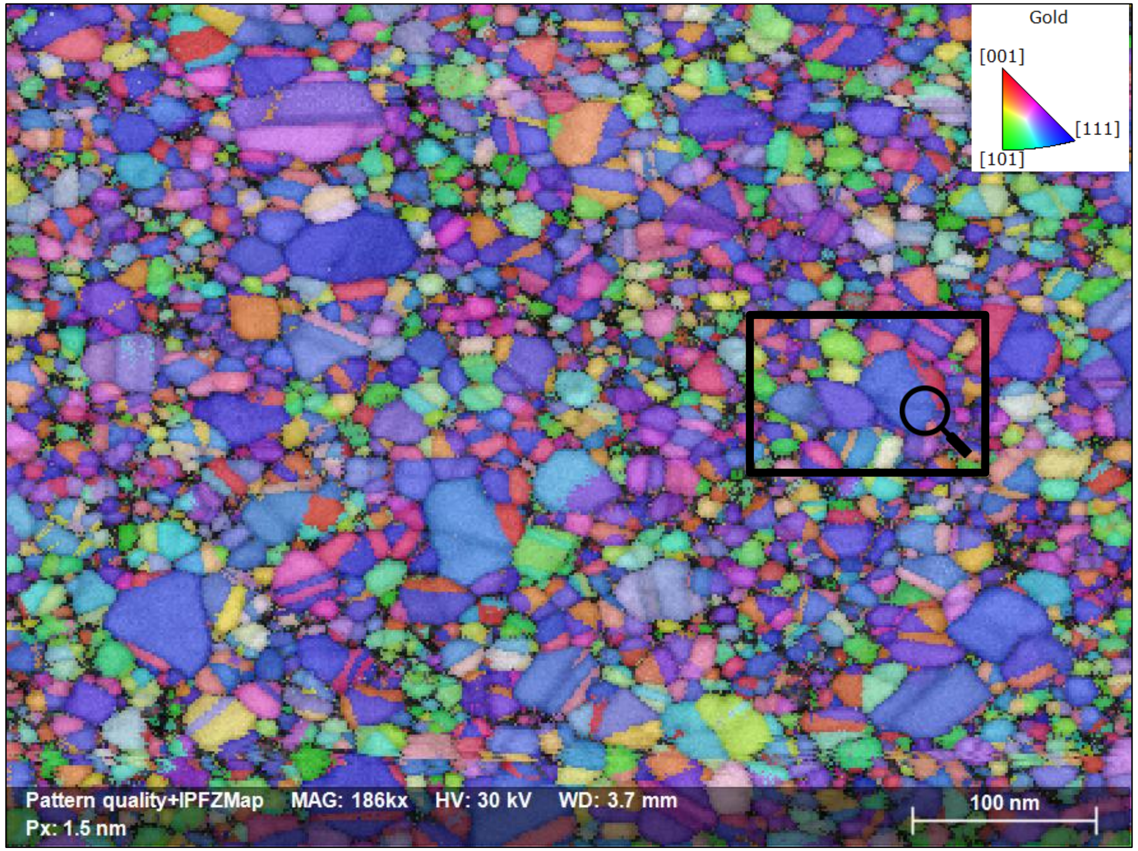


Ref: F. Niessen *et al.*, Ultramicroscopy, 186, 158-170, 2018

OPTIMUS TKD™

Resolving sub-2 nm features with SEM

20nm Au film on 5nm Si_3N_4 membrane



- EHT: 30kV
- Probe current: 2nA
- Step size: 1.5nm
- Acq. speed: 320fps (3ms/point)
- Total acquisition time: 6:31min
- Map size: 126k pixels
- No data cleaning applied
- Zero sol.: 11.5%
- Resolving features <5nm
- Annealing twin: ~**4nm wide**

Sample is courtesy of **Alice Da Silva Fanta** from DTU Nanolab in Copenhagen, Denmark

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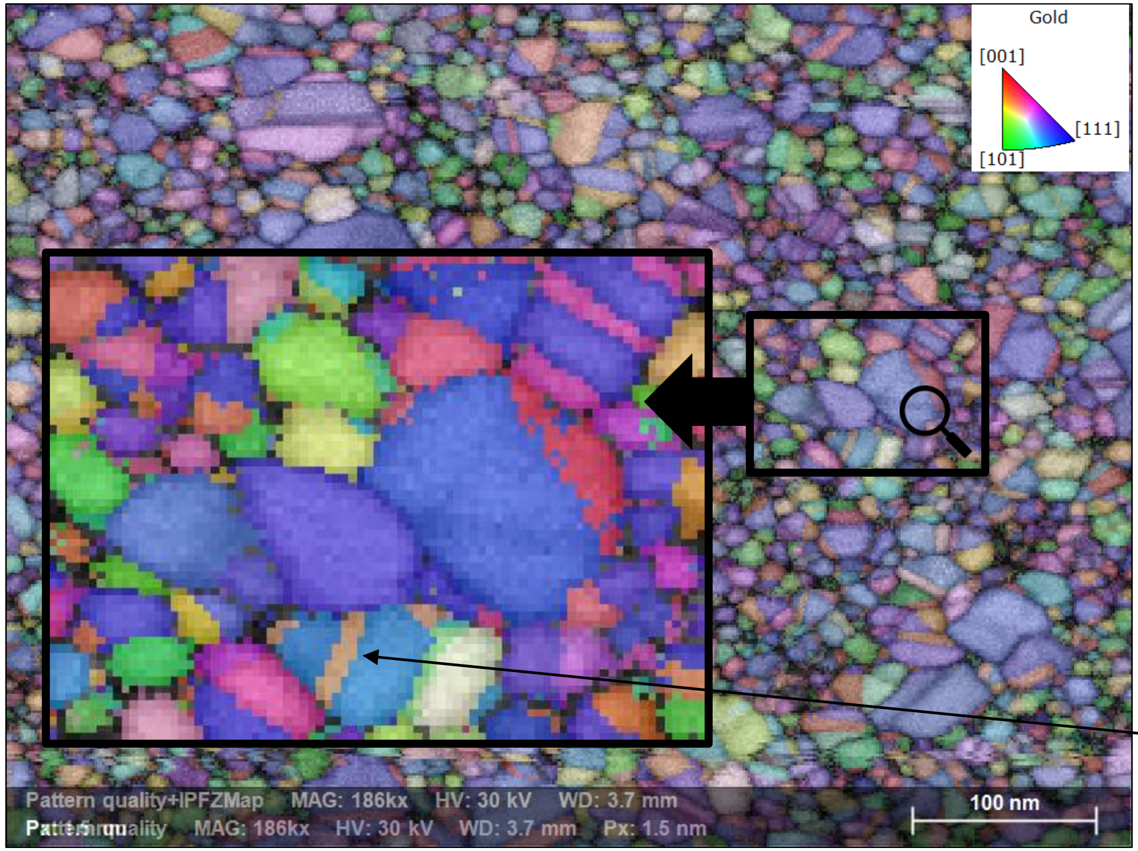

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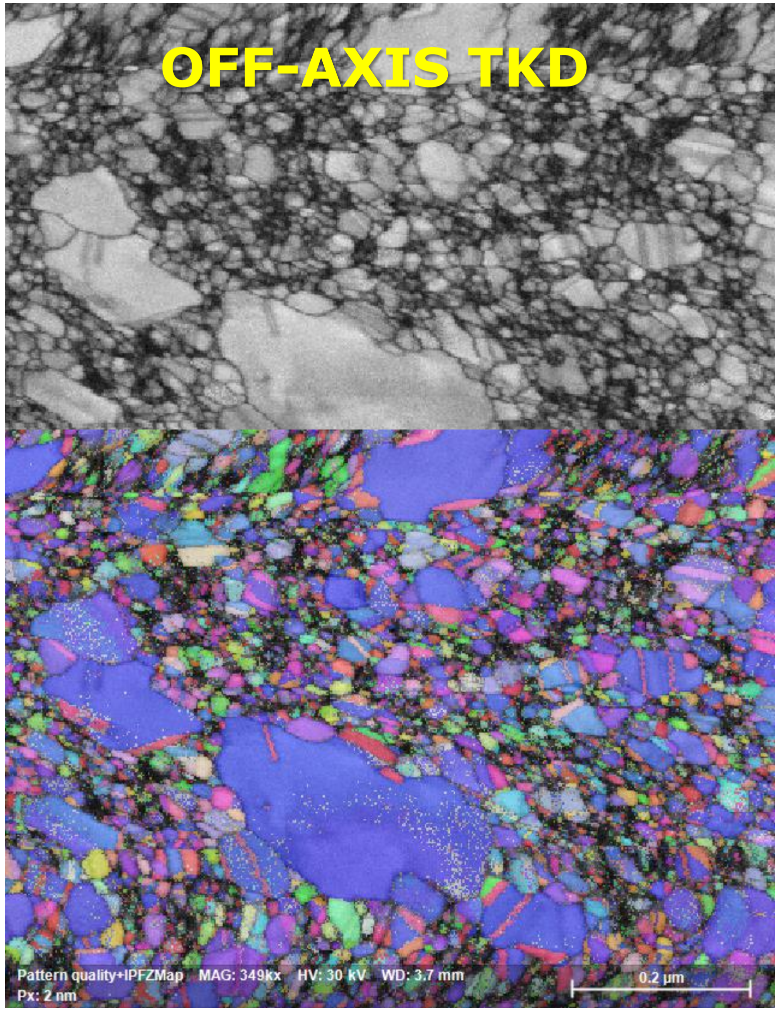
OPTIMUS TKD™ vs off-axis TKD

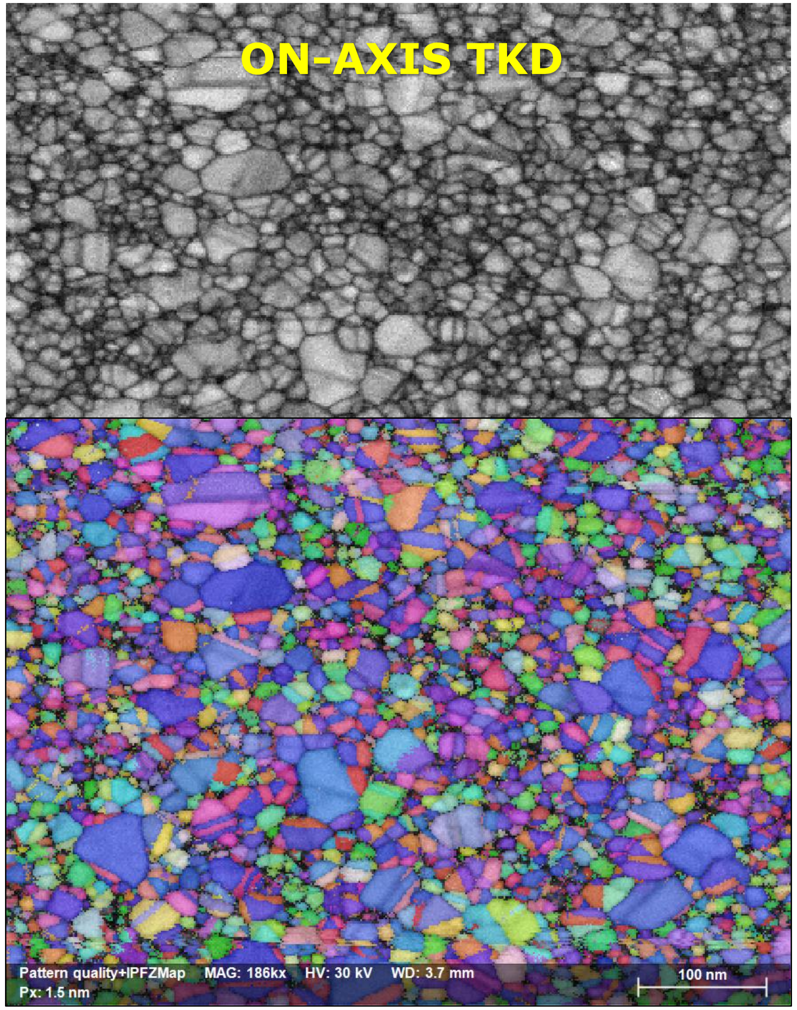
improved speed, spatial resolution and indexing rate



- EHT: 30kV
- Probe current: 2 nA
- Step size: 2 nm

- **Speed: 50 fps vs. 320 fps**
- **Zero sol.: 30 % vs 11.5%**

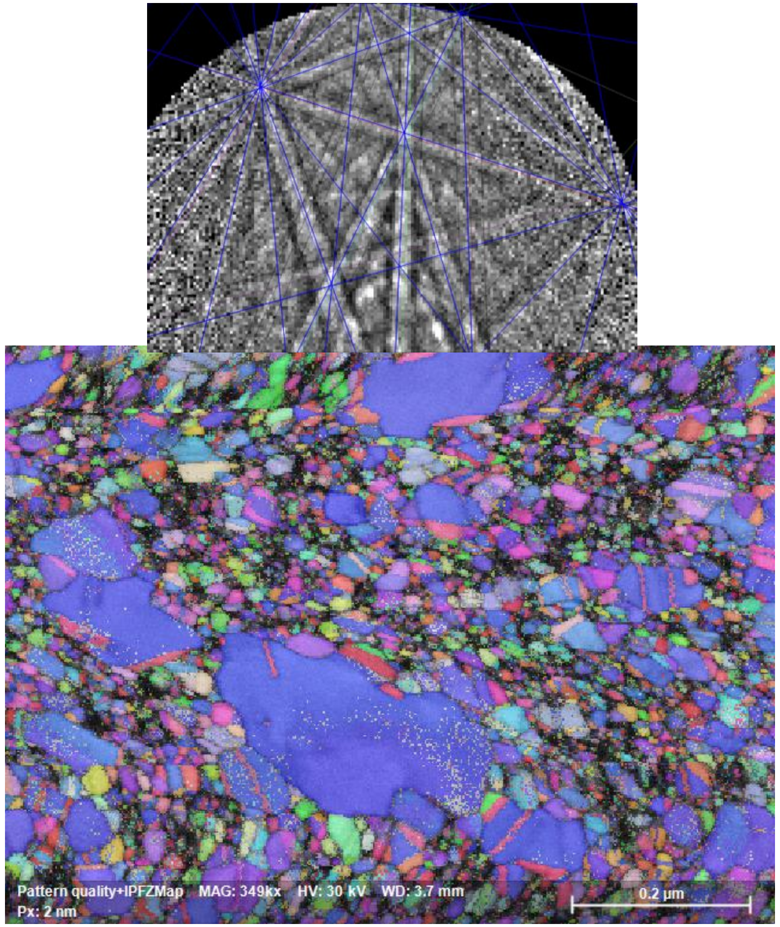
2nm spatial resolution is only achieved with on-axis geometry



