Optimal Sample Preparation for Large-Area Transmission Kikuchi Diffraction Analysis

Dr. Laurie Palasse
Bruker Nano Analytics, Berlin (DE)

Dr. Pawel NOWAKOWSKI
E. A. Fischione Instruments, Inc., Pittsburgh (US)

Presenters

- Dr. Laurie Palasse

Global Application Manager EMA

Bruker Nano Analytics, Berlin (DE)

- Dr. Pawel NOWAKOWSKI

Sr. Applications Scientist

E. A. Fischione Instruments, Inc., Pittsburgh (US)

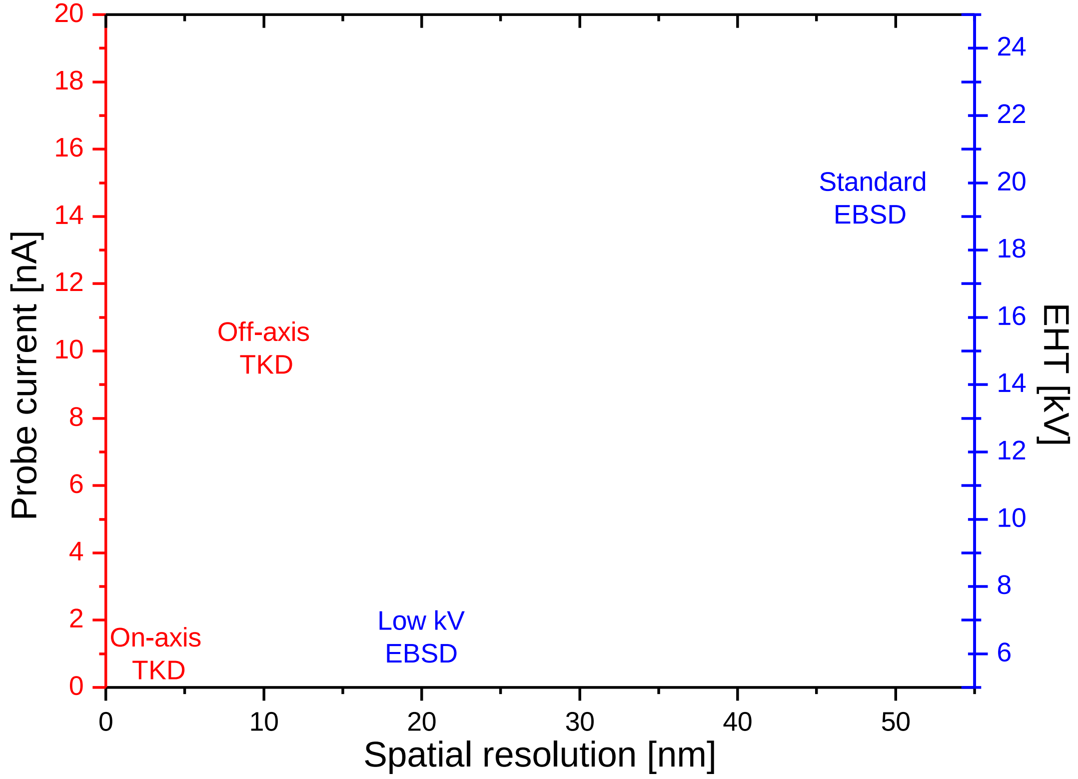
Outline

01

What is on-axis TKD?

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



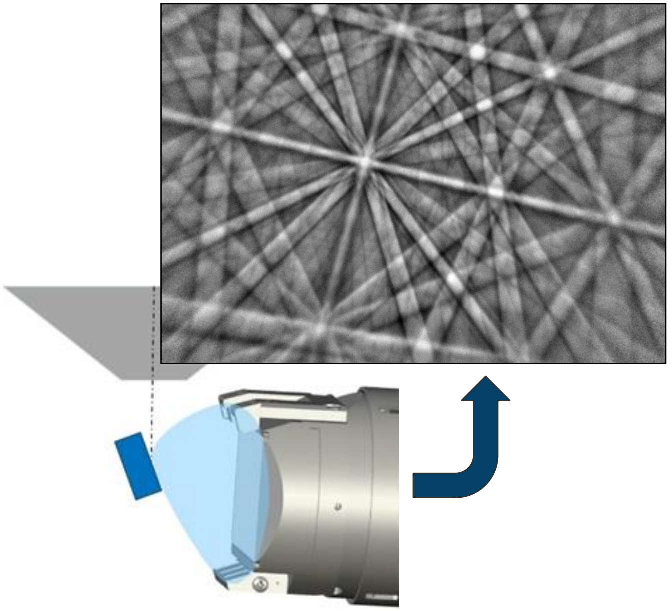
Effective spatial resolution values:

- EBSD at 20kV: down to ~50nm (low probe current)
- Low-kV EBSD: down to ~20nm
- Conventional TKD : down to ~8nm
- On-Axis TKD: down to ~2nm

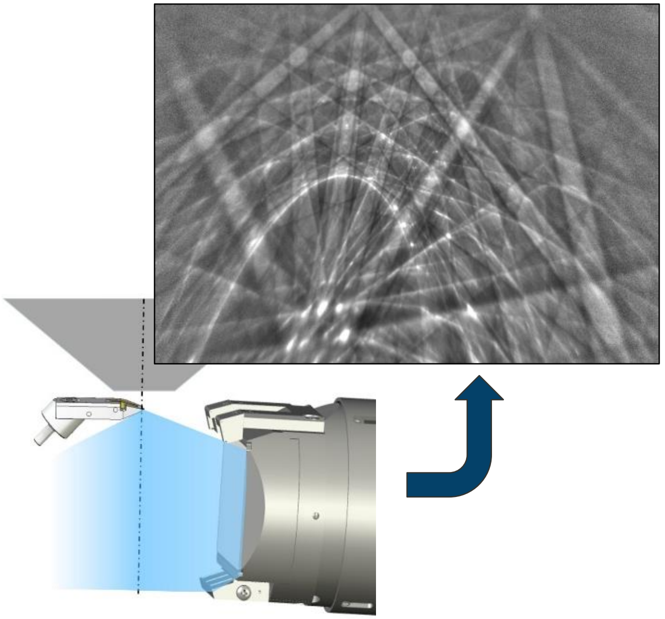
Experimental setup

EBSD vs. conventional TKD vs. On-Axis TKD

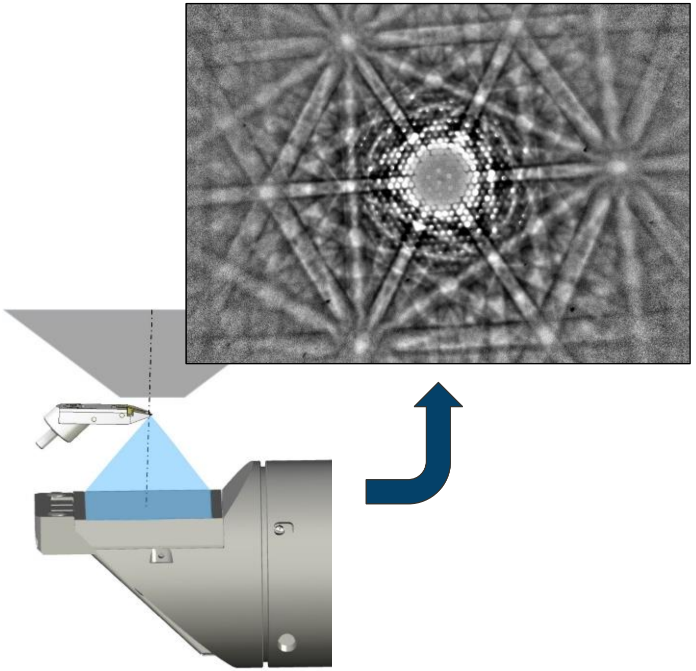
Standard EBSD



Conventional (Off-Axis) TKD



On-Axis TKD



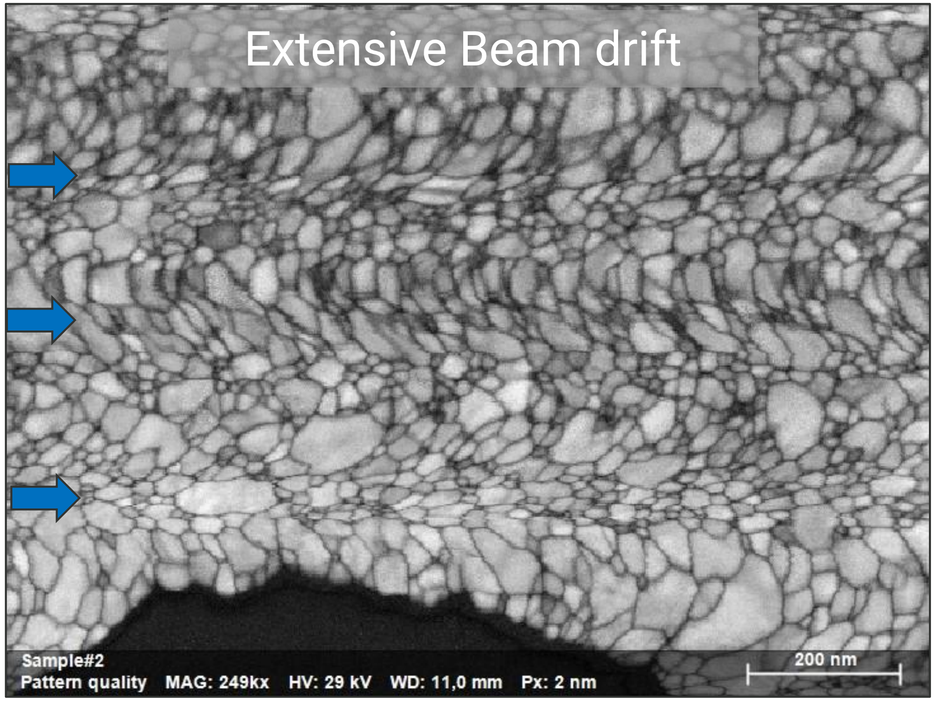
- EBSD and standard TKD use the same hardware & software
- Non-optimum sample-detector geometry for TKD ⇔ weak signal

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

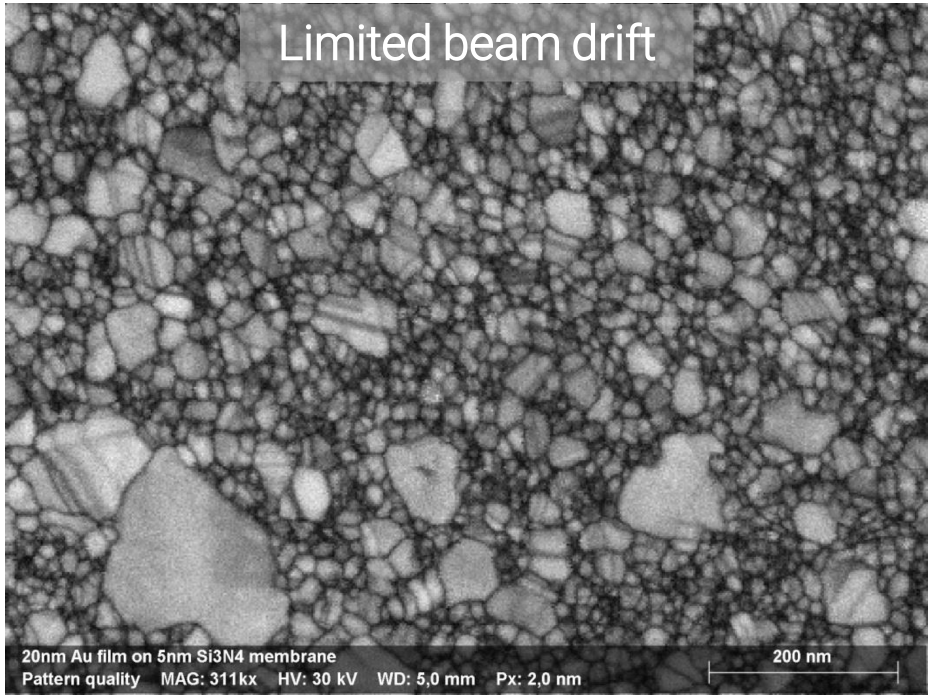
„*Orientation mapping by transmission-SEM with an on-axis detector*" J.J. Fundenberger et all, Ultramicroscopy, 161, 17–22, 2016.
"*A systematic comparison of on-axis and off-axis transmission Kikuchi diffraction*" F. Niessen et all, Ultramicroscopy, 186, 158-170, 2018.