OPTIMUS TKD™ vs off-axis TKD

improved speed, spatial resolution and indexing rate

OFF-AXIS TKD

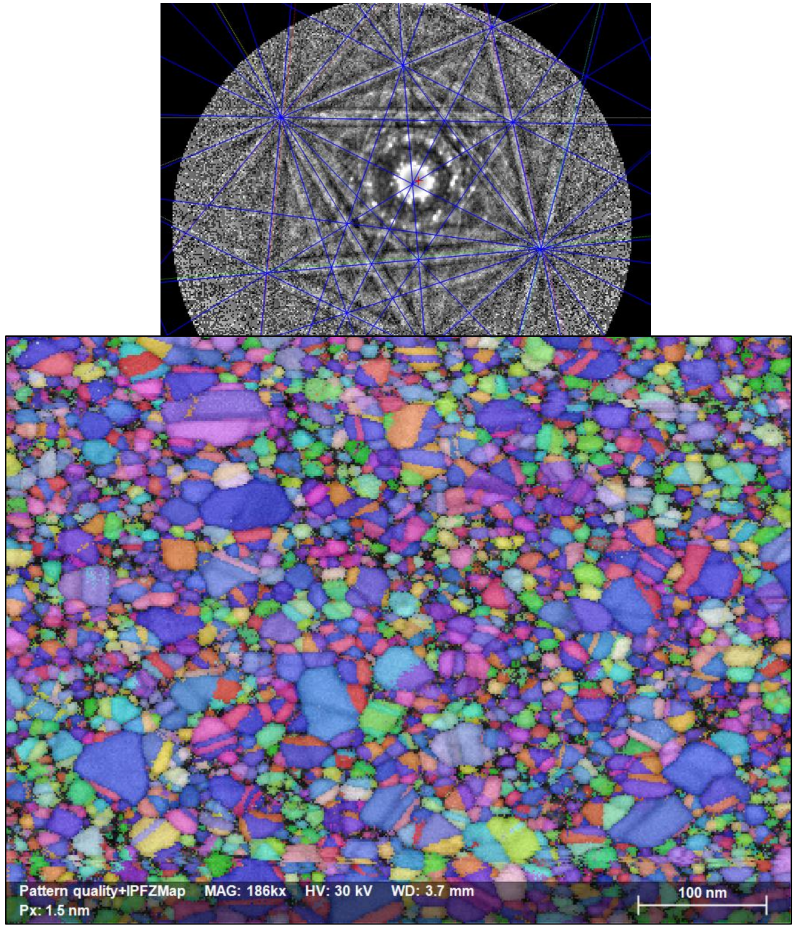


- EHT: 30kV
- Probe current: 2 nA
- Step size: 2 nm

- **Speed: 50 fps vs. 320 fps**
- **Zero sol.: 30 % vs 11.5%**

2nm spatial resolution is only achieved with on-axis geometry

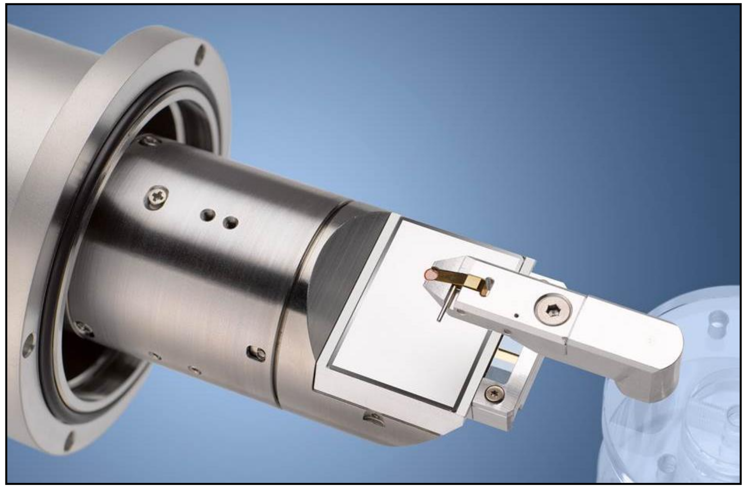
ON-AXIS TKD

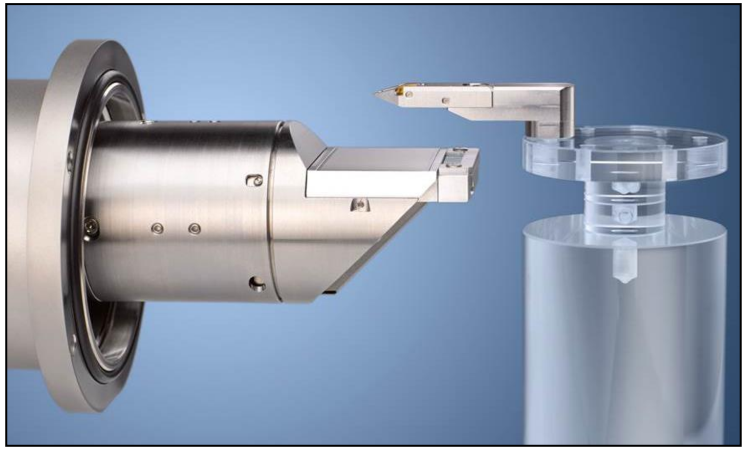


OPTIMUS™ TKD

OPTIMUS TKD™

Complete TKD solution



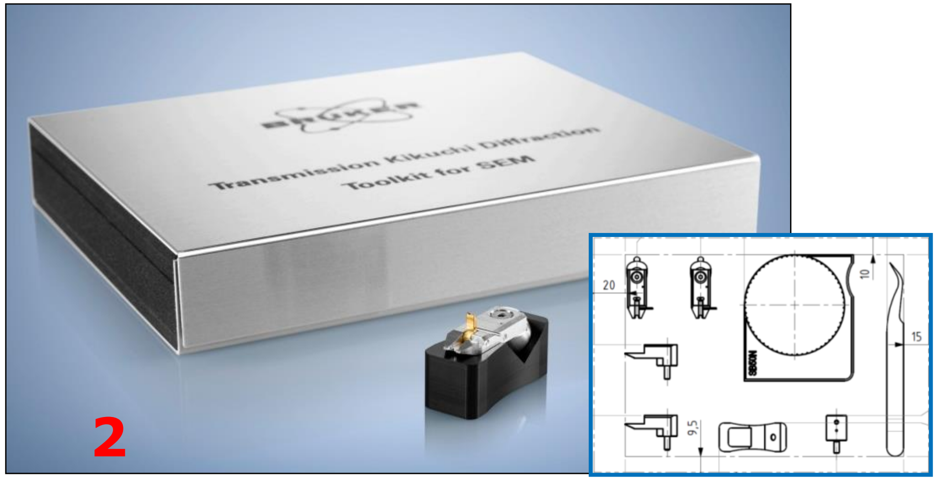


OPTIMUS Main features:

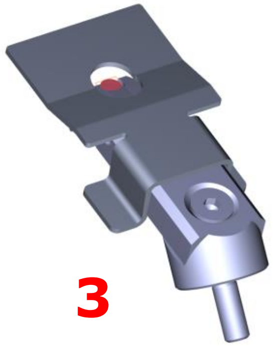
- Horizontal phosphor screen – for capturing the signal around SEM's optical axis
- e- fwd scattered images giving STEM like DF/BF like imaging with Argus™
- Easy to use/switch between EBSD and TKD modes
- Compatible with all existing *e*$^{-}$Flash detectors

OPTIMUS TKD™

Complete TKD solution



- OPTIMUS™ TKD detector head
- Professional TKD toolkit (with patented sample holder)
- X-rella for optimum combined EDS/TKD measurement (patent pending)
- OPTIMUS™ TKD fully integrated in ESPRIT



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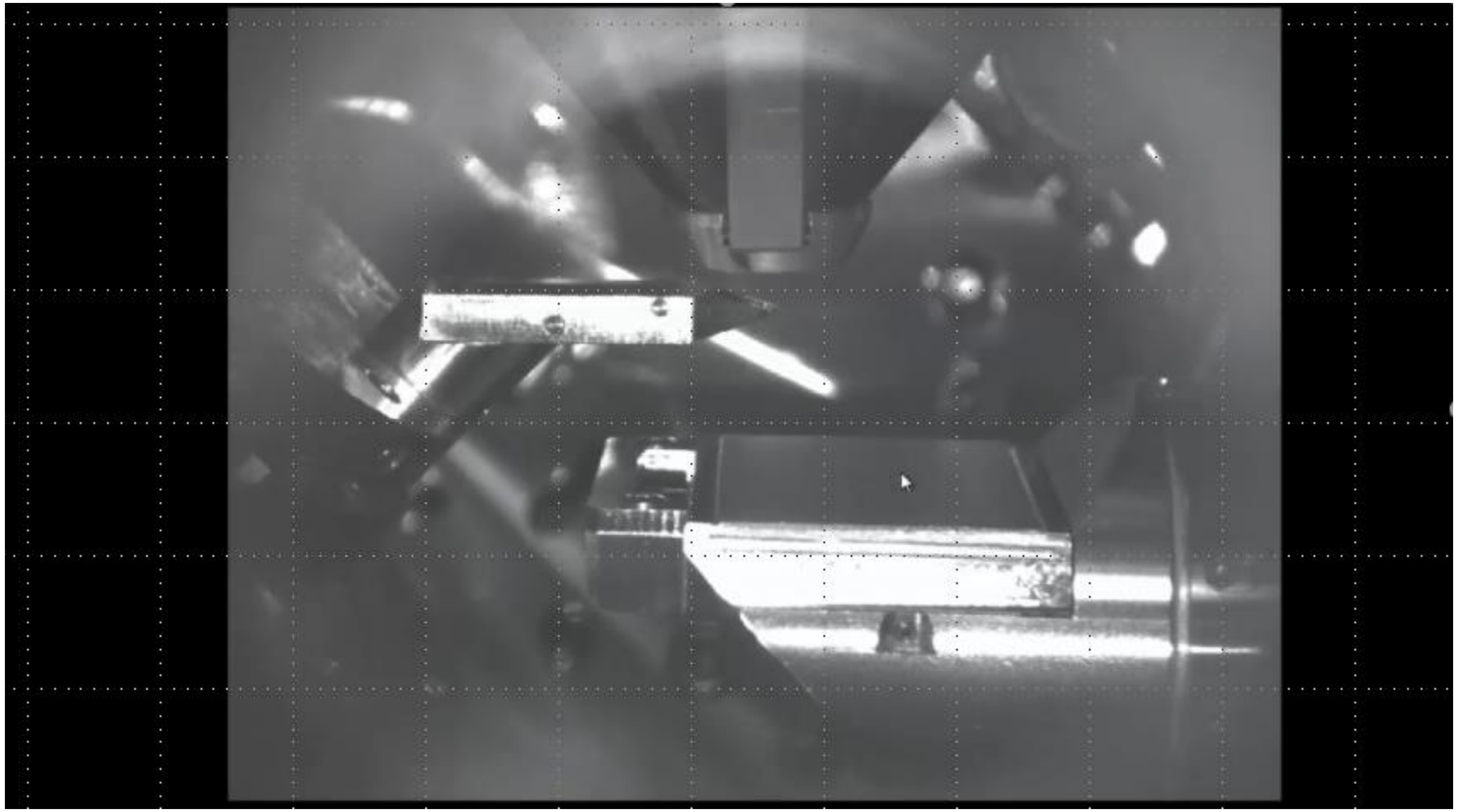
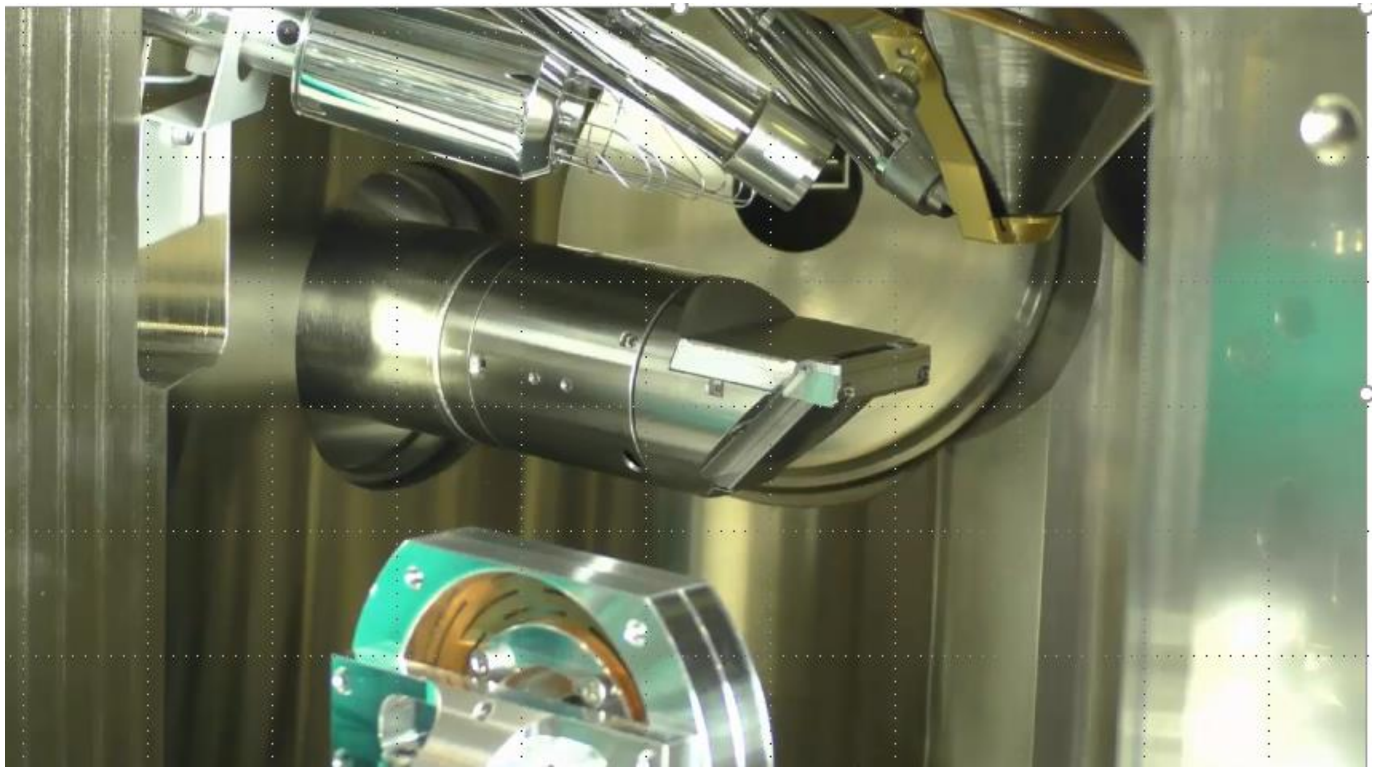
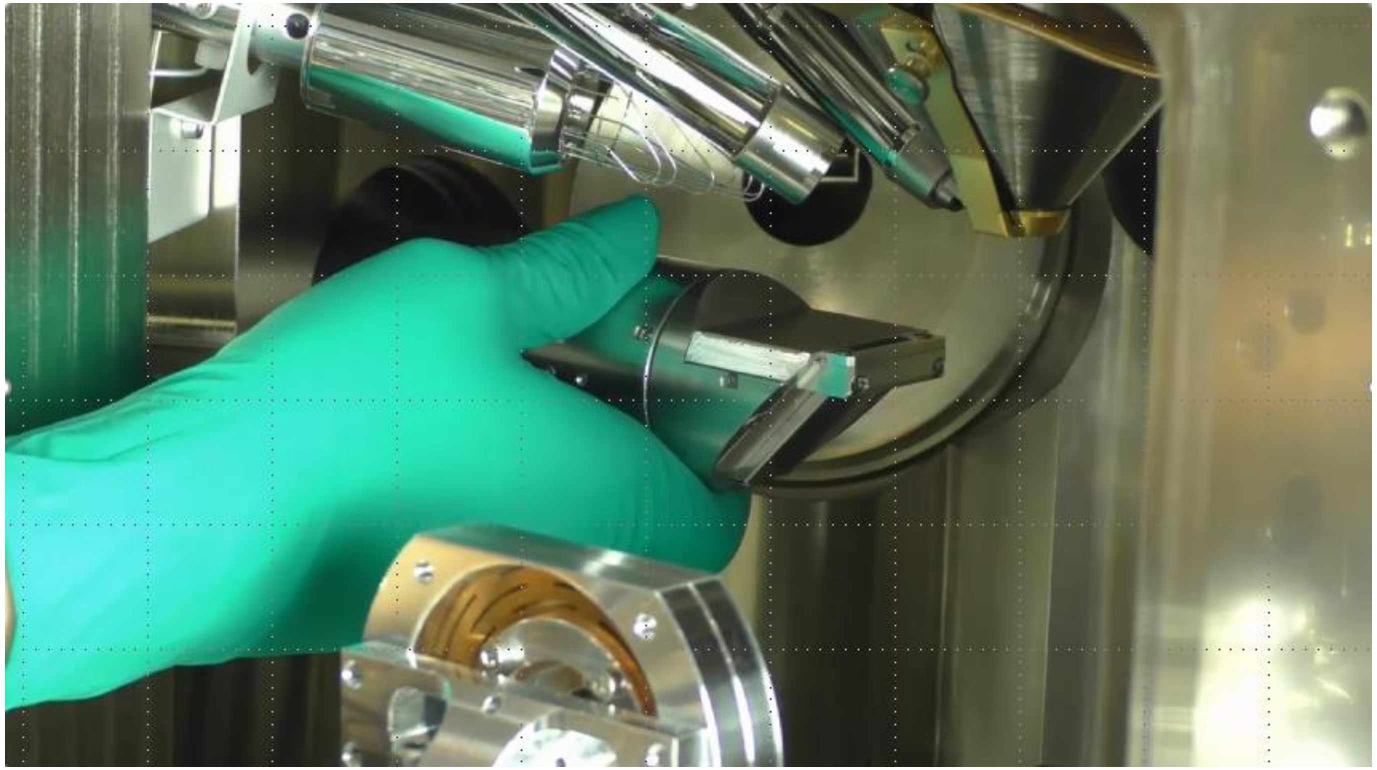
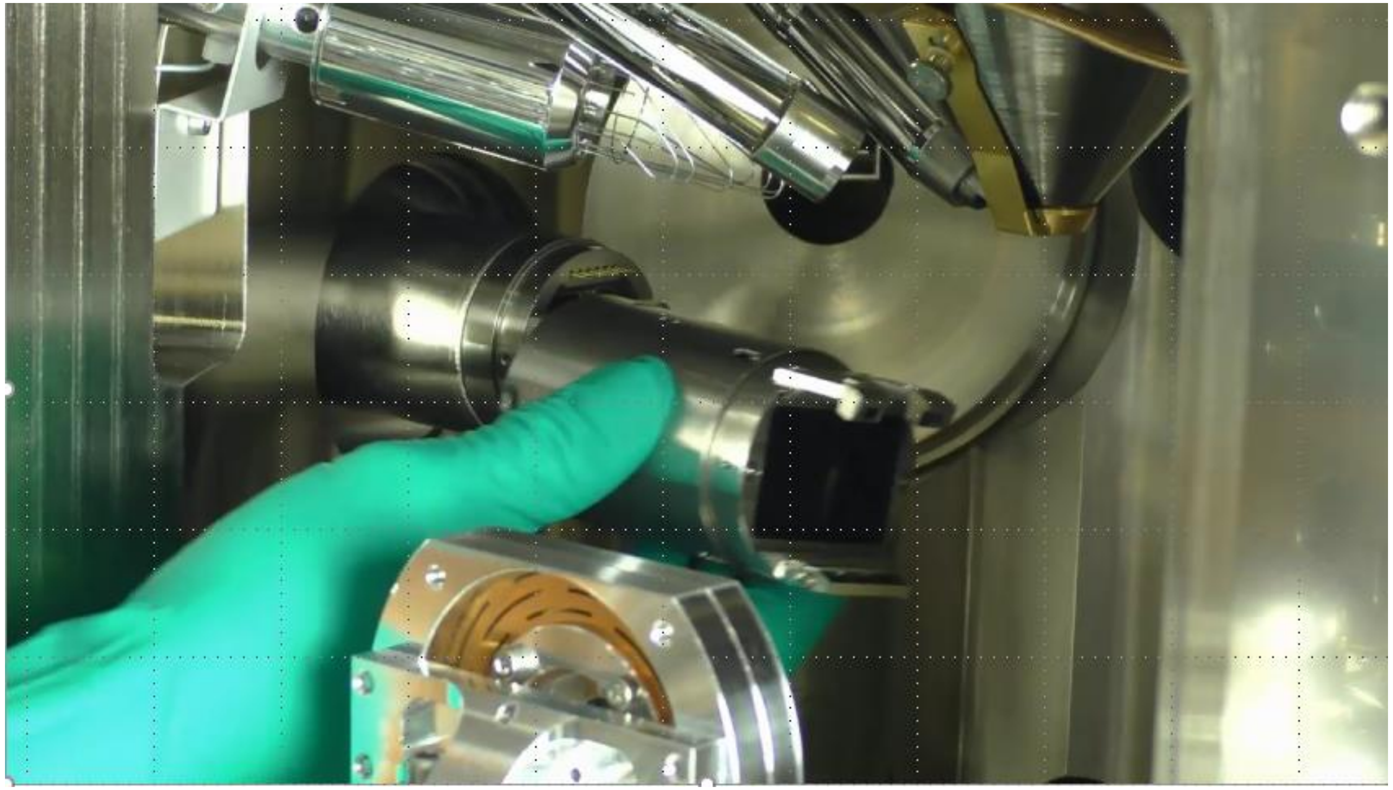
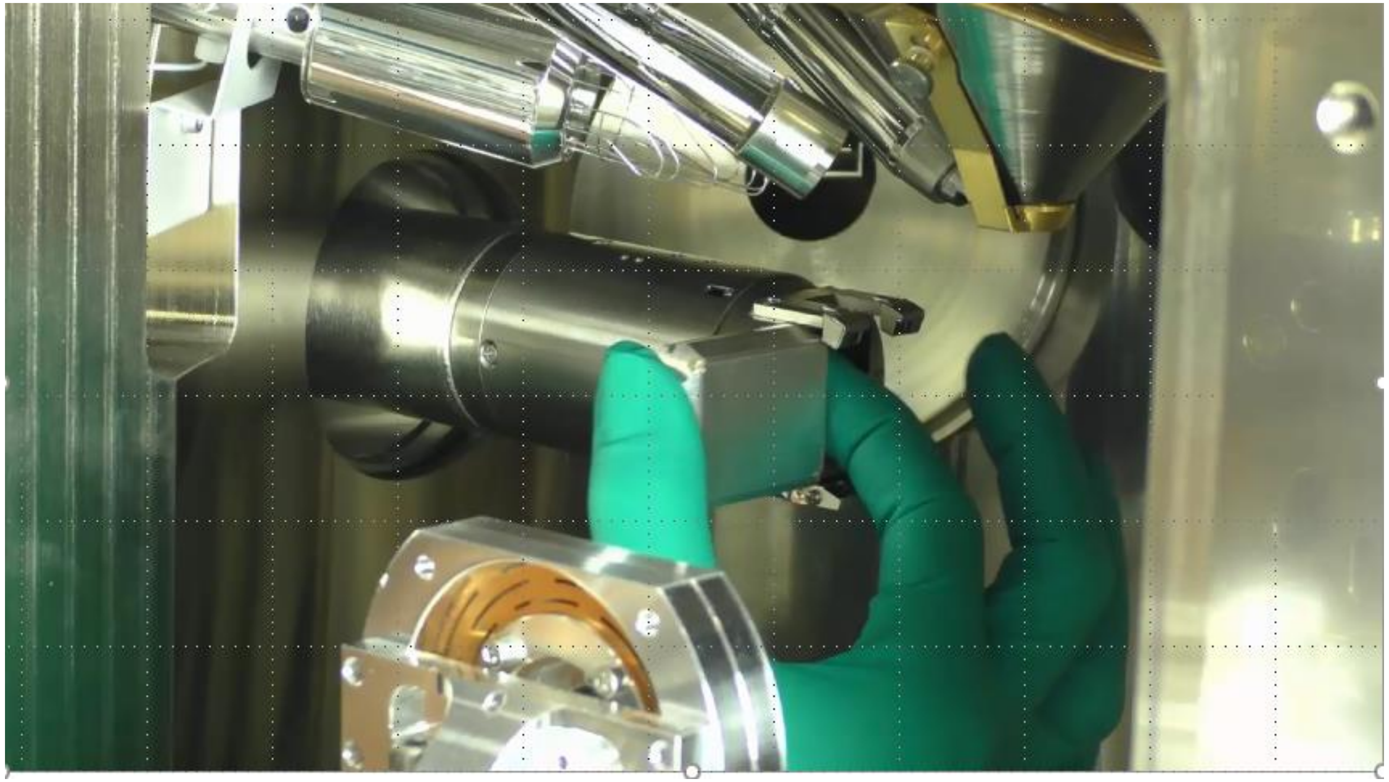