On-Axis TKD in SEM
DF imaging with OPTIMUS

Off-axis TKD



On-axis TKD



- 2.6nA and 10ms/point vs. 1.75nA and 3ms/point
- 43:05 min vs. 14:04min

Lower probe currents and faster data acquisition – less prone to beam instability

On-Axis TKD
unique advantages

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:

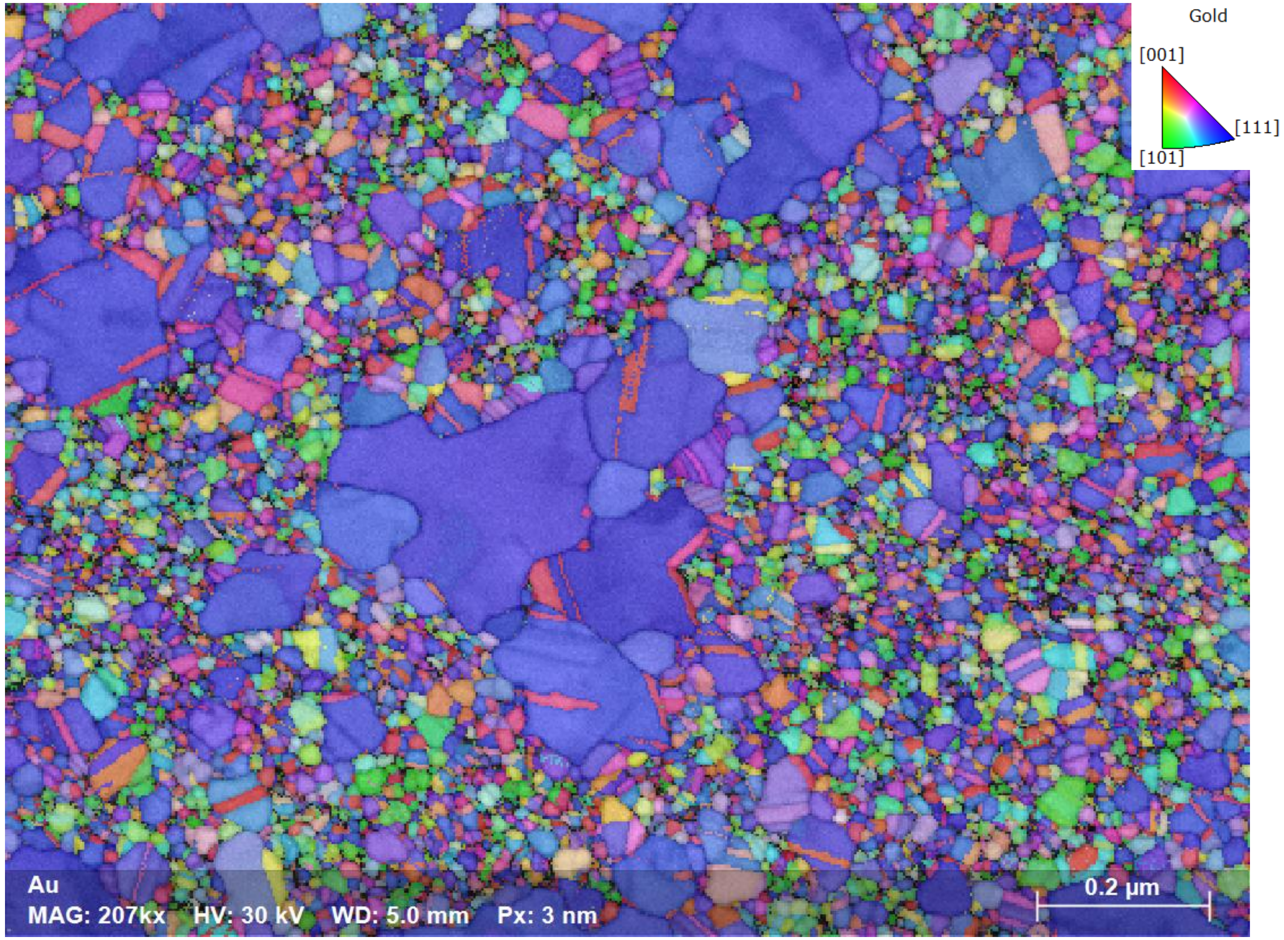
1. High spatial resolution requires **low probe diameter** (low current) => to keep the pattern quality constant we need signal
2. High speed ⇔ **short exposure times** => to keep the pattern quality constant we need signal
3. High indexing rates require high quality patterns which can only be obtained by having a **strong signal** (SNR)

On-Axis TKD

Best spatial resolution, speed and data quality

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

20nm Au film on 5nm Si_3N_4 membrane

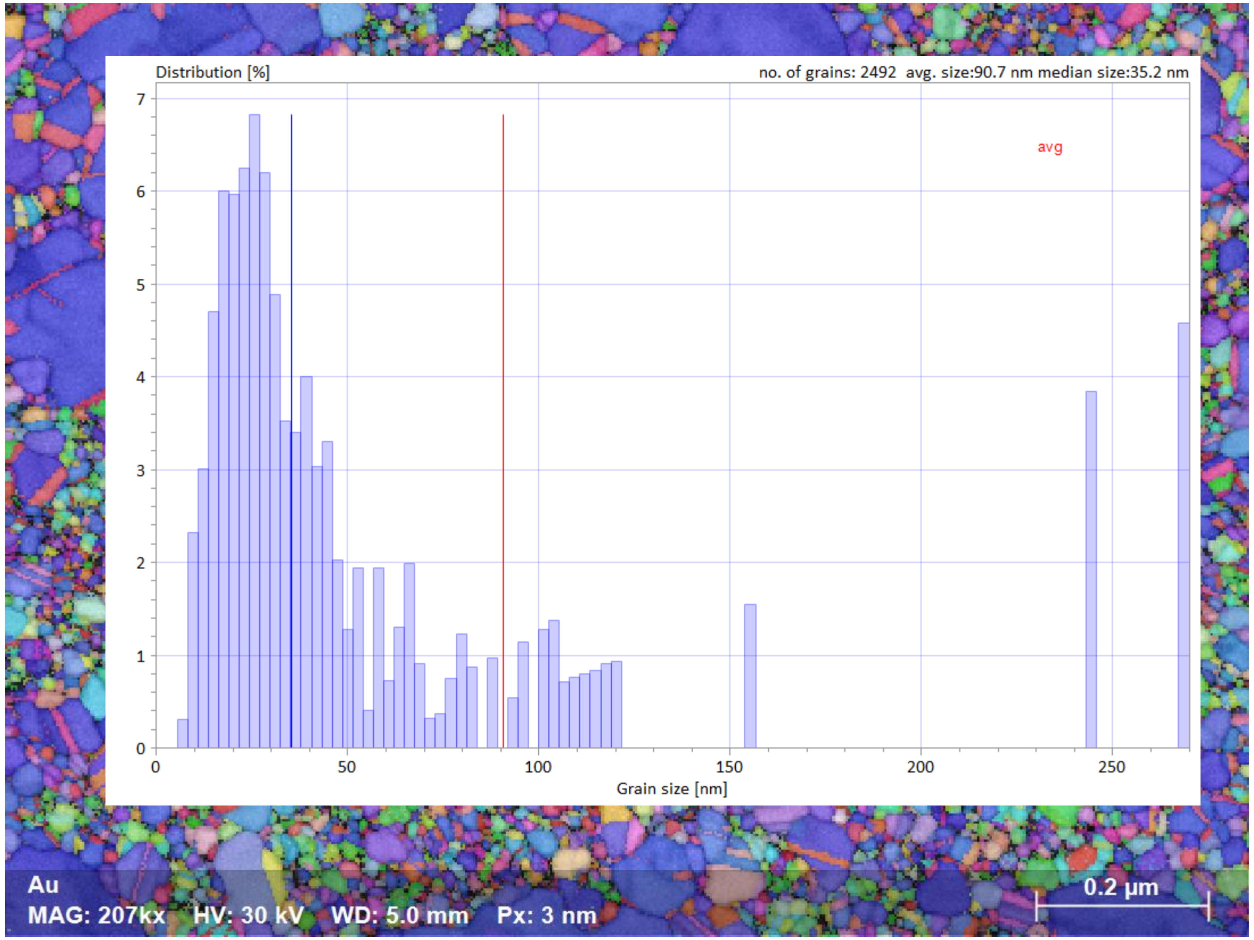


On-Axis TKD

Best spatial resolution, speed and data quality

- Important parameters:
- EHT: 30kV
- Probe current: 2nA
- Step size: 3nm
- Mapping speed: 320fps (3ms/point)
- Zero solutions: 11.5%
- Annealing twin: <6 nm wide
- No data cleaning!

20nm Au film on 5nm Si_3N_4 membrane

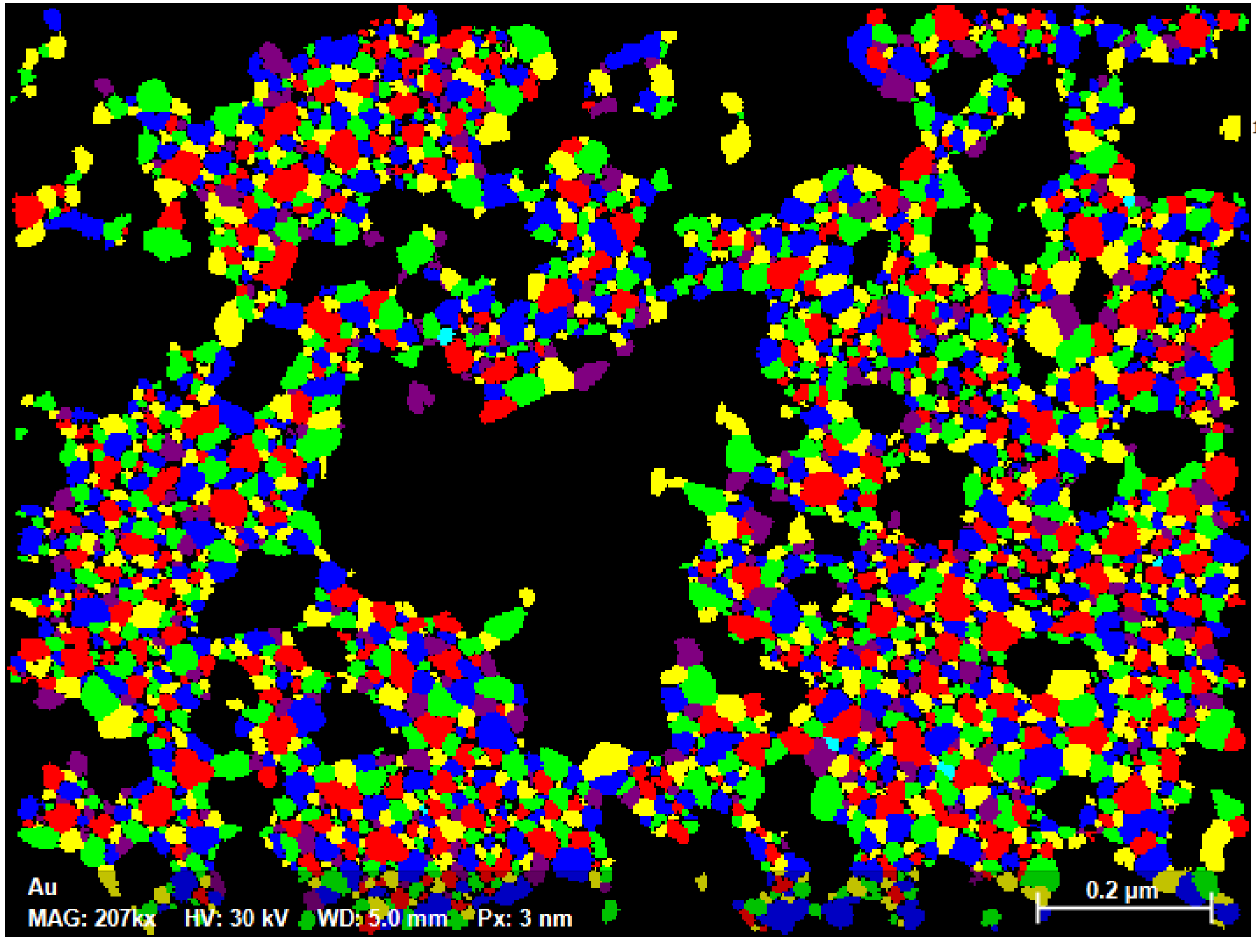


On-Axis TKD

Best spatial resolution, speed and data quality

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

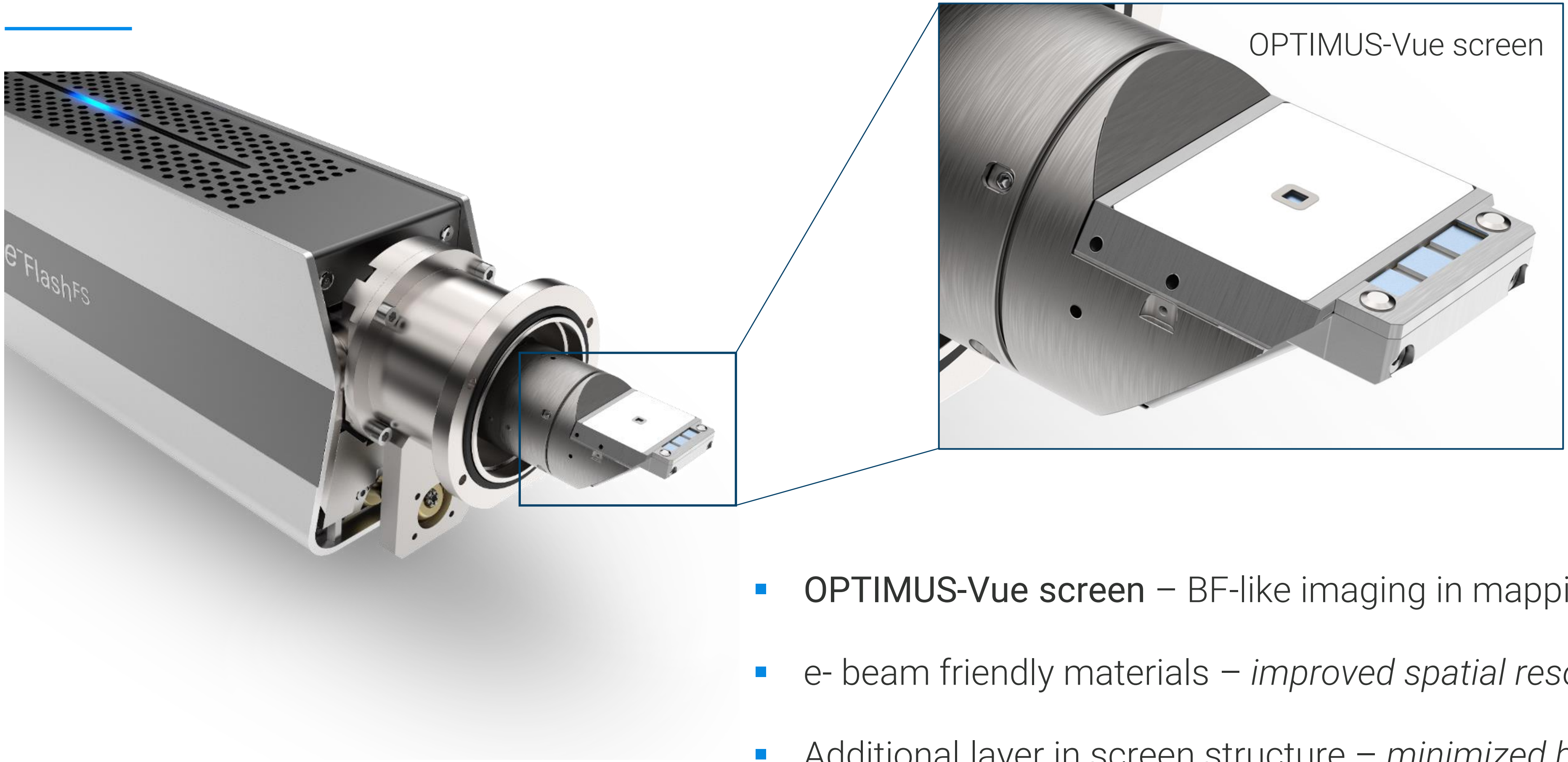
20nm Au film on 5nm Si_3N_4 membrane



02

on-axis TKD – capabilities and benefits

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

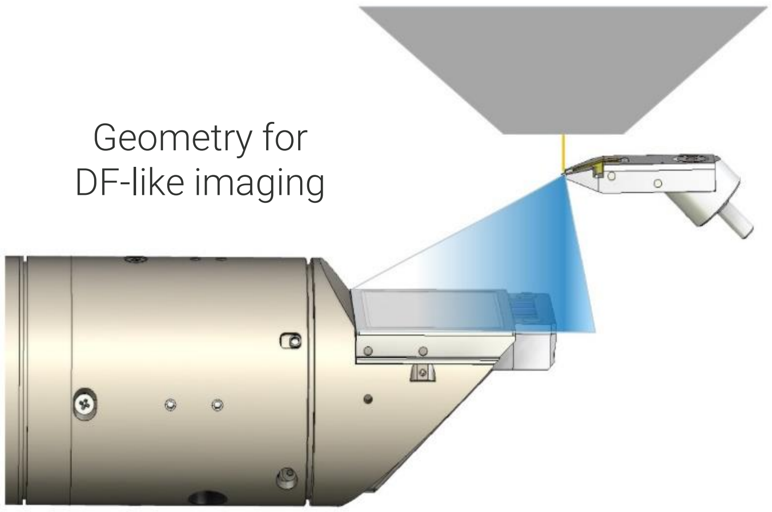


- **OPTIMUS-Vue screen** – BF-like imaging in mapping position (more details later)
- e- beam friendly materials – *improved spatial resolution and minimized drift*
- Additional layer in screen structure – *minimized beam interference*
- Redesigned screen frame – *easier/safer to use*

High resolution DF imaging with OPTIMUS 2

15 nm Au film deposited on 10 nm Si_3N_4 membrane

Geometry for DF-like imaging



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

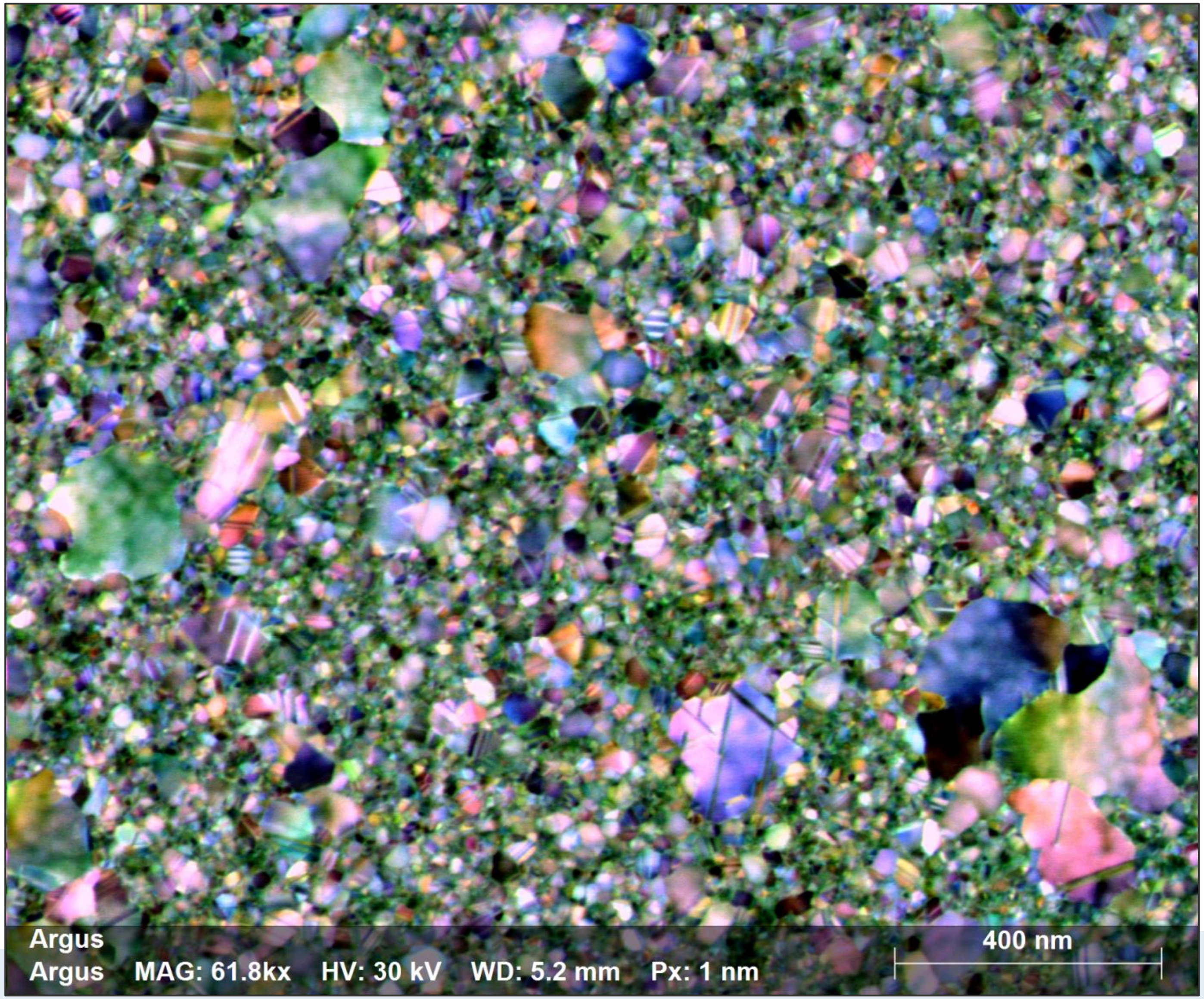
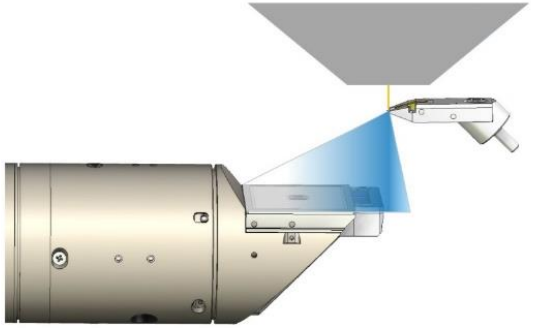