Semiconducting nanostructures

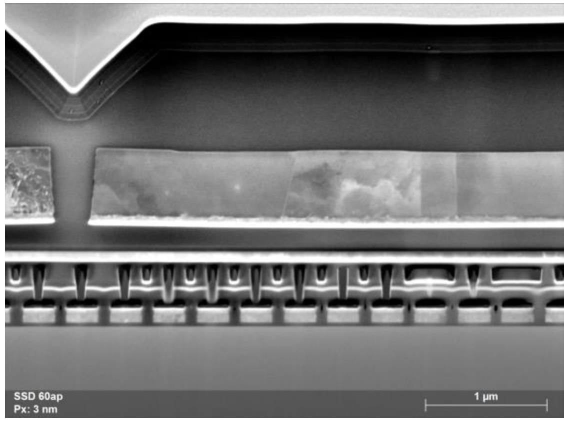
Semiconducting nanostructures
Challenges

- Grain/feature/layer size < 200 nm: spatial resolution < 10 nm
- Need low probe current measurements → (+) Resolution (-) drift
- Off axis mode: signal overlap/low signal at GB or interfaces of different layers

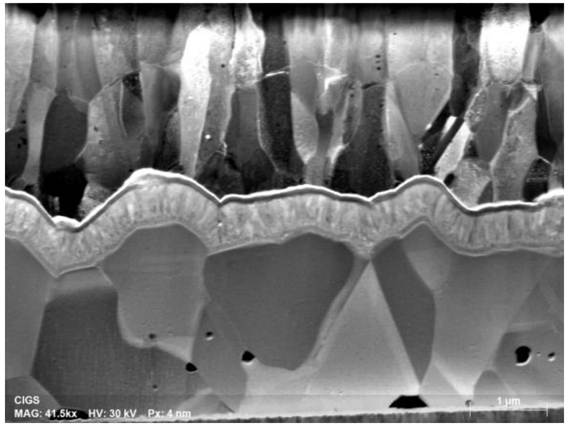
OPTIMUS™ TKD & *e*-Flash^{FS}

- Spatial resolution: unmatched spatial resolution < 2nm
- Fast: high speed TKD with *e*-Flash^{FS}
- Sensitive: speed and data quality not compromised even at low beam currents (e.g. 2 nA)

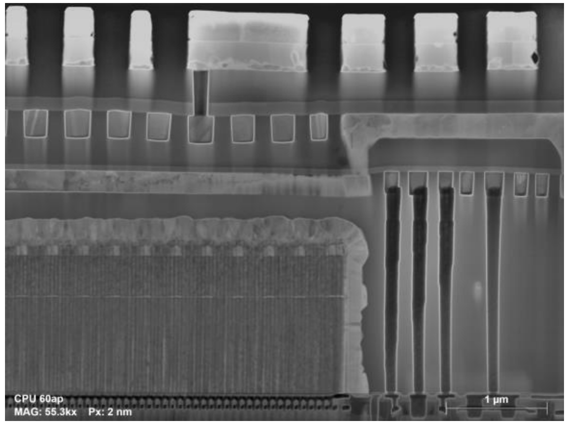
Application examples
STEM bright field images



Solid state drive



CIGS solar cell

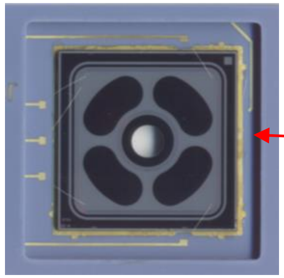


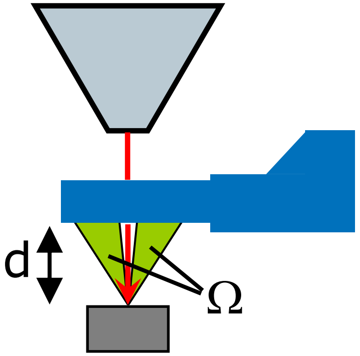
Microprocessor/FinFET

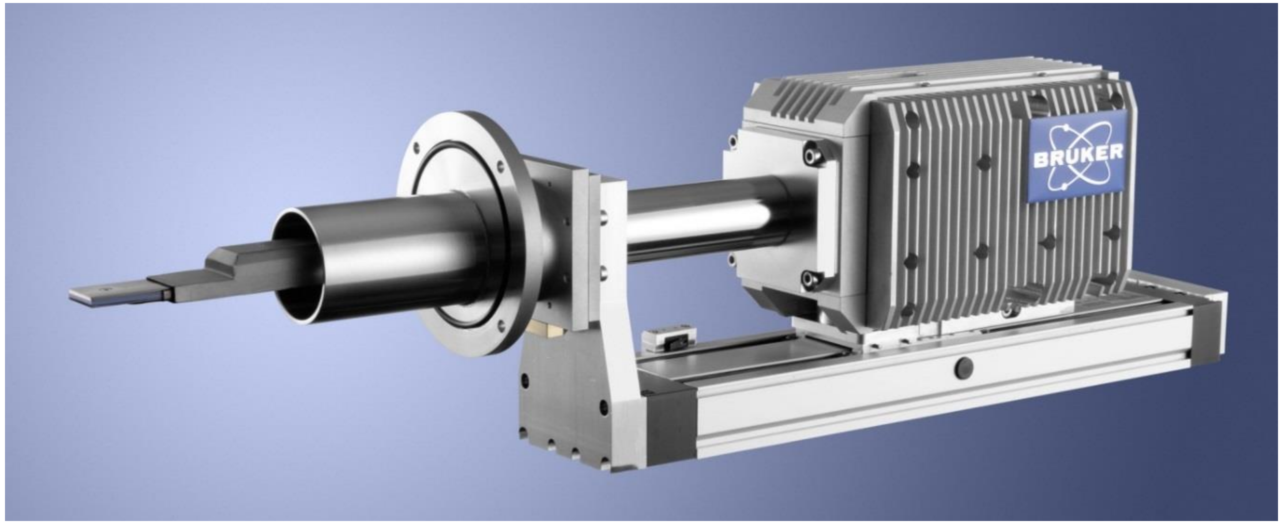
- FIB Lamellae
- Lift outs from bulk samples using Ga-ion FIB
- Trenching/coarse thinning at 30 kV
- Fine milling at 5 kV and 2 kV
- Final sample thickness: 40nm - 70nm
- TKD: OPTIMUS TKD head mounted on *e*⁻Flash^{FS} detector
- EDS: FlatQUAD annular EDS detector

TKD/EDS simultaneous analysis

XFlash FlatQUAD





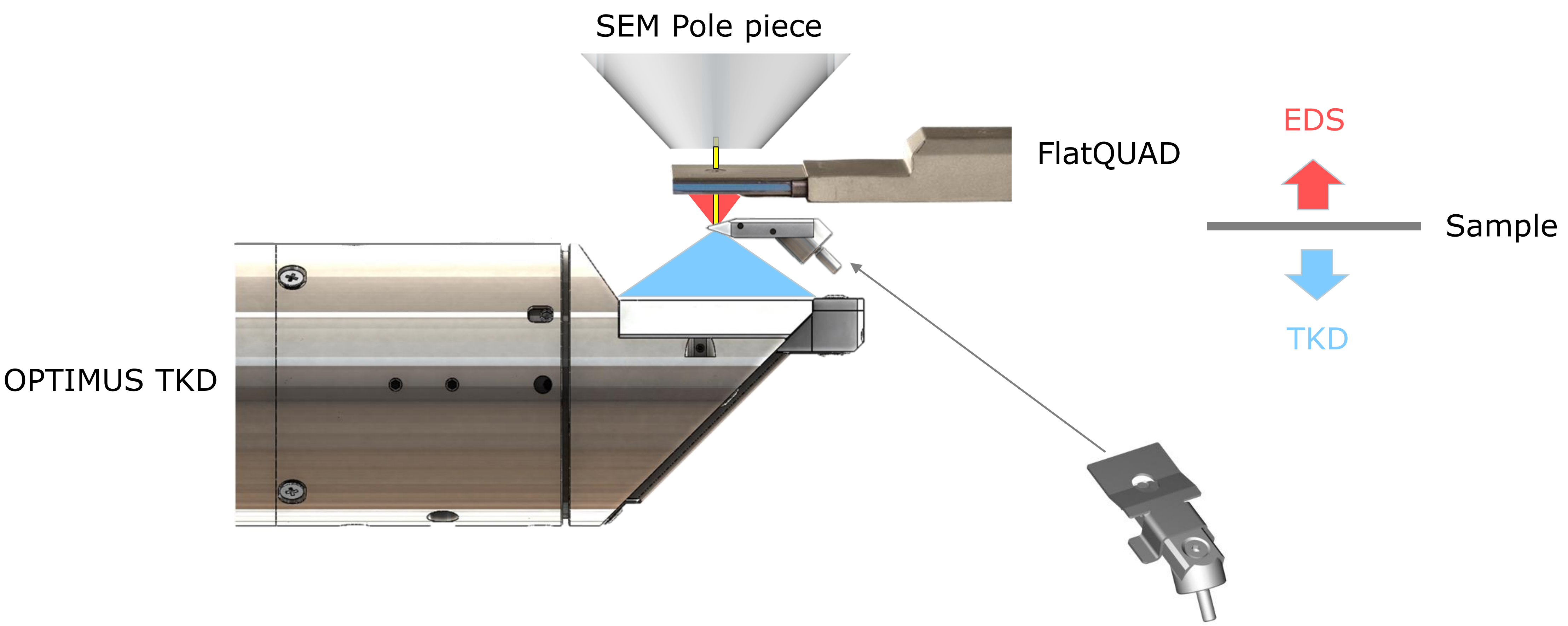


XFlash FlatQuad EDS detector:

- Side entry, annular design
- Central aperture for primary beam
- 4x15mm²
- Highest solid angle (up to 1.1 sr)
- High count rate at low beam currents
- Ideal for samples with low x-ray yield → light elements

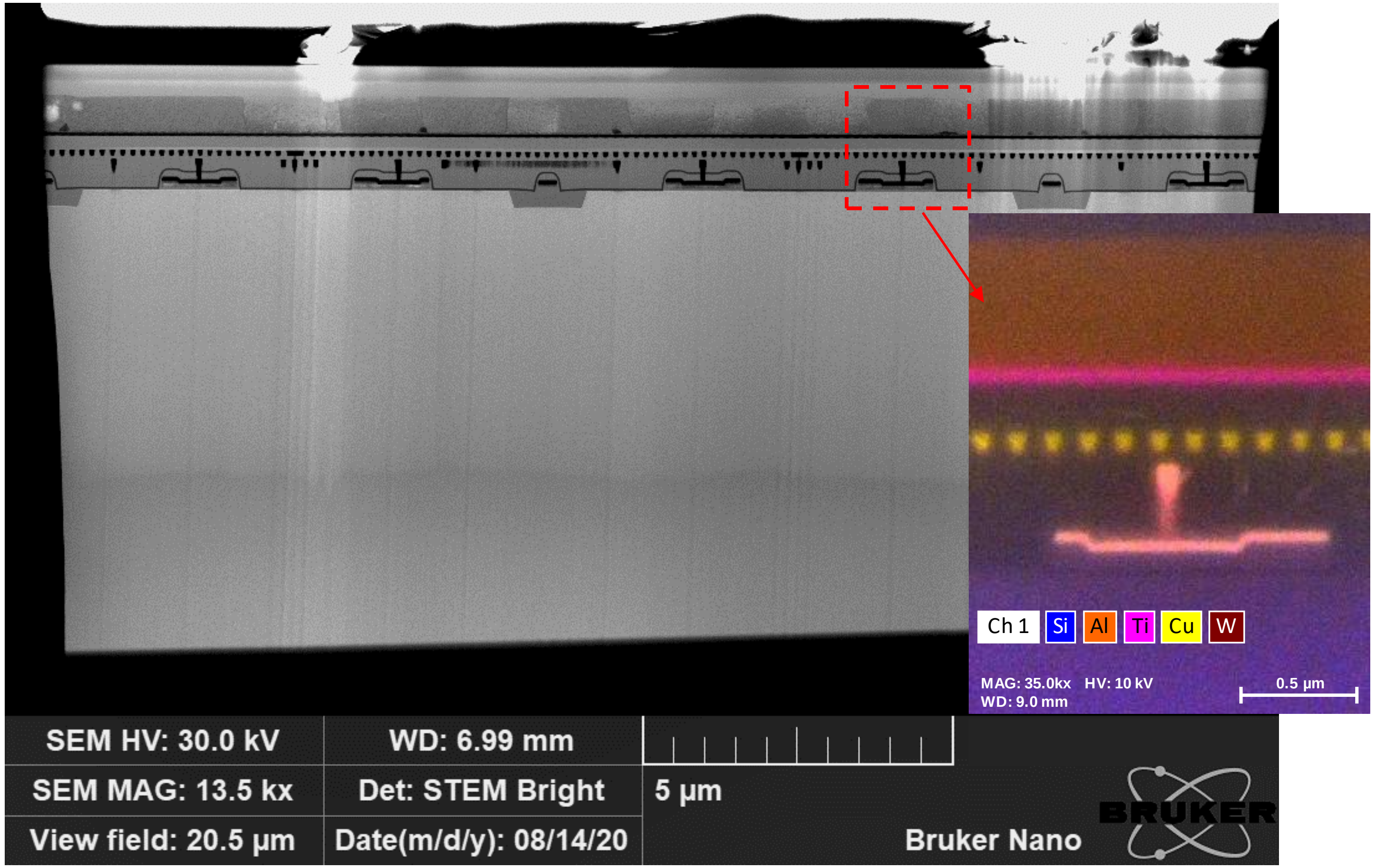
TKD/EDS simultaneous measurement

XFlash FlatQUAD



Solid-state memory device (SSD): FIB lamella

STEM-BF and EDS map

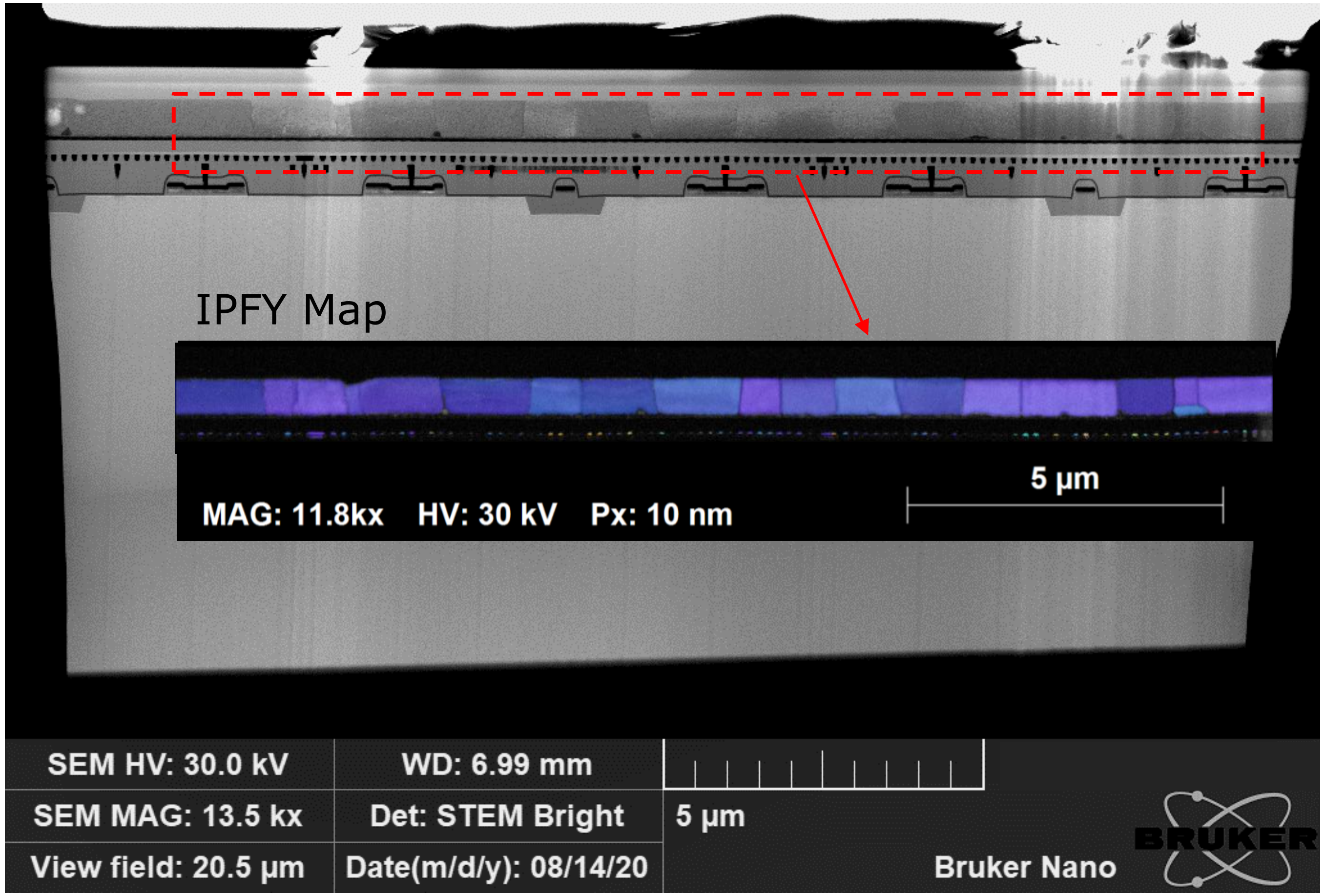


EDS mapping parameters:

- Det: FlatQUAD
- High voltage: 10 kV
- Abs. current: 910 pA
- WD: 11 mm
- Map size: 82.6 K pixels
- **Map time: 30 s**
- Input counts: 224.3 Kcps
- Output counts: 153.3 Kcps
- **Total counts: 4.65E+06**
- DT: 32%

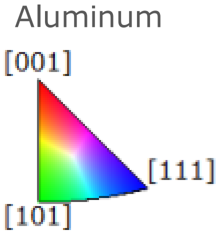
Solid-state memory device (SSD): FIB lamella

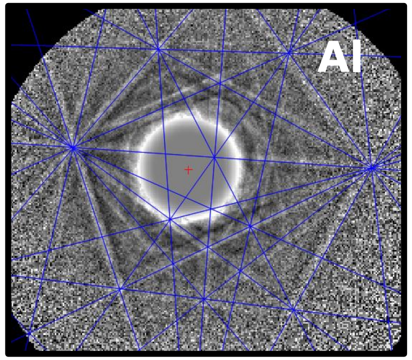
STEM-BF and IPF maps



TKD mapping parameters:

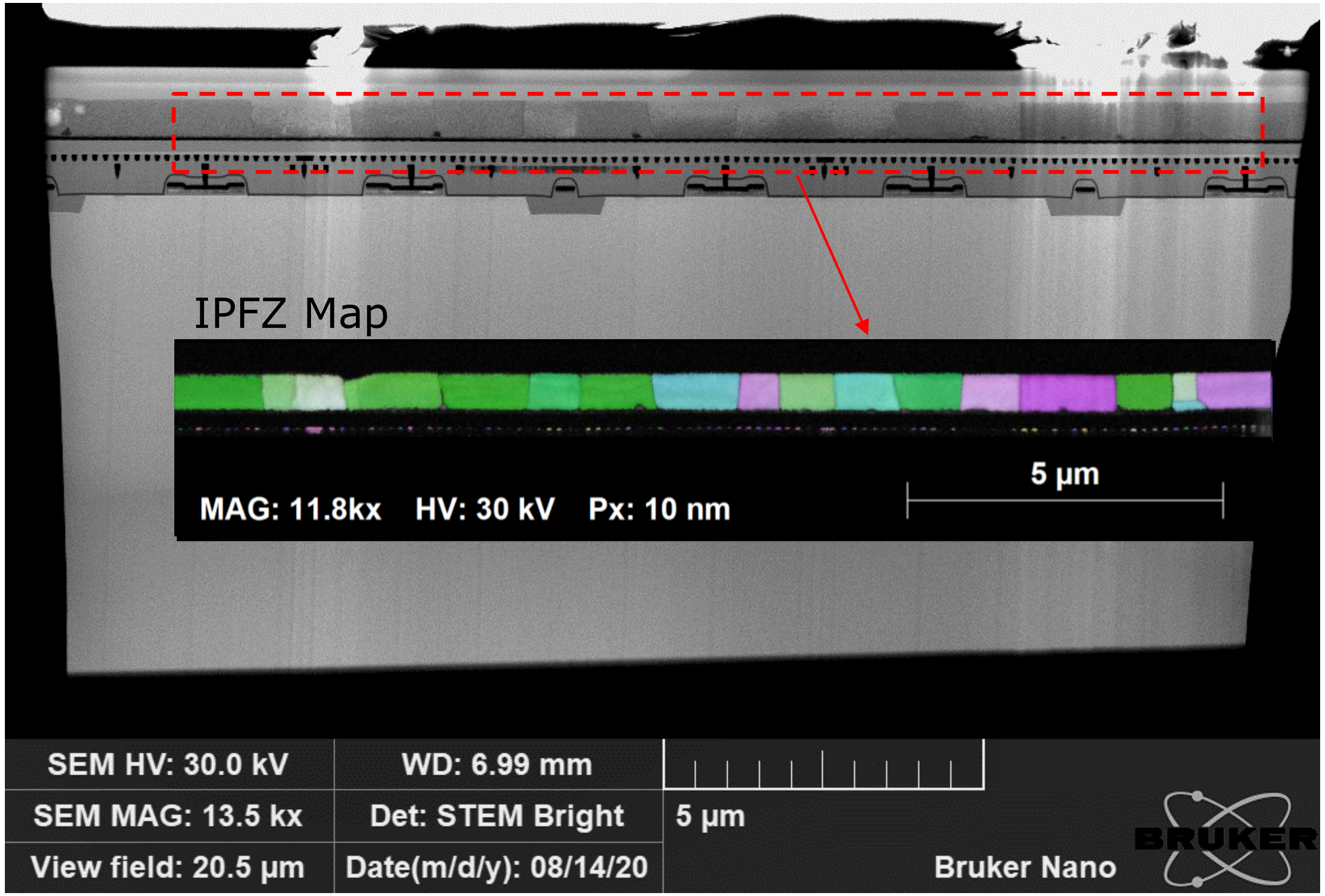
- EHT: 30kV
- Probe current: 1.6 nA
- Step size: 10nm
- Acq. speed: 161.4 fps
- Map size: 258k pixels
- **Zero sol.: 1.9% Al layer**





Solid-state memory device (SSD): FIB lamella

STEM-BF and IPF maps



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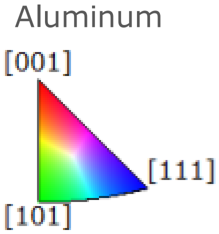
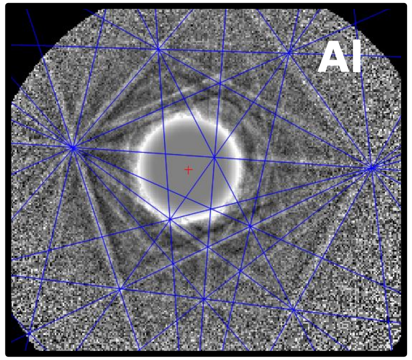
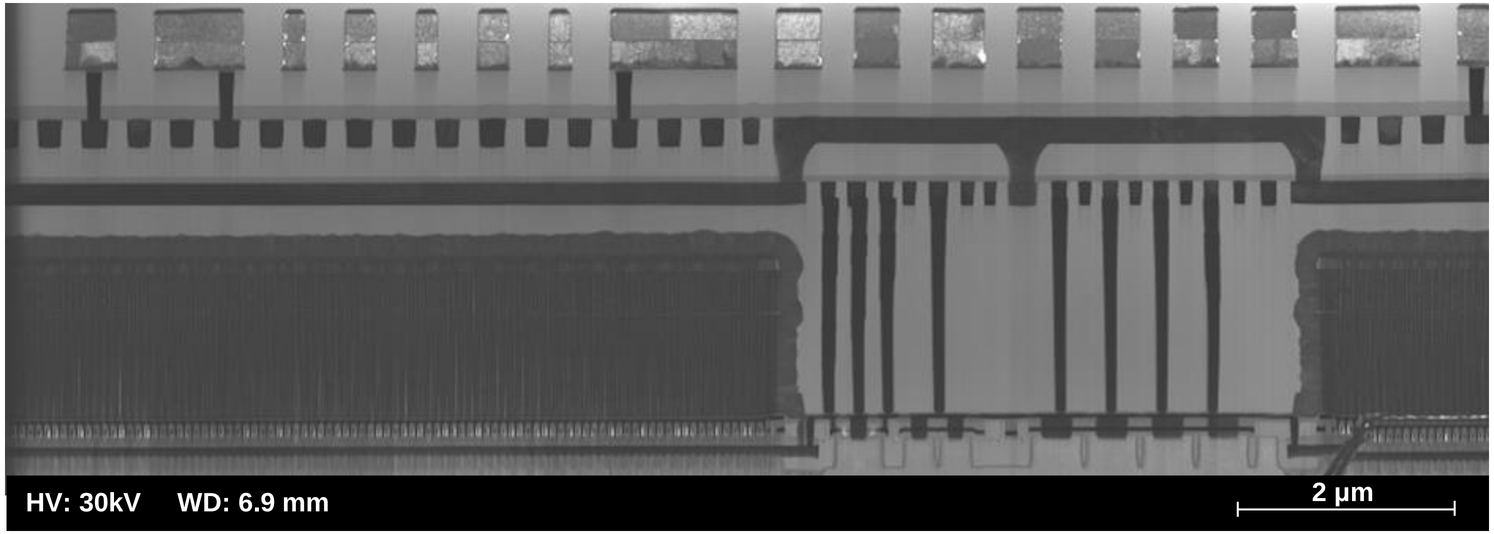