Image is courtesy of Hong Zhang from Eurofins in Santa Clara CA, USA

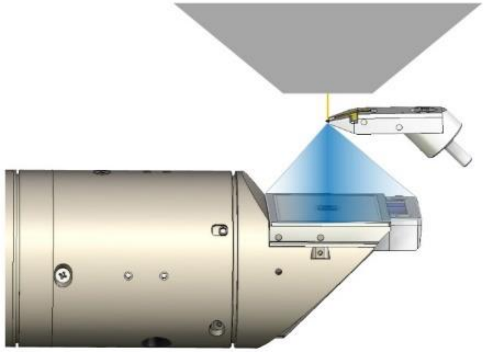
DF & BF imaging with OPTIMUS 2

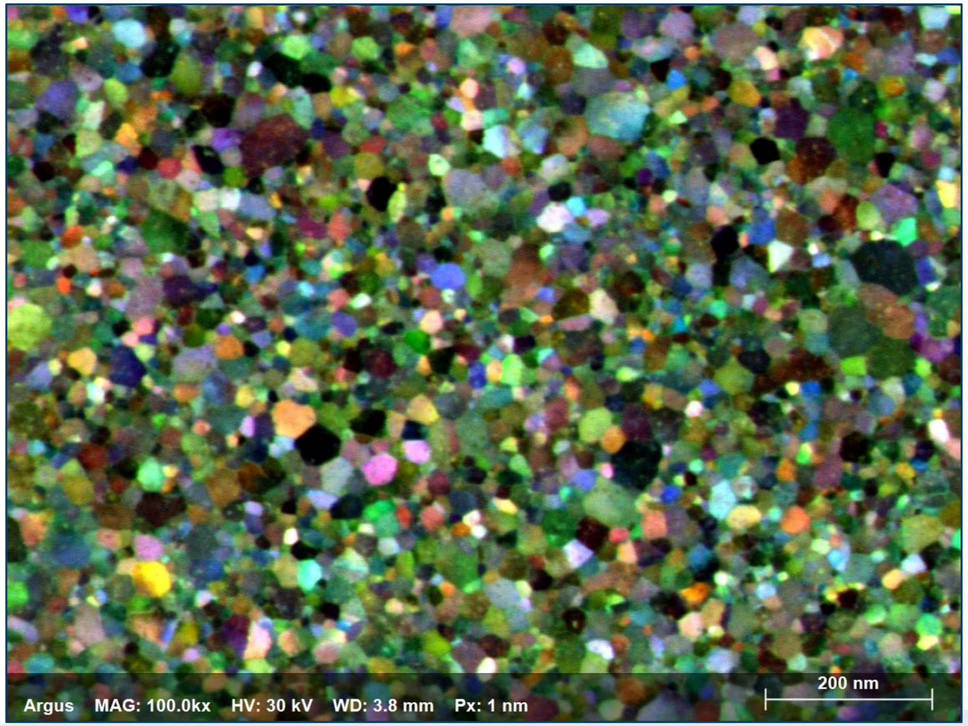
Ruthenium film

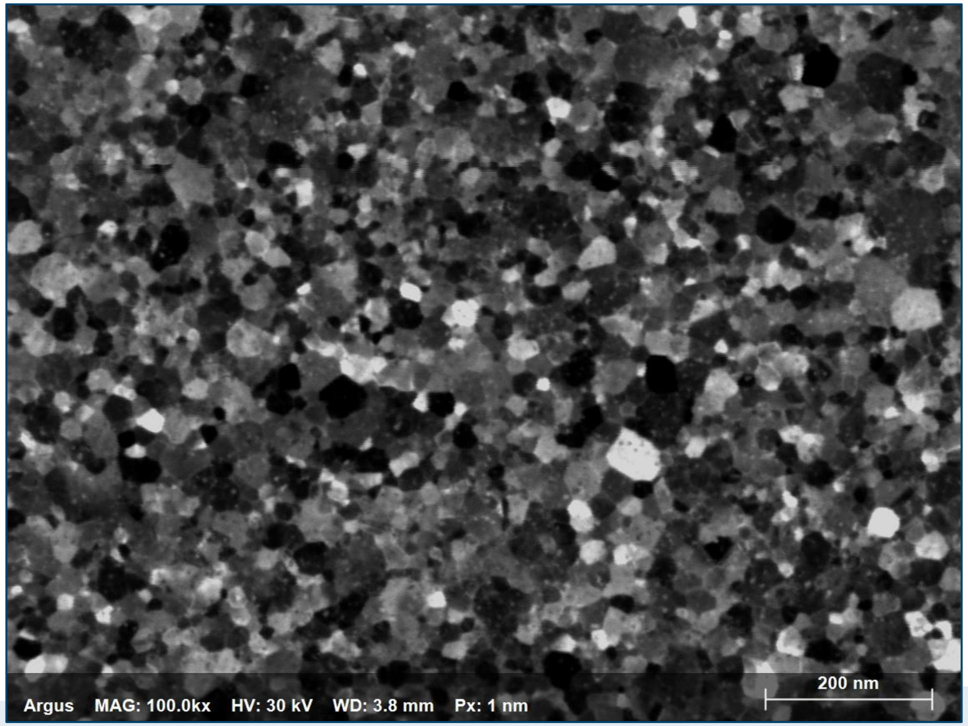


Applications & benefits:

- 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

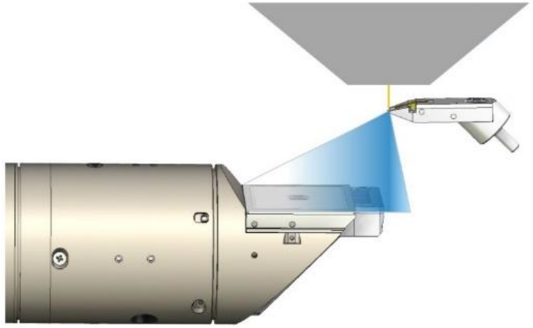


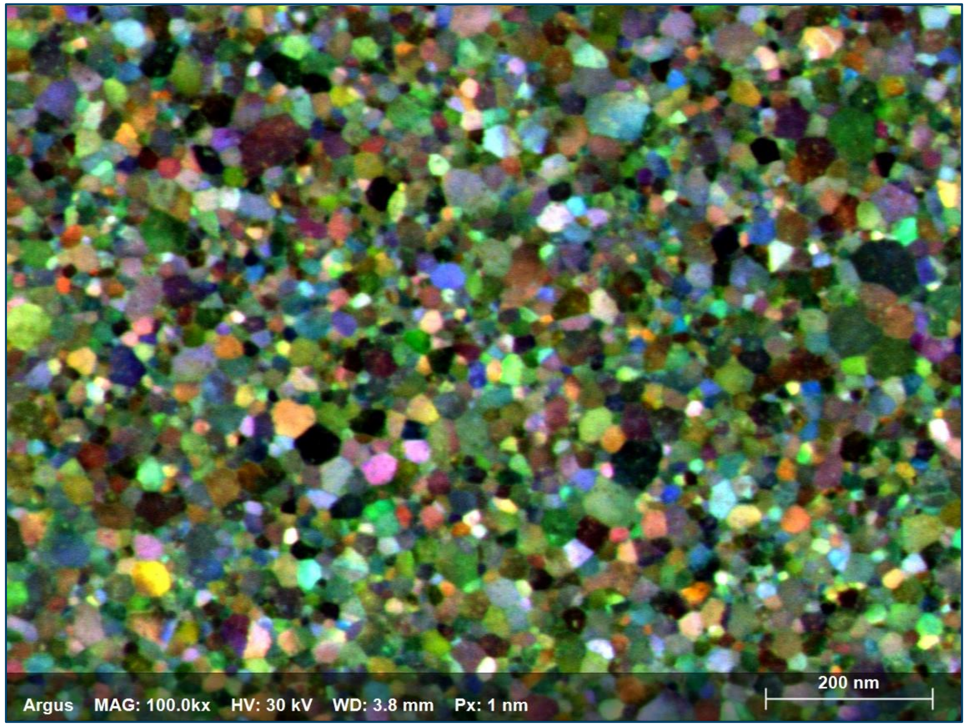


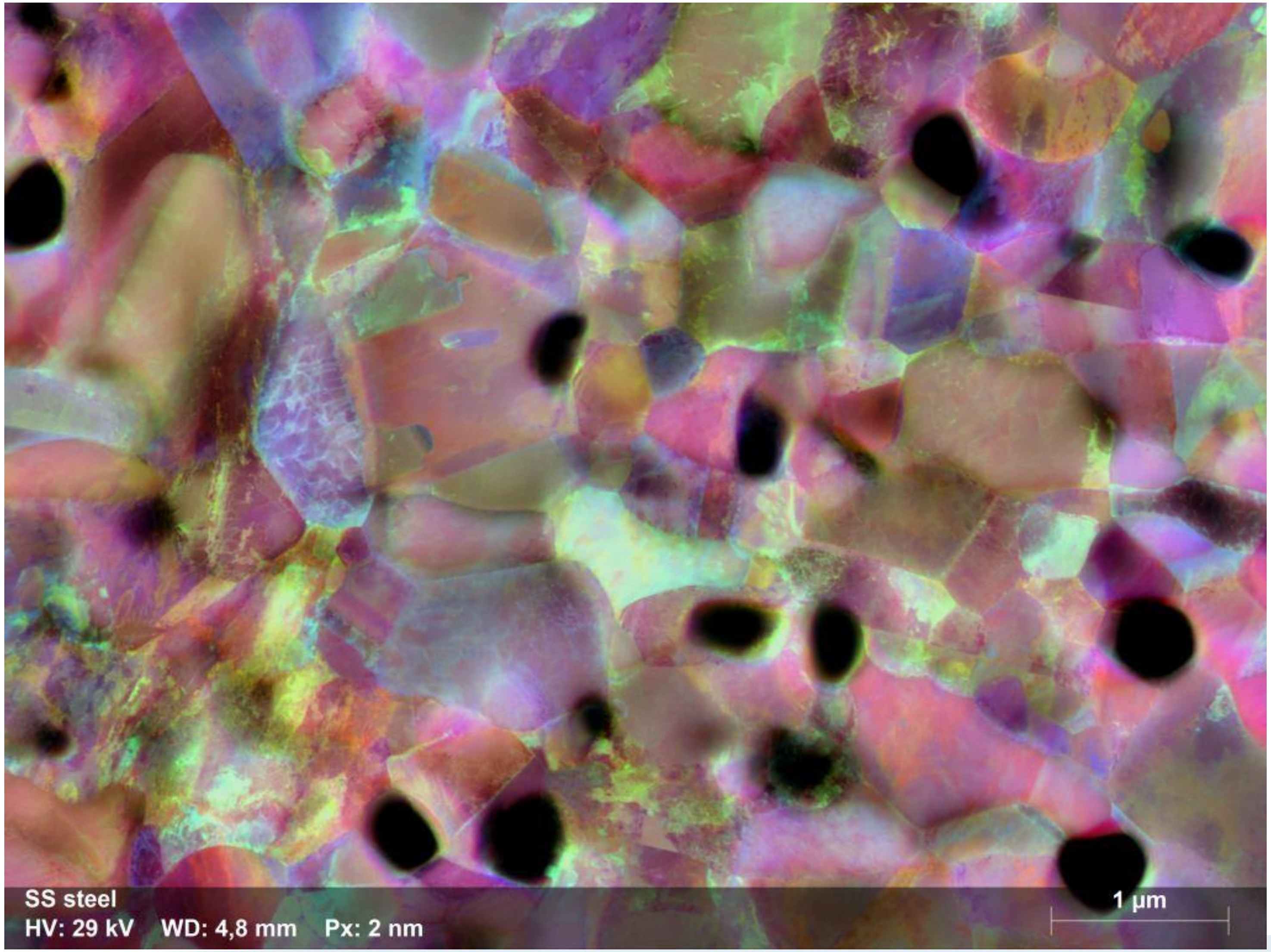


DF & BF imaging with OPTIMUS 2

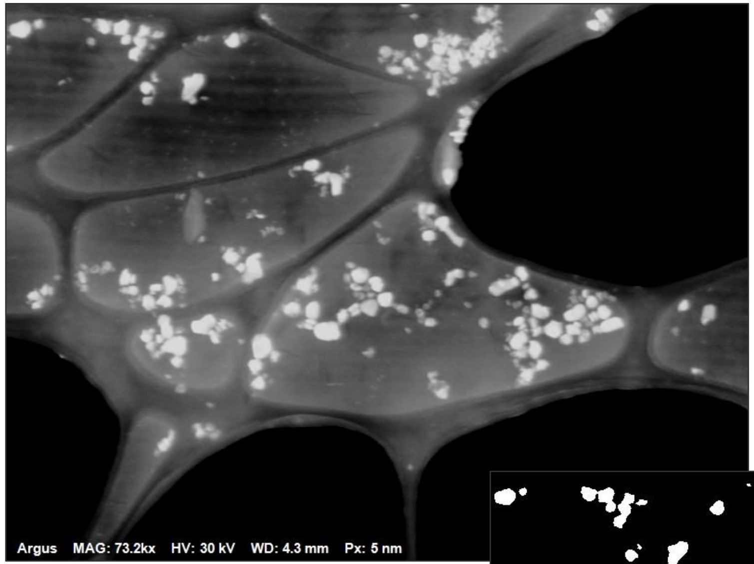
color-coded dark field ARGUS™ imaging





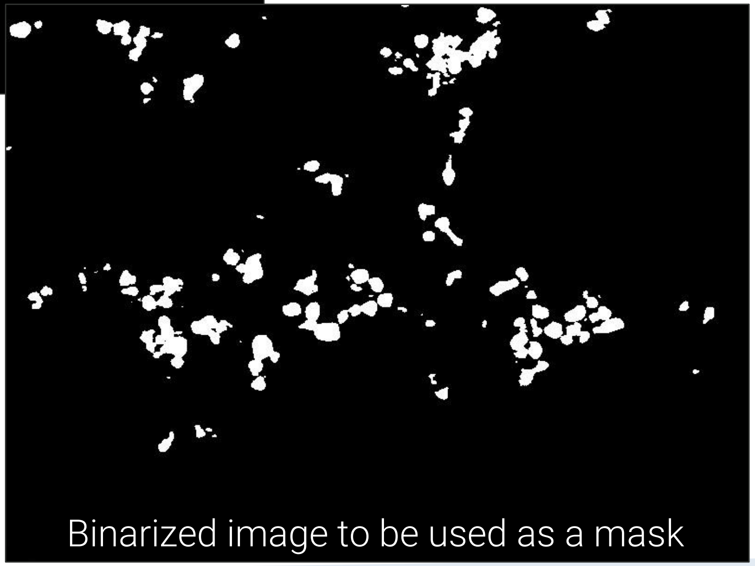


ESPRIT MaxYield
Productivity boost



ARGUS image of nanoparticles on C-lacey support film

MaxYield

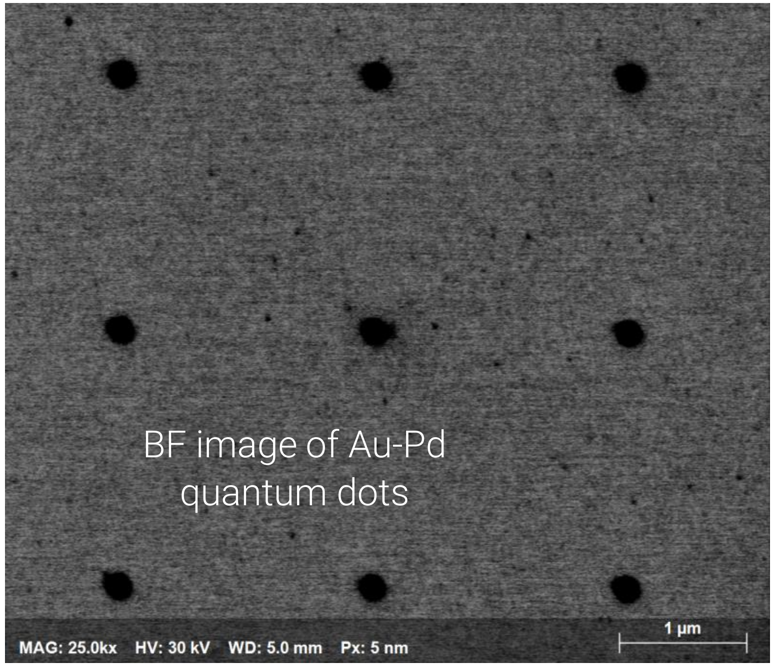


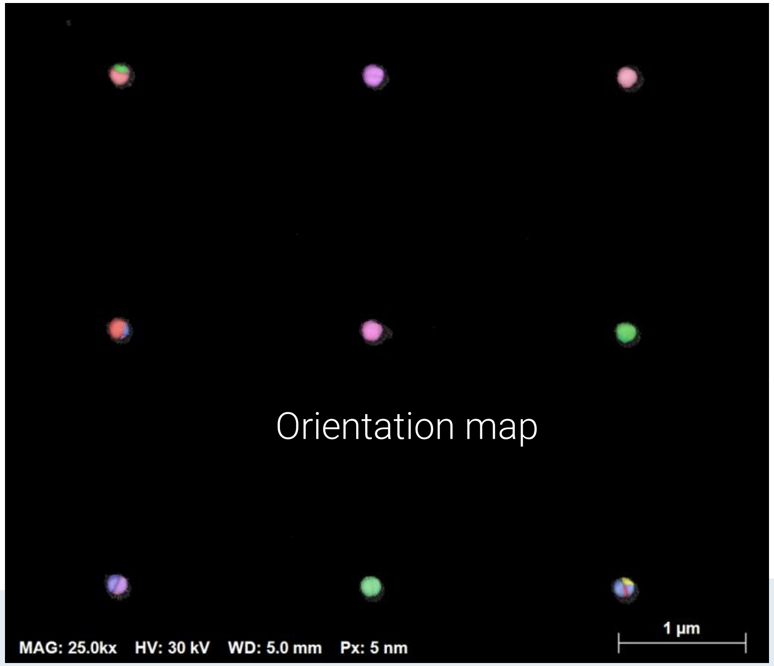
ESPRIT MaxYield => key details / *benefits*:

- 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



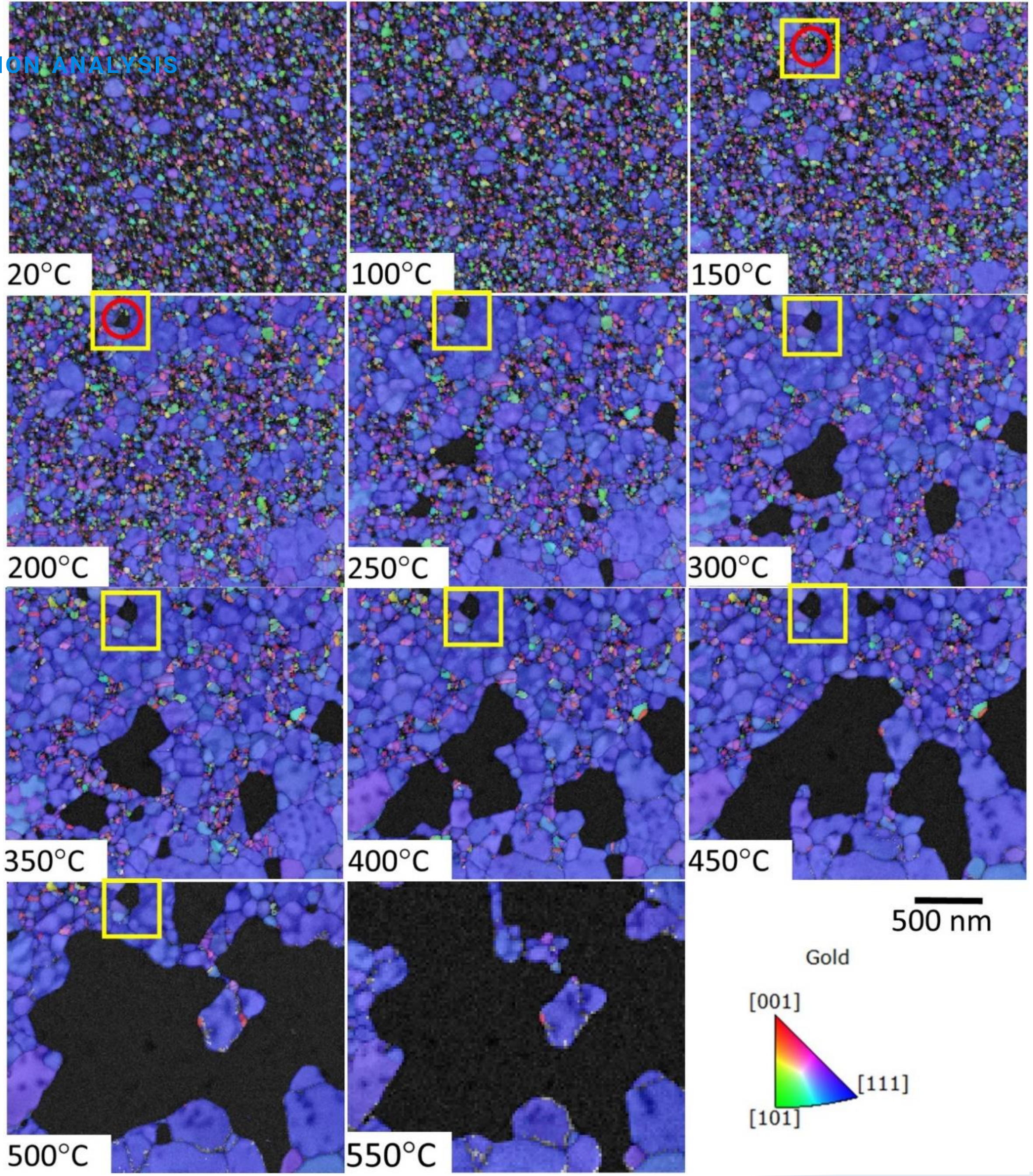


ESPRIT TRM

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

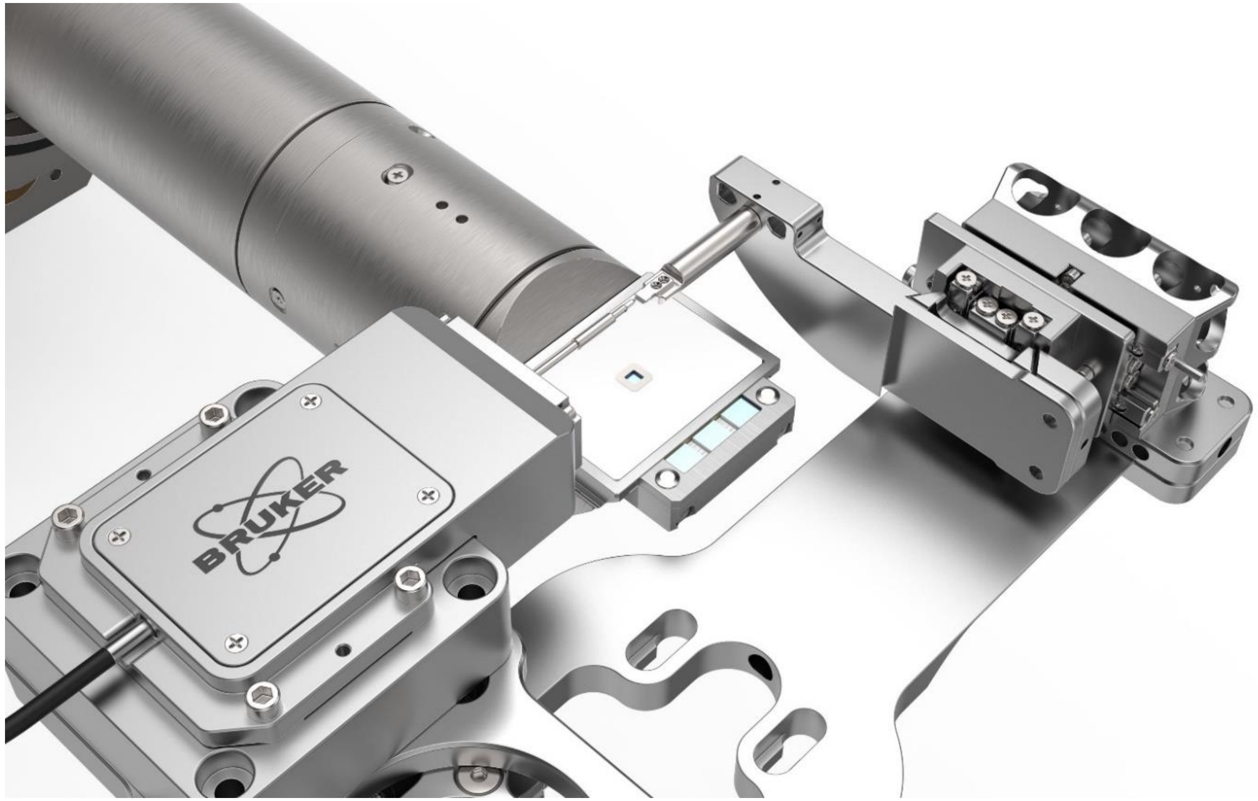


See: "Elevated temperature transmission Kikuchi diffraction in the SEM" Fanta et all, Materials Characterization, Vol ´.139, May 2018, Pages 452-462

Integration of multiple instruments

OPTIMUS 2 and PicoIndenter PI 89 with Push-to-Pull device

Sample and detectors setup for Time Resolved Measurements during in-situ tensile testing of nanomaterials

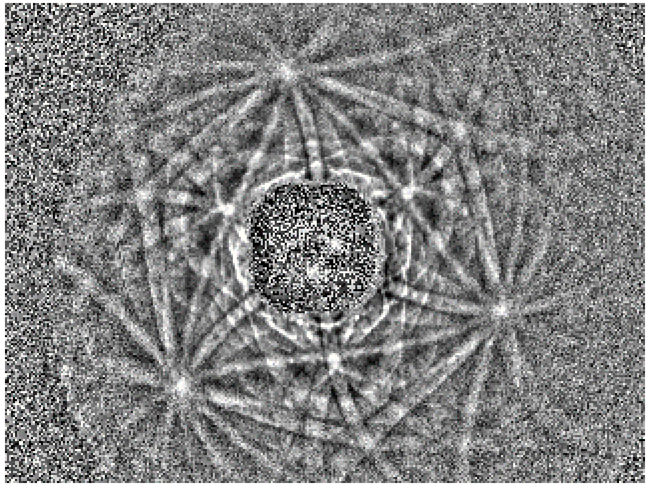


Time Resolved Measurements during in-situ experiments:

- Imaging at **125,000 pixels/second** during in-situ tensile testing with Push-to-Pull device on PI 89
- Time resolved mapping at up to **600 points/second** – quantify every microstructural change in the sample
- Applicable for in-situ heating and electrical biasing experiments

ESPRIT FIL-TKD
Enabling nano-scale TKD mapping

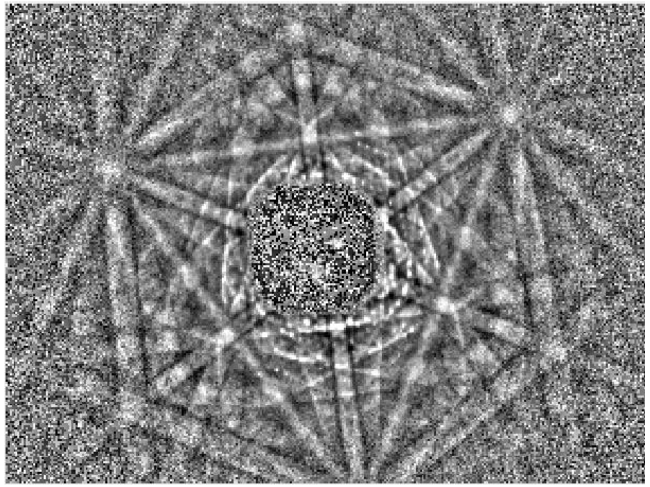
With field

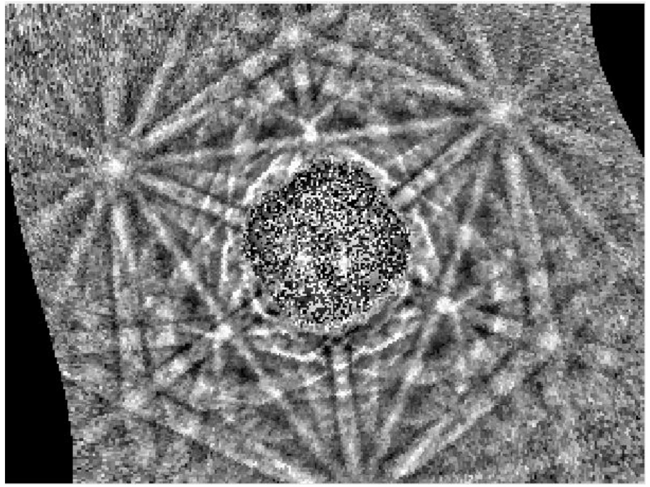


- Immersion mode ⇔ Strong Magnetic Field inside SEM chamber
  - Electrons are constrained within a narrow space around the optical axis of SEM – *limits their spread laterally*
  - Electron trajectories are affected – *distorted patterns*
- OPTIMUS 2 and ESPRIT FIL-TKD (EP000003835768B1)

FIL-TKD

No field





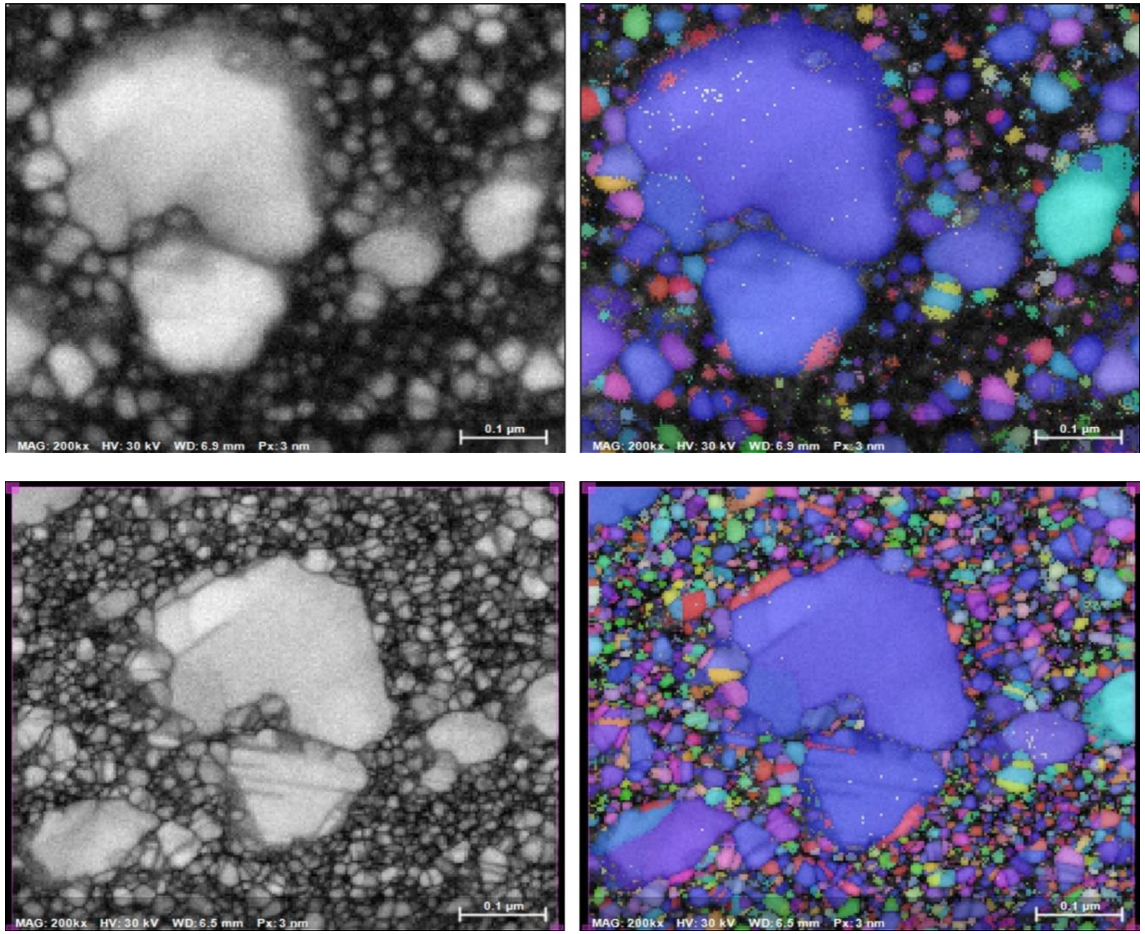
Goal: Achieve the best spatial resolution possible!

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

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

No magnetic field ⇔ analytical mode



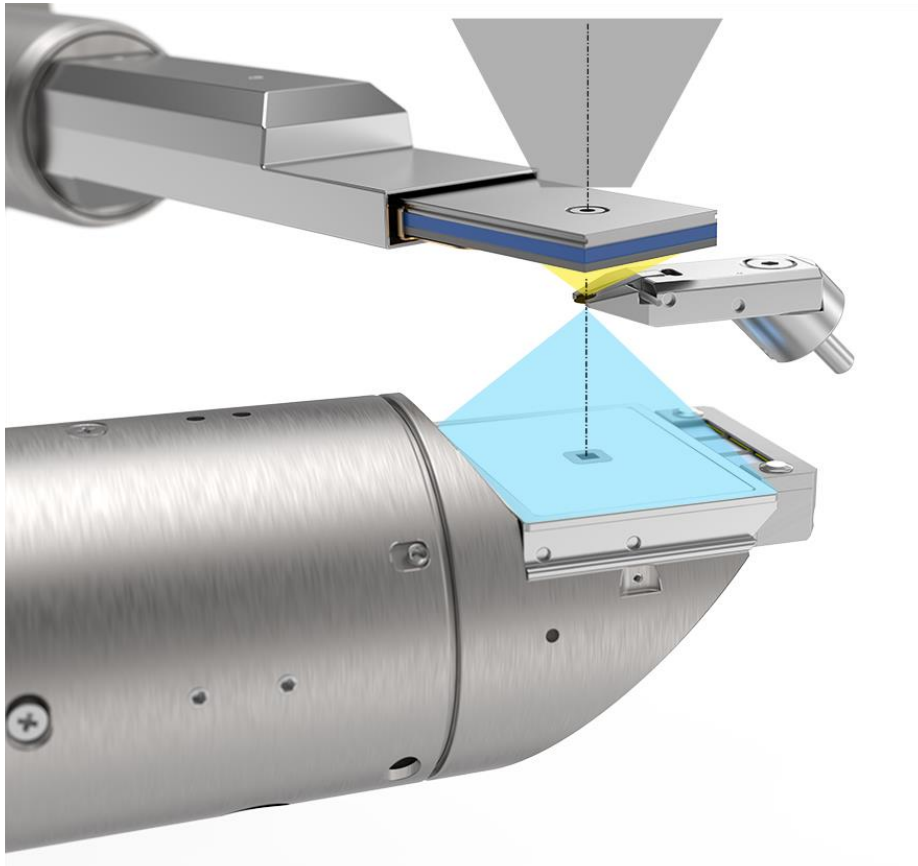
With magnetic field ⇔ Ultra-High Resolution (UHR) mode

Application examples

Integration of multiple instruments

OPTIMUS 2 and FlatQUAD EDS

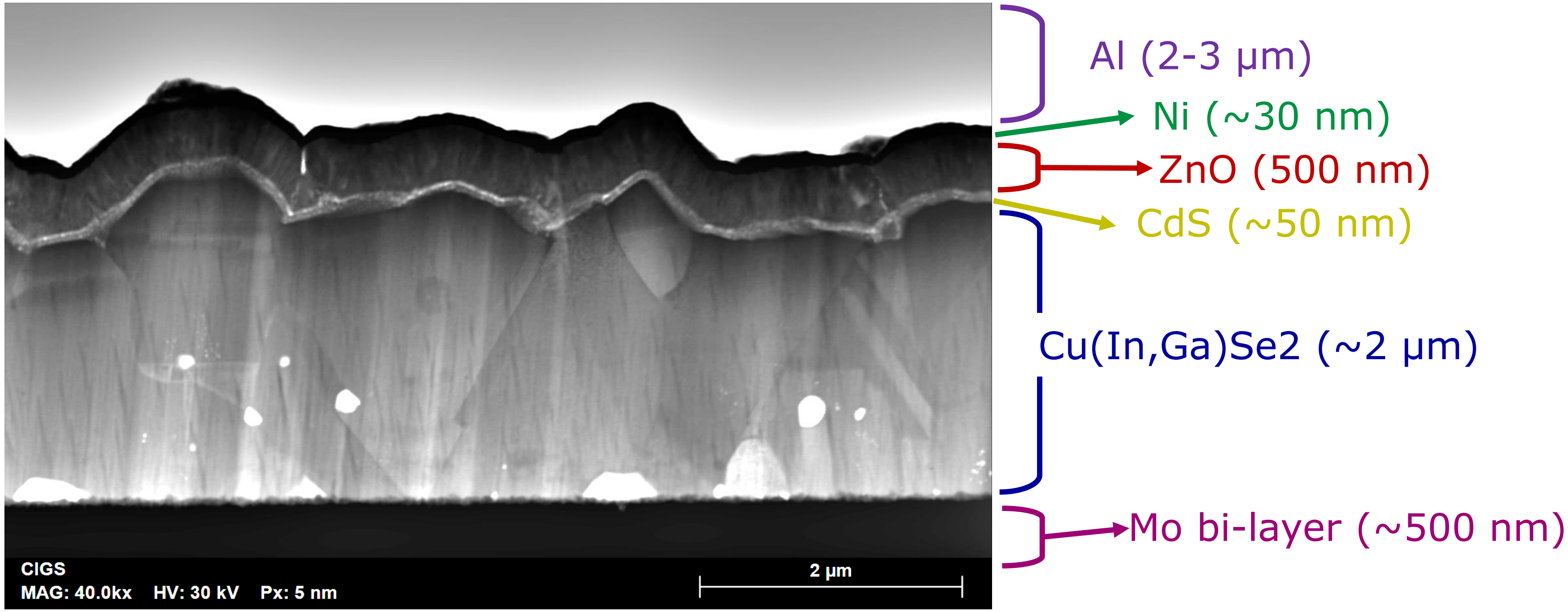
Sample and detectors setup for simultaneous on-axis TKD and EDS measurements on nanomaterials



Combined on-axis TKD and EDS => benefits

- Fastest simultaneous TKD&EDS (enabled by ultra-high solid angle of FlatQuad 1sr)
- X-Rella mask for blocking most stray X-Rays from reaching the FlatQUAD
- Applications:
  - Phase identification & distribution (same as for bulk samples)
  - Characterization of complex samples with crystalline, semicrystalline and amorphous regions
  - Discrimination of phases creating similar Kikuchi patterns using EDS information