Microprocessor/FinFET: Large area mapping

STEM bright field and EDS map



Microprocessor/FinFET: Large area mapping

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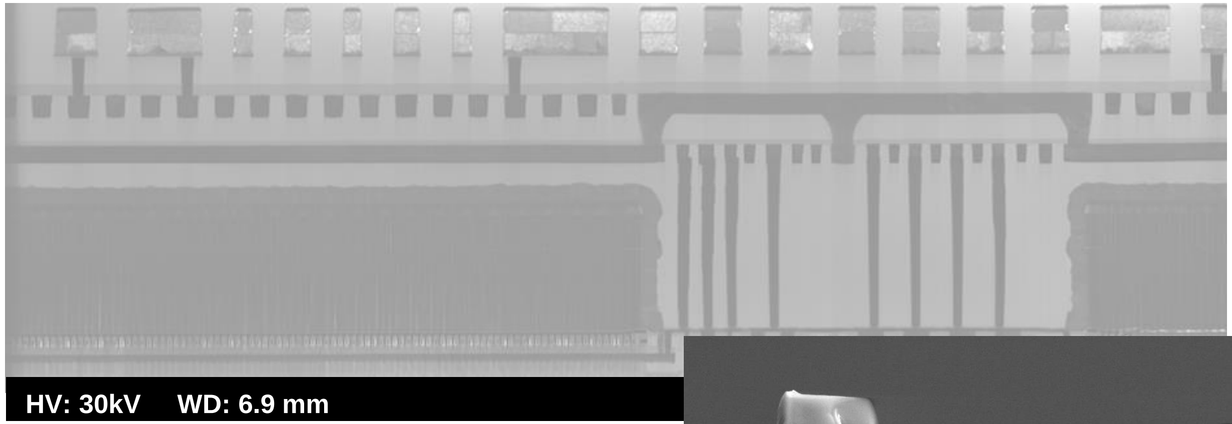

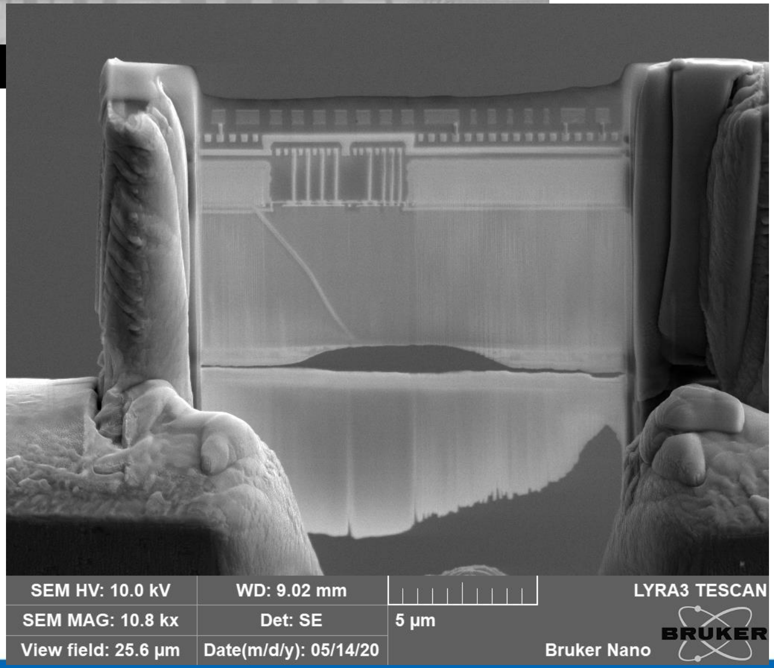

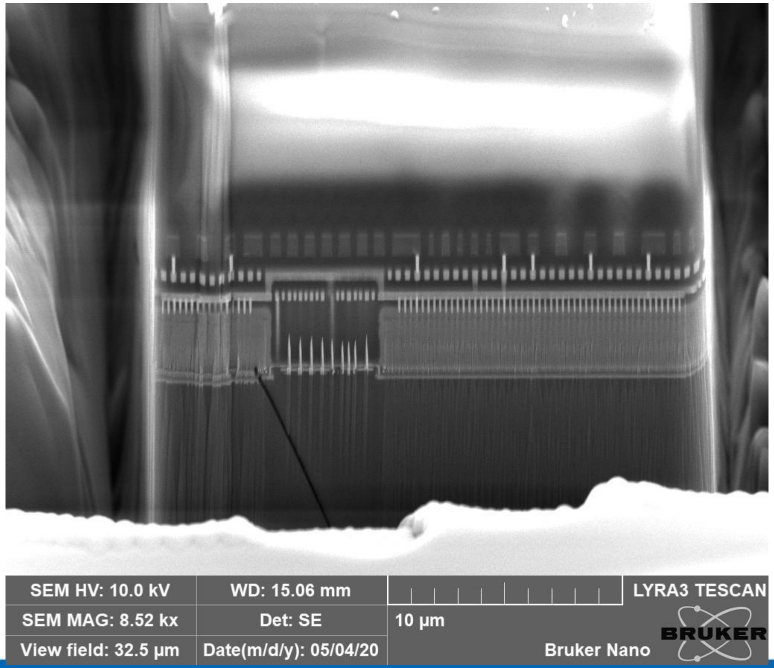

Microprocessor/FinFET: Large area mapping

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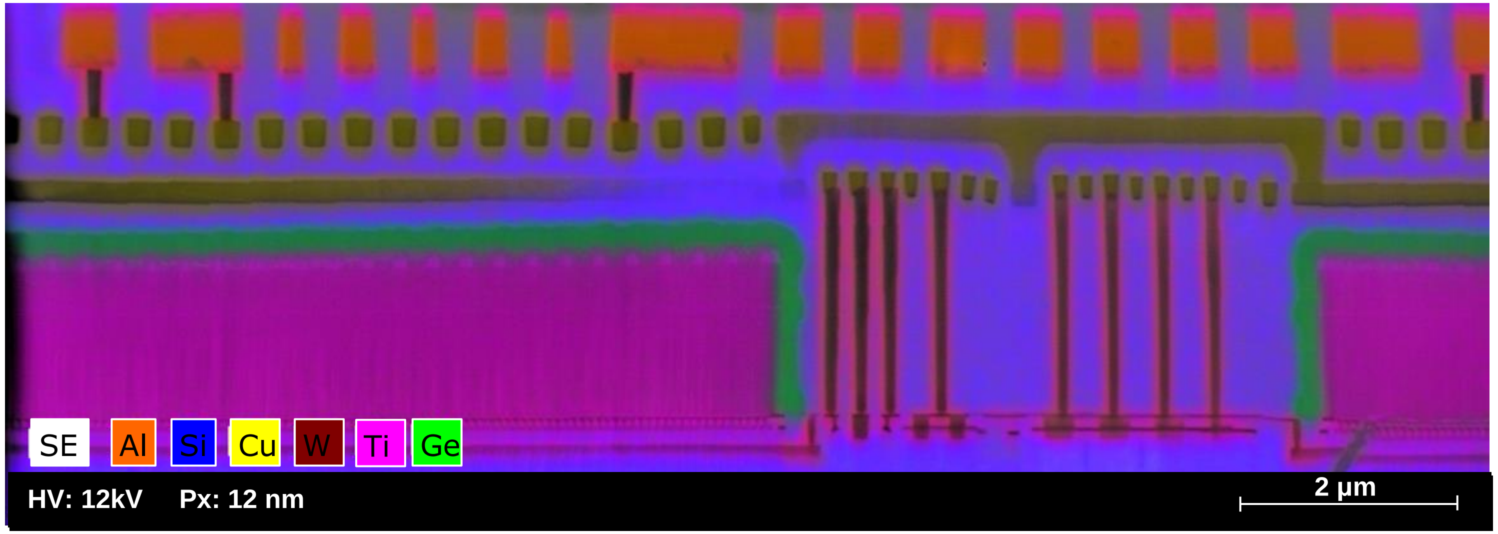
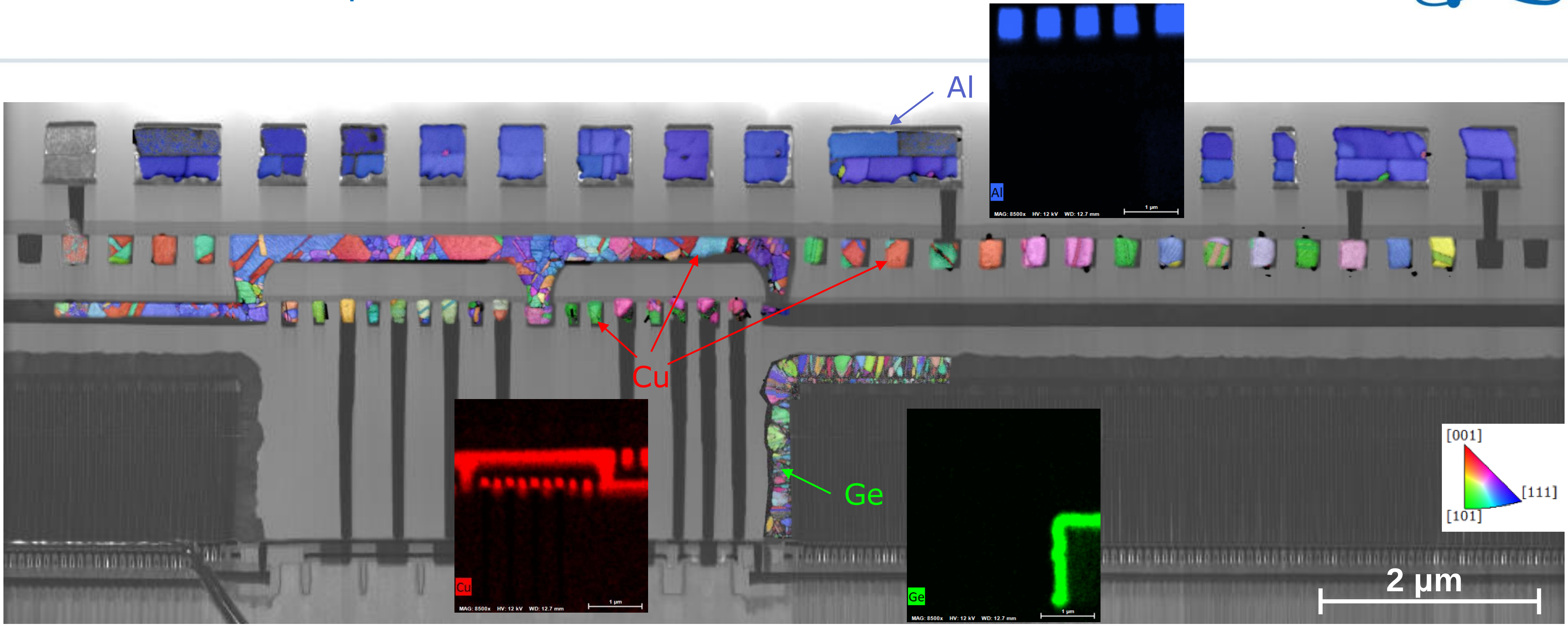


EDS mapping parameters:

- High voltage: 12 kV
- WD: 11 mm
- Map size: 4.88E+05 pixels
- Map size: 13.67 × 4.80 µm
- **Acq. time: 300 s**
- Input counts: 504 Kcps
- Output counts: 188.5 Kcps
- **Total counts: 5.74E+07**
- DT: 49%

Microprocessor/FinFET: Large area mapping

STEM and IPFY maps



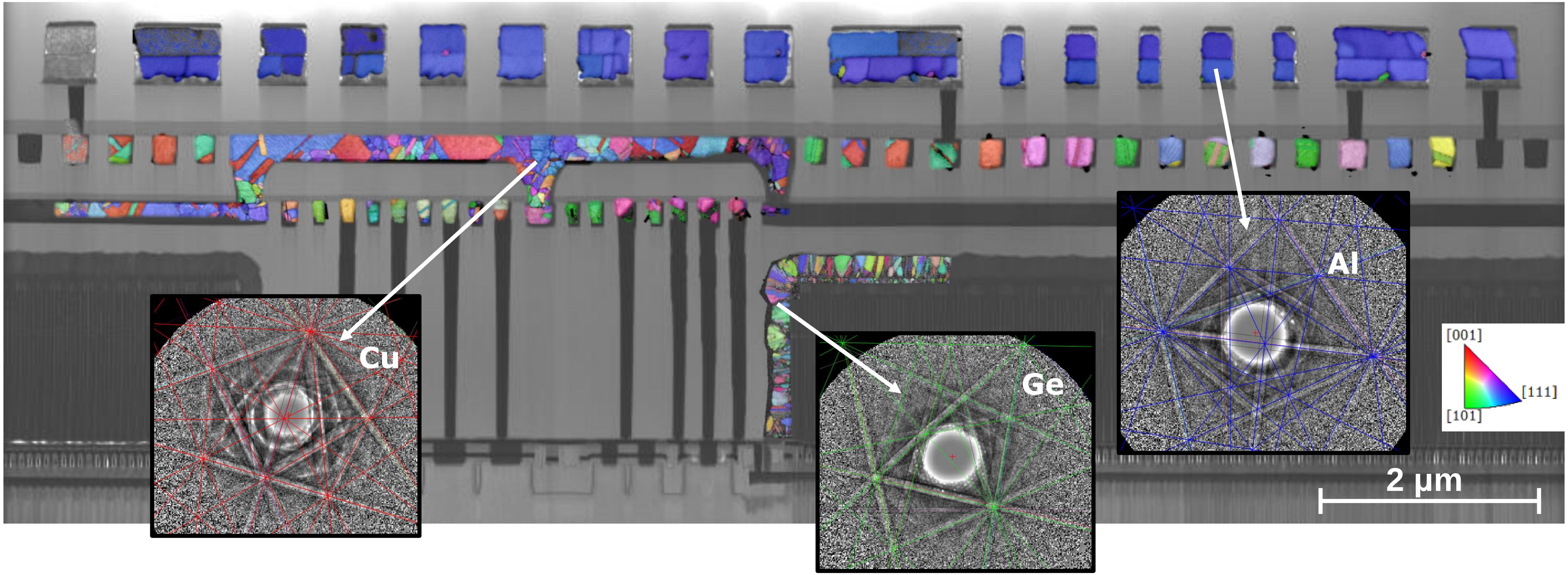
Cu mapping parameters:
- Step size: 5nm
- Acq. speed: 139 fps
- Map size: 198 k pixels

Ge mapping parameters:
- Step size: 3nm
- Acq. speed: 148 fps
- Map size: 38.8 k pixels

Al mapping parameters:
- Step size: 5nm
- Acq. speed: 328 fps
- Map size: 515 k pixels

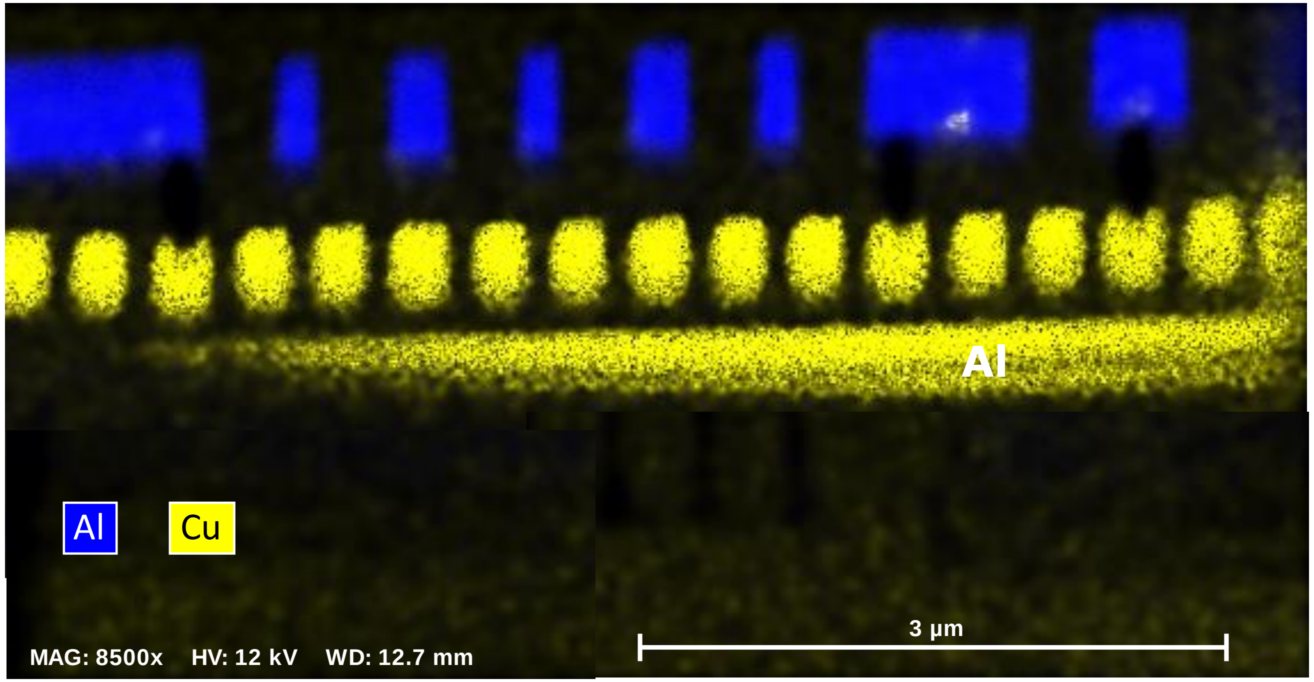
Microprocessor/FinFET: Large area mapping

STEM and IPFY maps



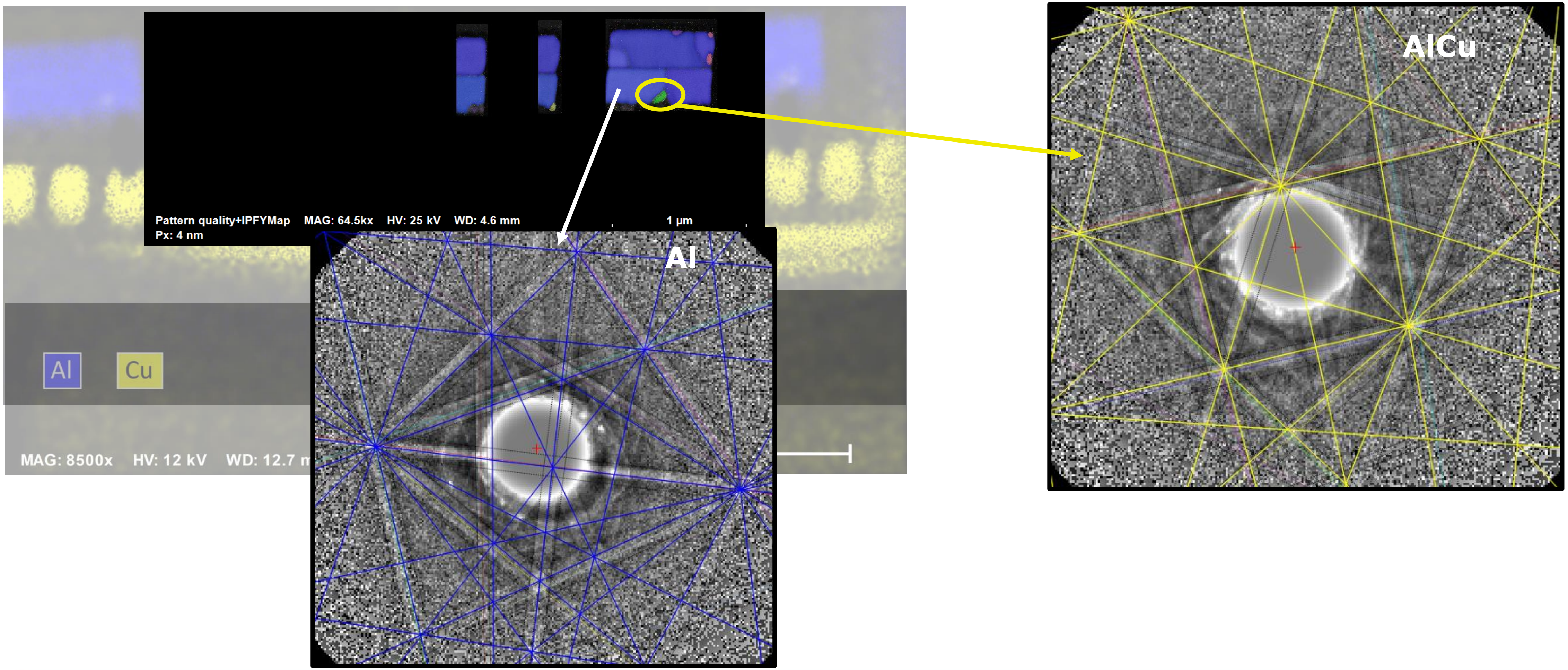
Microprocessor/FinFET: impurities/intermetallics

Cu intermetallics in Al layer



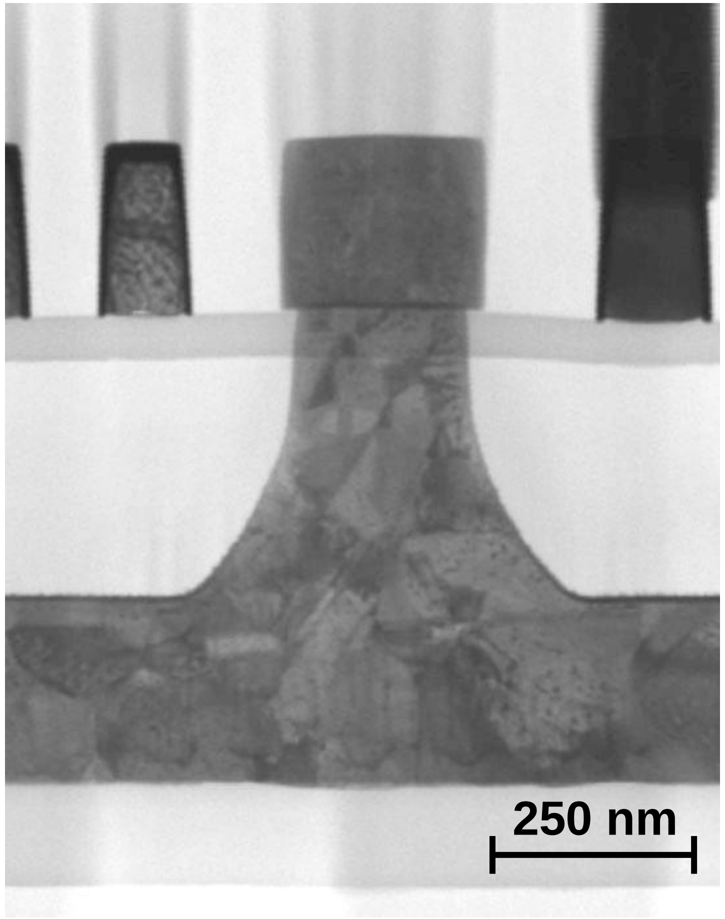
Microprocessor/FinFET: impurities/intermetallics

Cu intermetallics in Al layer



Microprocessor/FinFET: spatial resolution

STEM and IPFY map on Cu interconnect

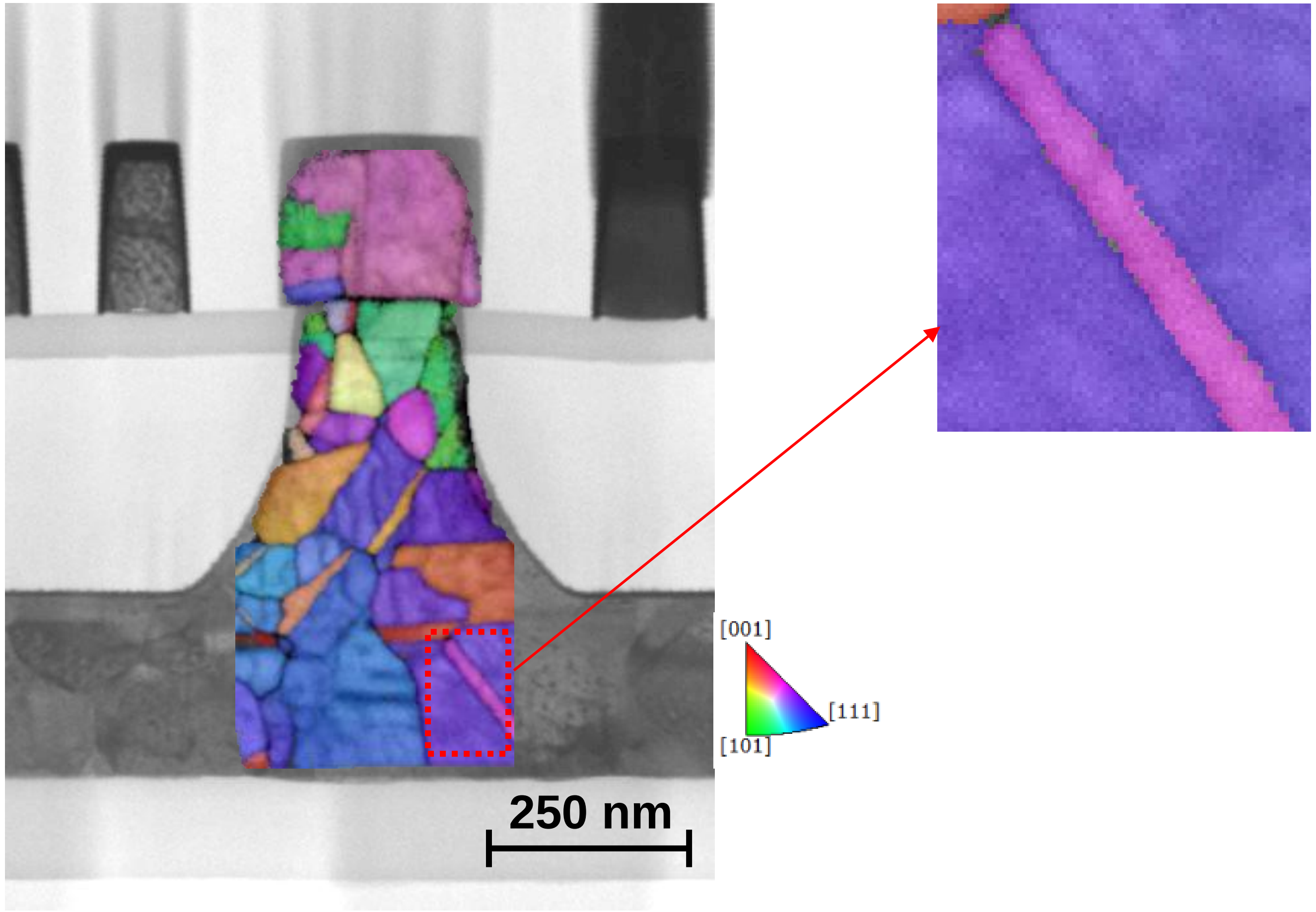


TKD mapping parameters:

- EHT: 30kV
- Probe current: 1.6 nA
- **Step size: 2 nm**
- Acq. speed: 185 fps
- **Zero sol.: 1.47%**

Microprocessor/FinFET: spatial resolution

STEM and IPFY map on Cu interconnect

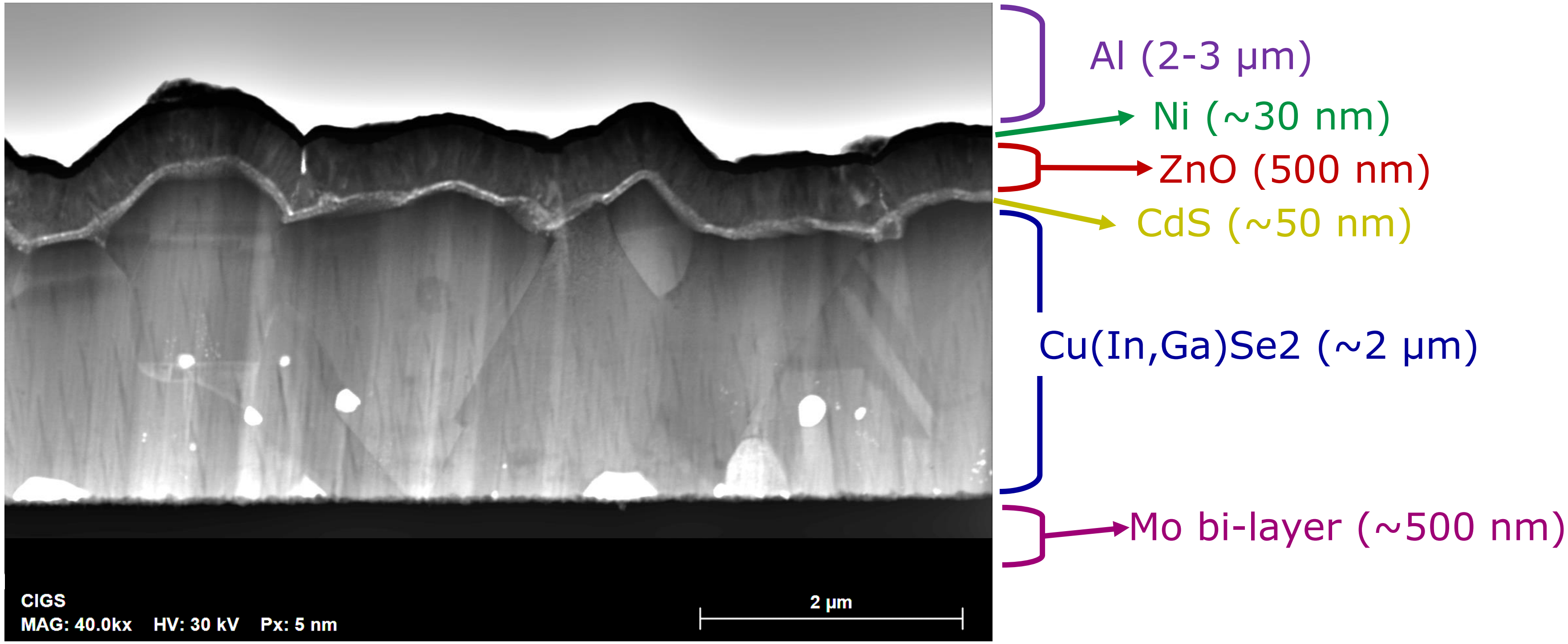


TKD mapping parameters:

- EHT: 30kV
- Probe current: 1.6 nA
- **Step size: 2 nm**
- Acq. speed: 185 fps
- **Zero sol.: 1.47%**

Application example: Cu(InGa)Se_2 solar cell

STEM BF

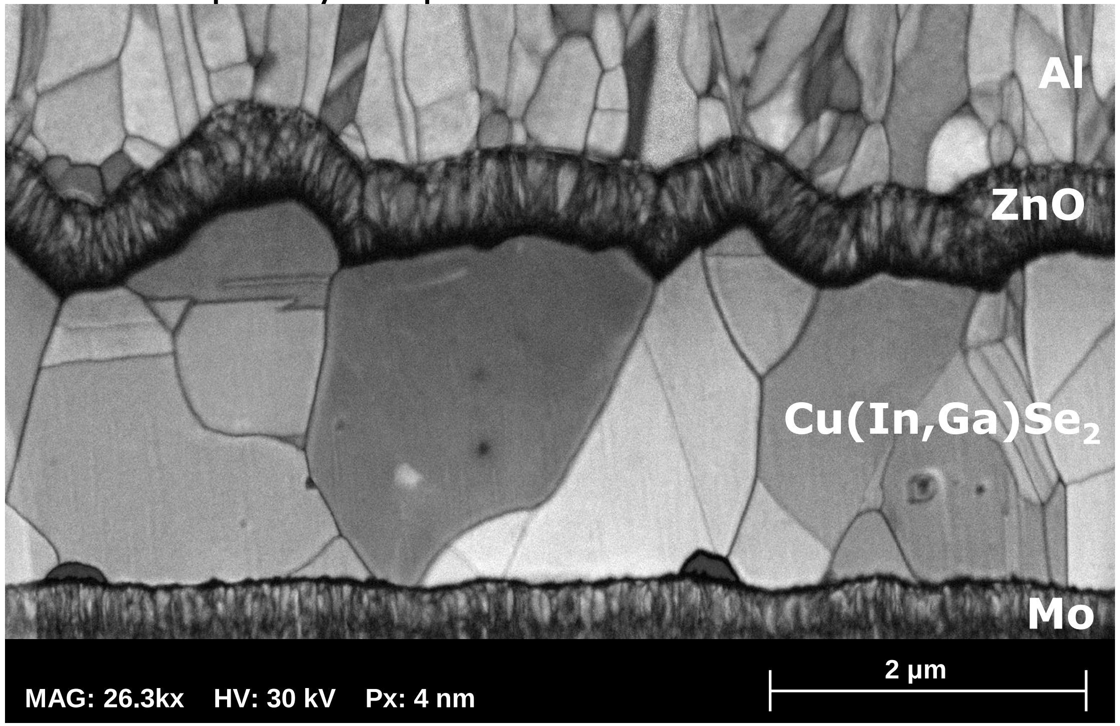


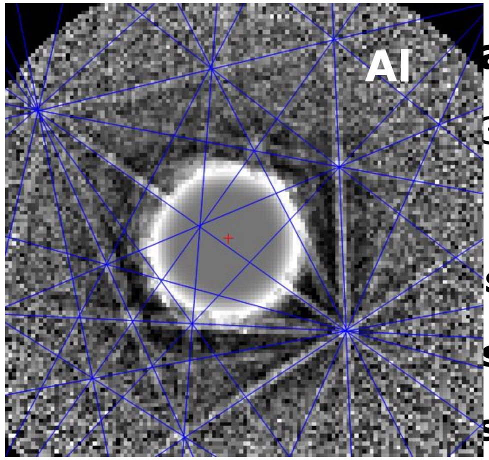
Sample courtesy: M. Raghuwanshi, RWTH Aachen University, Aachen, Germany

Application example: $Cu(InGa)Se_2$ solar cell

Large area mapping

Pattern quality map





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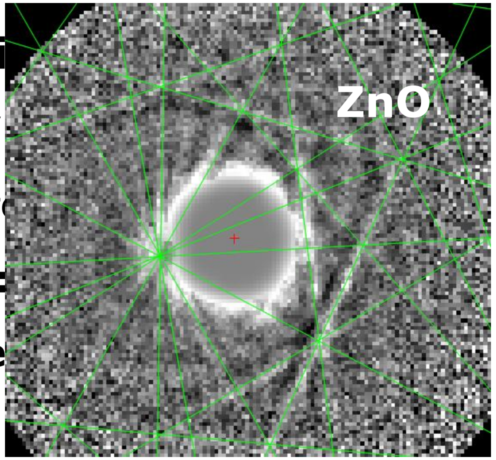
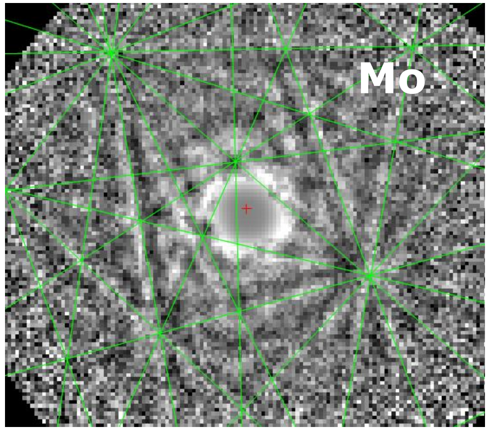
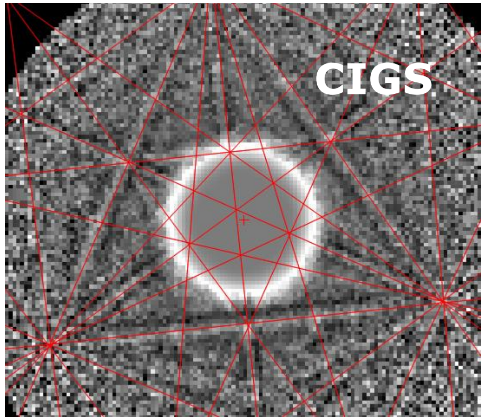
size:



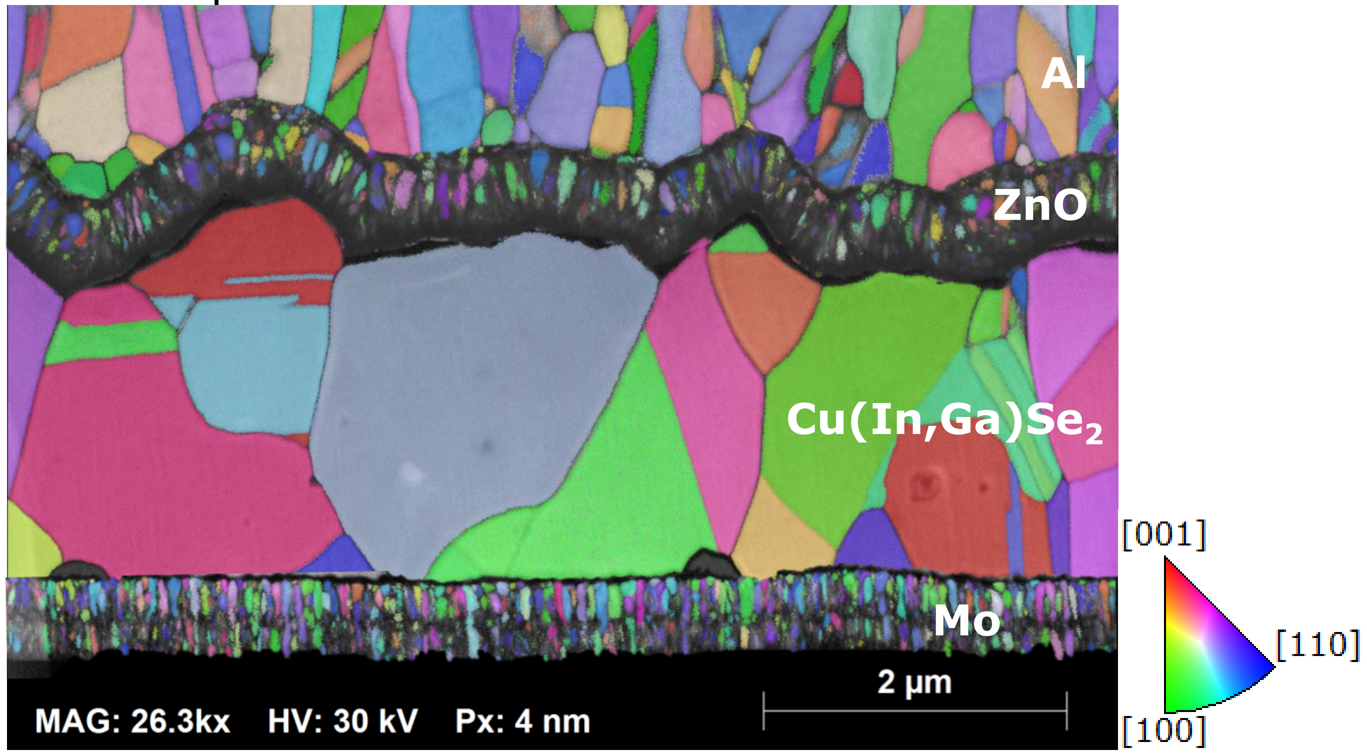




Application example 1: Cu(InGa)Se_2 solar cell

Large area mapping

IPF-Z map



Mapping parameters:

- EHT: 30kV
- Probe current: 1.6 nA
- Step size: 4nm
- Acq. speed: 330 fps
- **Map size: 1.93 million pixels**
- Zero sol.: 19.3% (whole map)
- **Zero sol.: 1.18% on CIGS**

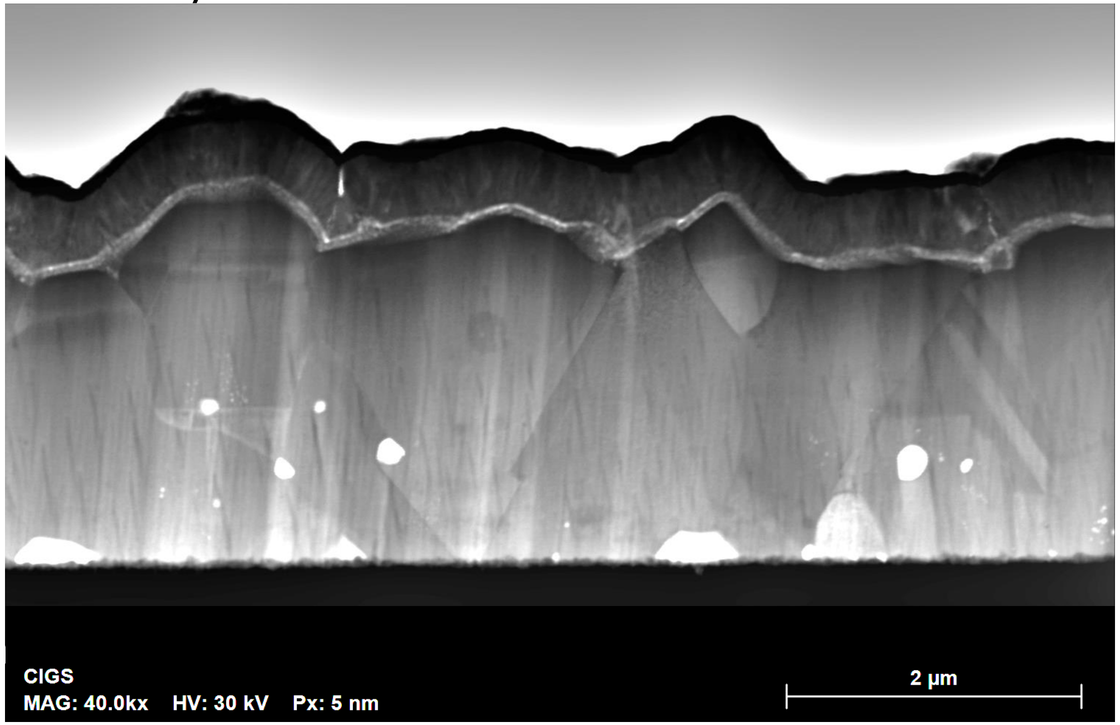
Indexing rate in CIGS: 98.82%

ARGUS^{TM} imaging system

ARGUS imaging

STEM like BF/DF imaging

CIGS layers

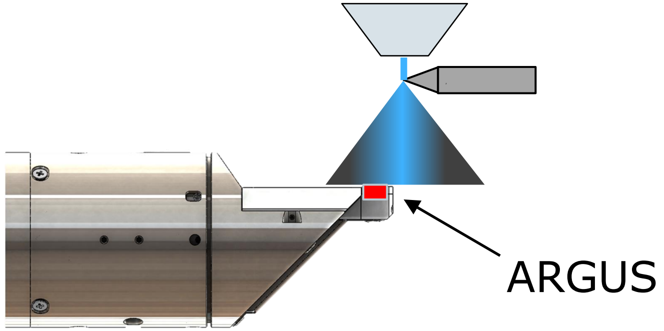


Imaging parameters:

- EHT: 30kV
- Probe current: 1.6 nA
- Step size: 5nm
- Image size: 7.5 × 5.6 µm

Mode 1:

- STEM-like bright field

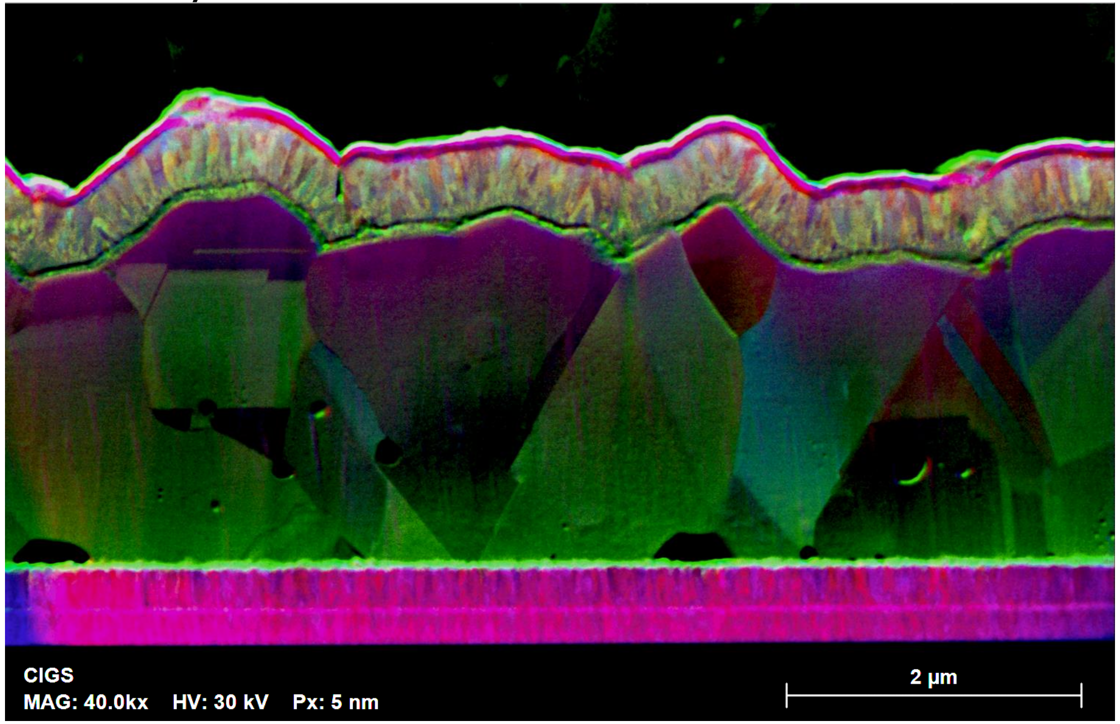


Sample courtesy: M. Raghuwanshi, RWTH Aachen University, Aachen, Germany

ARGUS imaging

Color coded orientation contrast imaging

CIGS layers

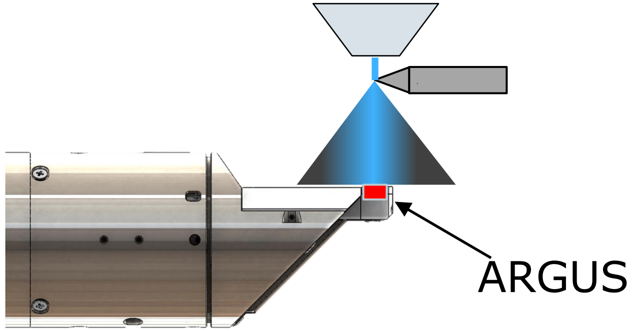


Imaging parameters:

- EHT: 30kV
- Probe current: 1.6 nA
- Step size: 5nm
- Image size: 7.5 × 5.6 µm

Mode 2:

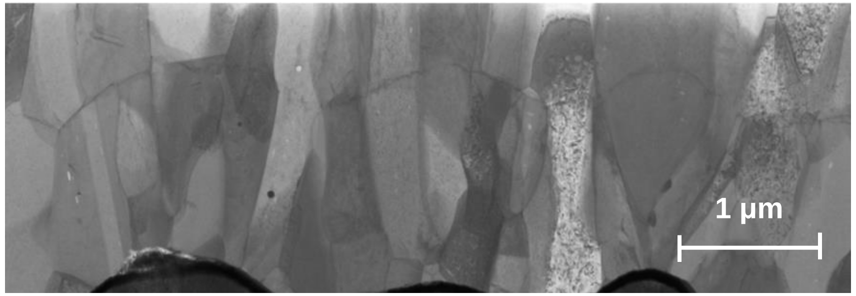
- STEM-like dark field

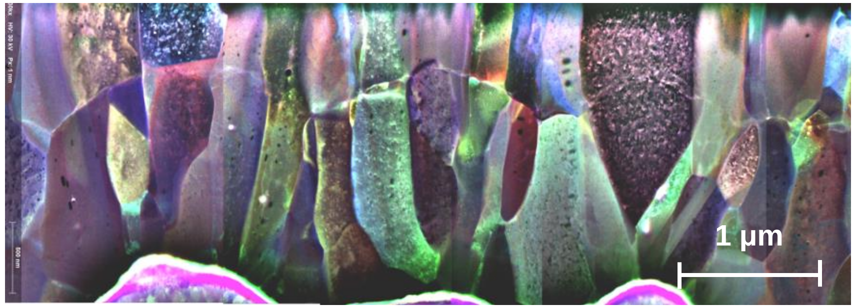


Sample courtesy: M. Raghuwanshi, RWTH Aachen University, Aachen, Germany

ARGUS imaging
Color coded orientation contrast imaging

Al layer





STEM Bright field

ARGUS STEM-DF like imaging:(color coded orientation contrast)

Argus Imaging parameters:

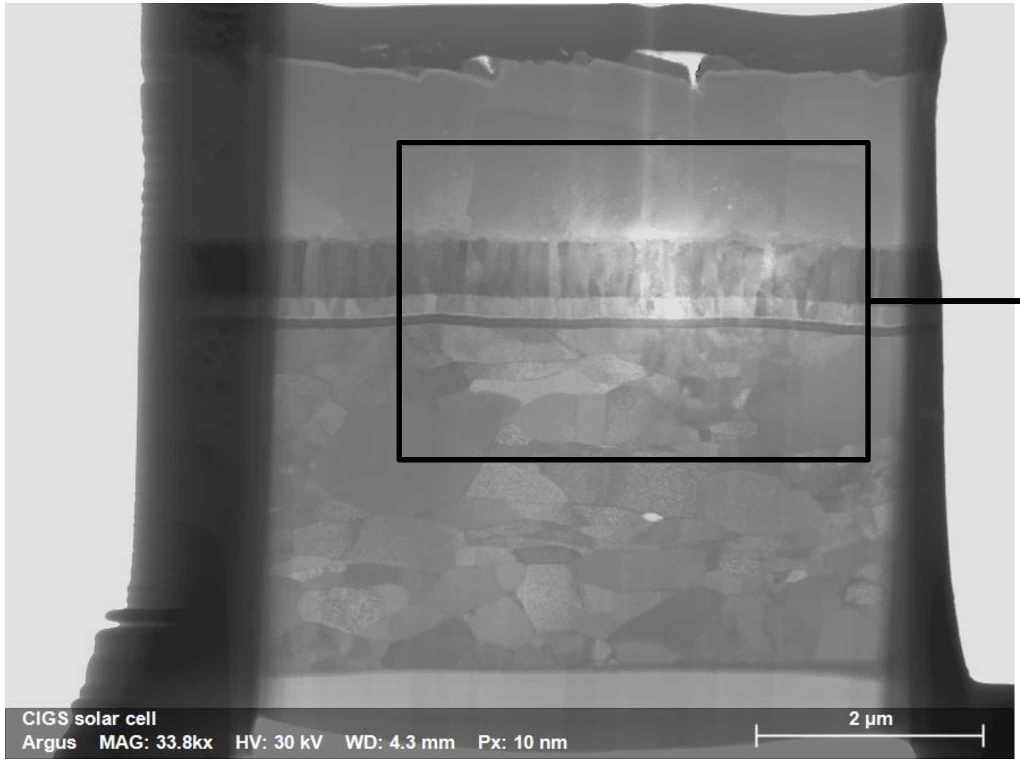
- EHT: 30kV
- Step size: 2 nm
- Mode: Argus DF
- **Acq. speed: 75 s (panorama)**

ARGUS imaging

Color coded orientation contrast imaging

CIGS sample #2

Bright field



ARGUS dark field

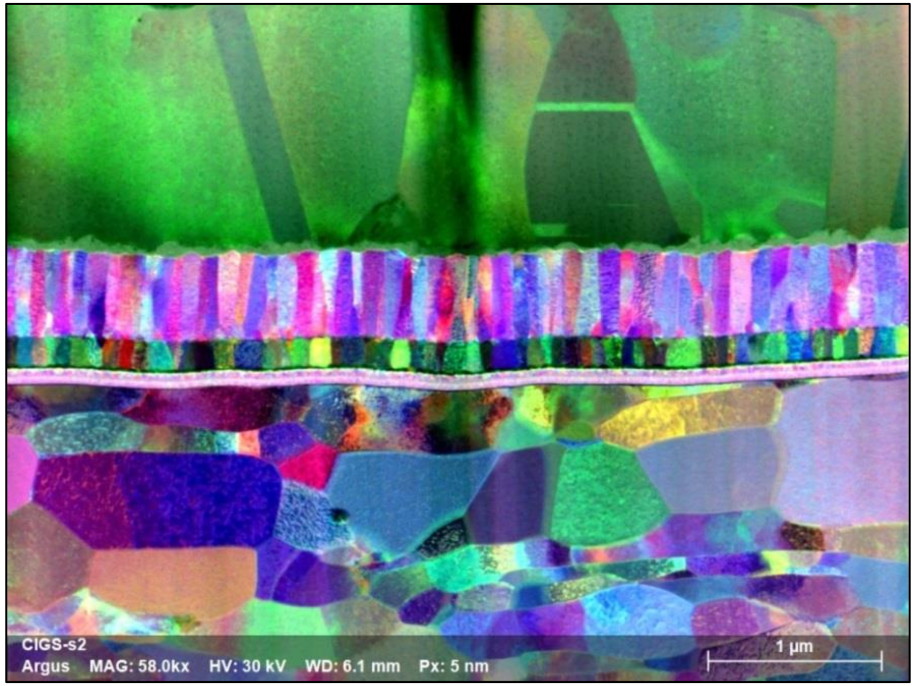


Image acquisition time: 16s

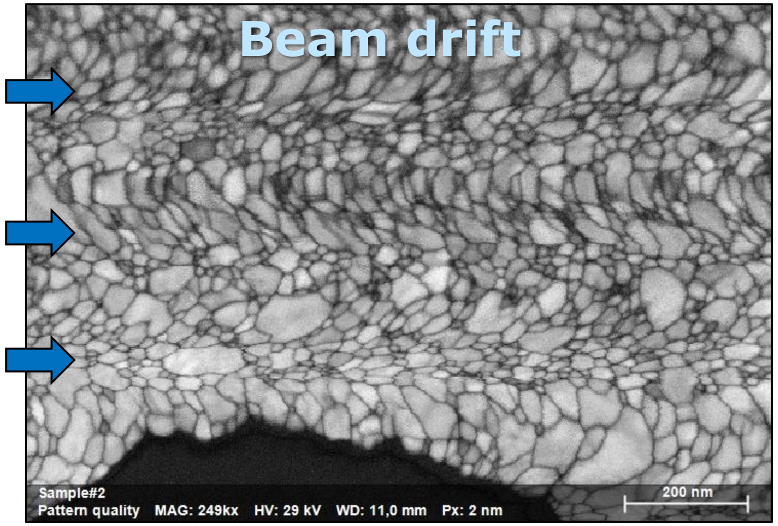
Practical recommendations

Practical recommendations

- FIB lamella: When to start low kV milling?
  - Low kV milling (5 kV, followed by lower kV's) step to be started by observing the SE signal of the lamella at 10 kV (SEM) or lower (e-transparency: Z dependent)

- For samples that may create beam instabilities (e.g. excessive charging/glass/insulating material in the lamella)
  - Use 90° scan rotation - let the electrons tunnel or sort of bleed out through these discharge paths
  - This also improves the pattern quality since, without charging, the landing energy is equal to the applied HV

on-axis TKD in SEM
Minimize drift induced artifacts



- 40nm Au film on Si (dimpled)
- 2.6nA and **10ms/point**
- 43:05 min

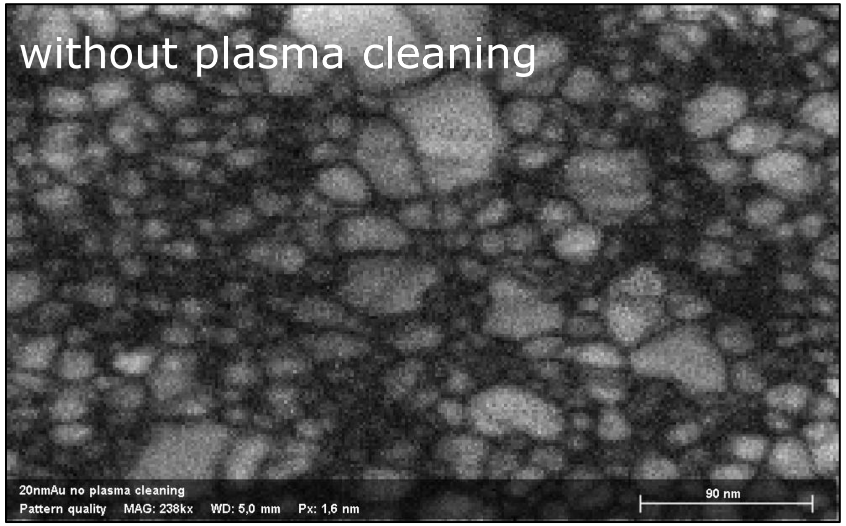
- 20nm Au film on 5nm Si_3N_4 membrane
- 1.75nA and **3ms/point**
- 14:04min

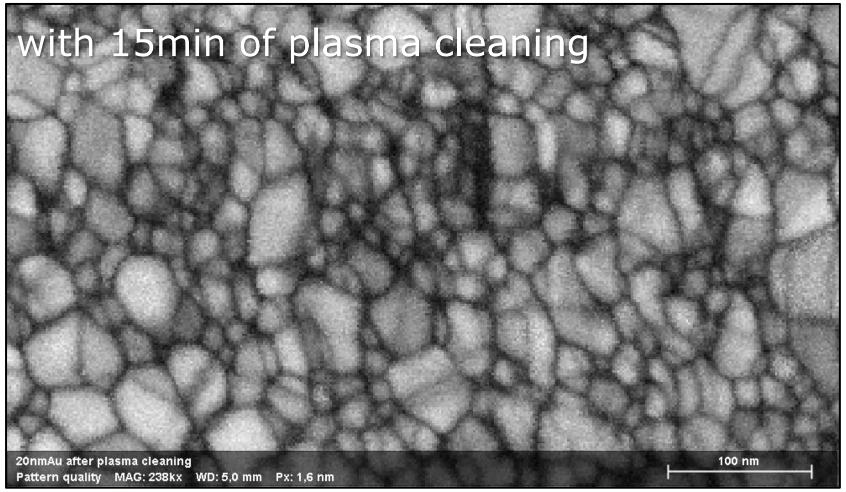
Lower probe currents and faster data acquisition:

less prone to beam instability !!

on-axis TKD in SEM

Effect of plasma cleaning on data quality





TKD results: Pattern Quality maps

- Sample:

20nm Au thin film on Si_3N_4 membrane

- Acquisition speed:

~250fps (4ms exposure time)

- Detector:

e$^-$Flash^{FS} with OPTIMUS TKD

- Beam parameters:

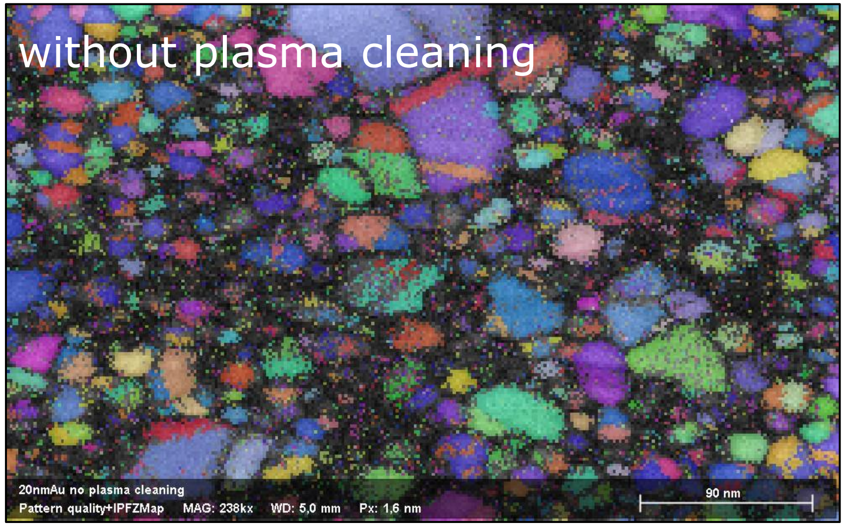
EHT: 30kV

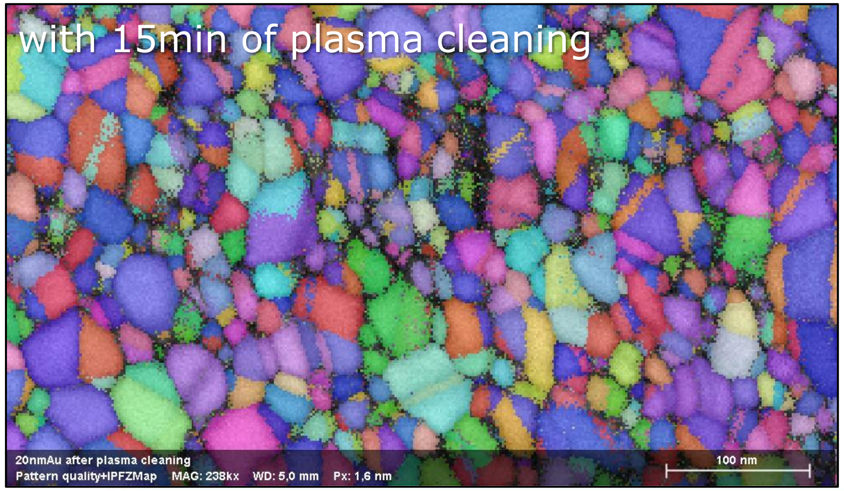
probe current: 1.75nA

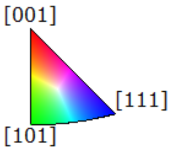
Sample is courtesy of Alice Da Silva Fanta from CEN, DTU, Denmark

on-axis TKD in SEM

Effect of plasma cleaning on data quality







TKD results: IPF maps

- Acquisition speed:

~250fps (4ms exposure time)

- Detector:

e⁻Flash^{FS} with OPTIMUS TKD

- Beam parameters:

EHT: 30kV

probe current: 1.75nA

- Hit rate: 46.5% vs. 87.6% with plasma cleaning

Nanostructural characterization of semiconductors with SEM
Summary

- Stronger signal
  - Improved spatial resolution > 1.5 nm
  - Faster measurements - up to 660fps
- Minimum gnomonic distortions
  - Improved band detection
  - Improved indexing quality
- Combined EDS/TKD measurement
- Direct electron detection imaging with ARGUS™ (Si diodes)
  - Dark & Bright field images – ~1nm resolution

>>Possible to map large area with nanometer resolution: better statistics!

Q&A

Are there any questions?

Please type in the questions you might have

in the Q&A box and press *Send*.

Innovation with Integrity