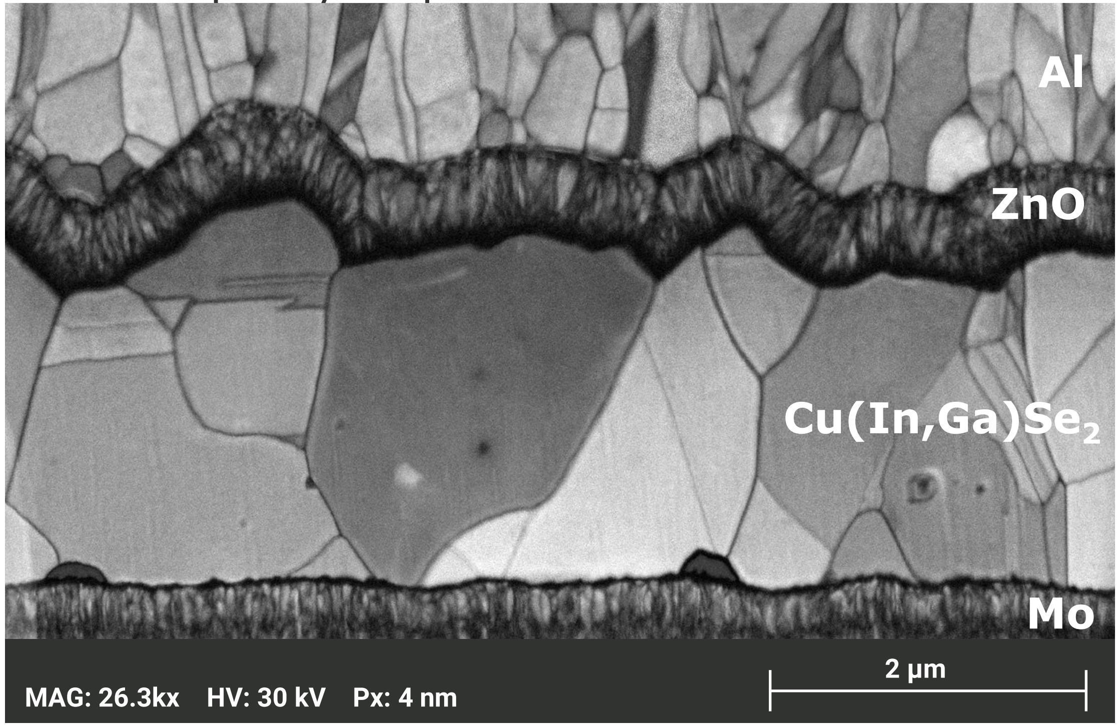
$Cu(InGa)Se_2$ solar cell
FIB lamella

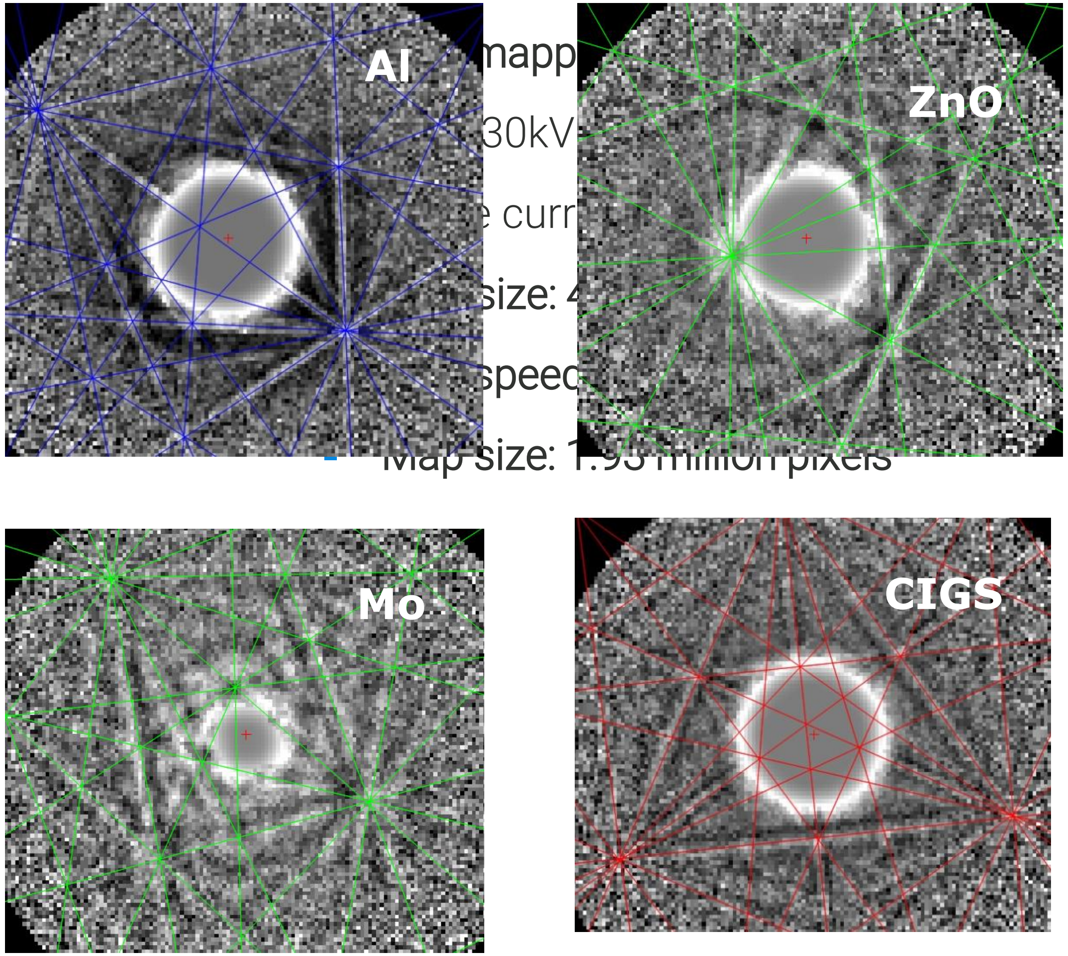


Sample courtesy: M. Raghuwanshi, RWTH Aachen University, Aachen, Germany

$Cu(InGa)Se_2$ solar cell
FIB lamella

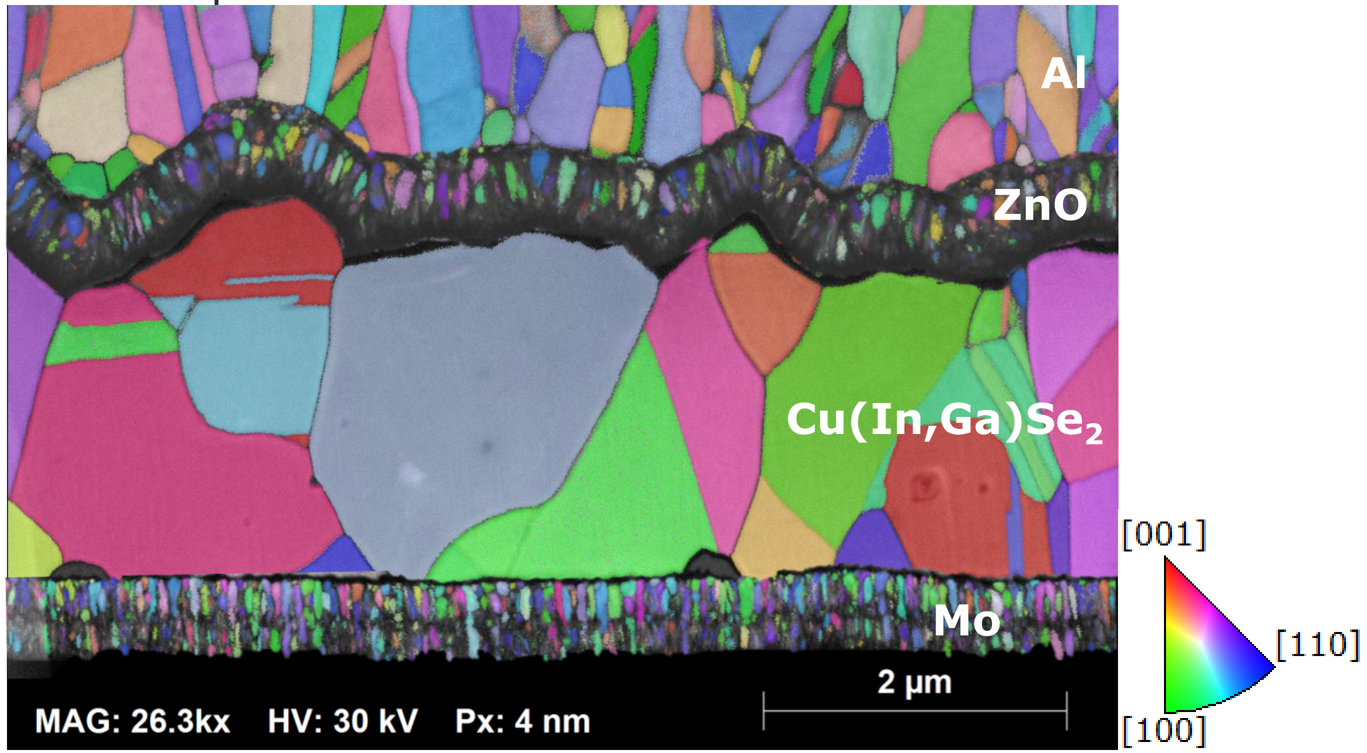
Pattern quality map





Cu(InGa)Se_2 solar cell
FIB lamella

IPF-Z map



Mapping parameters:

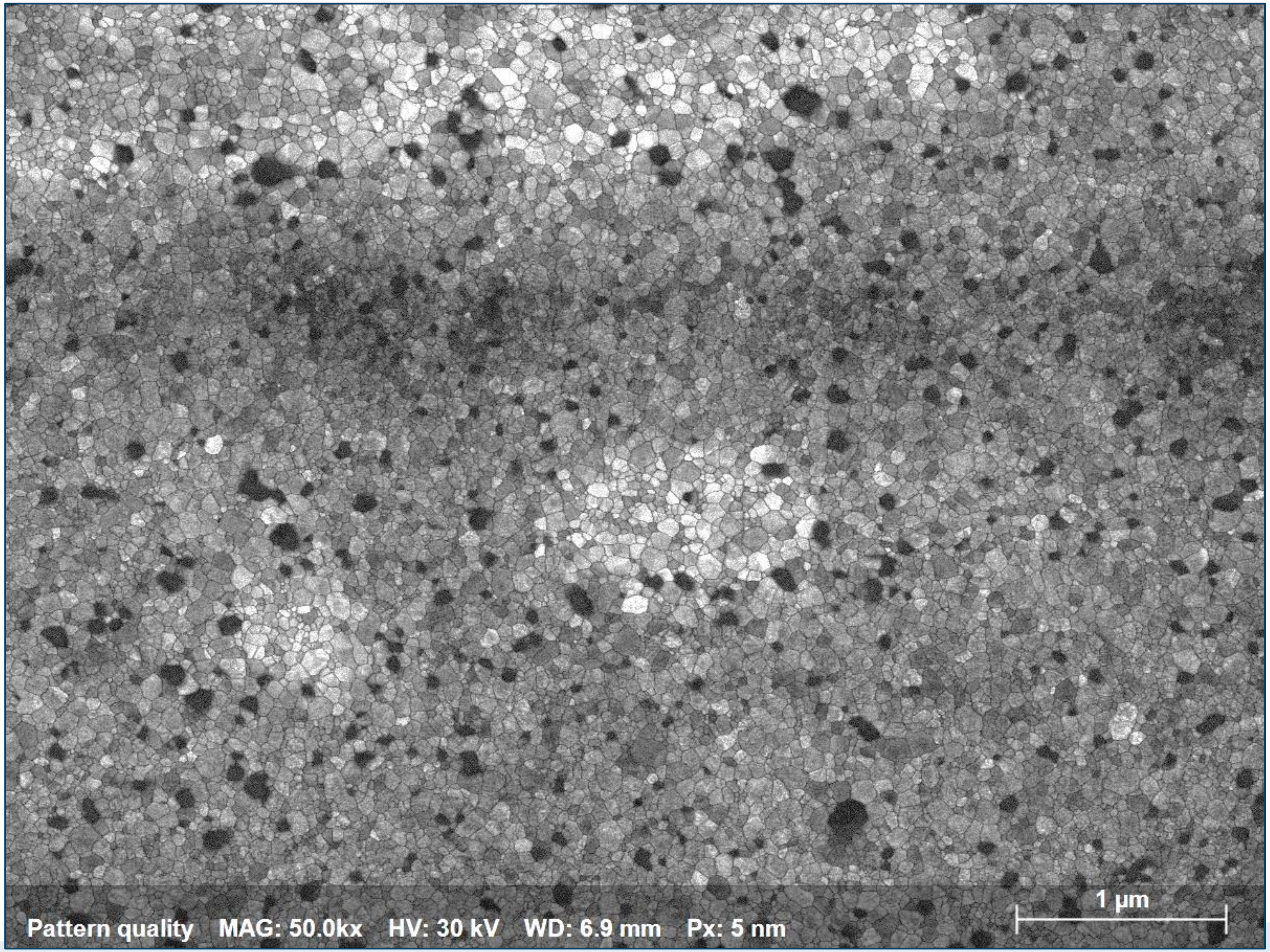
- EHT: 30kV
- Probe current: 1.6 nA
- Step size: 4nm
- Acq. speed: 330 fps
- Map size: 1.93 million pixels
- Zero sol.: 19.3% (whole map)
- Zero sol.: 1.18% on CIGS

Indexing rate in CIGS: 98.82%

On-Axis TKD in SEM

Orientation mapping of Mo thin film – plane view

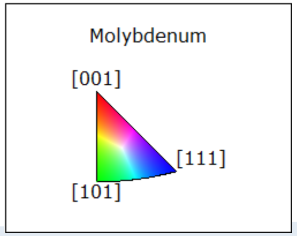
- Map size: +1Mpixels
- Acquisition speed: 626 fps
- Measurement time: 28:52 minutes
- Pixel size: 5 nm
- Field of view: 6x4.4 μm^2
- Pattern Quality Map

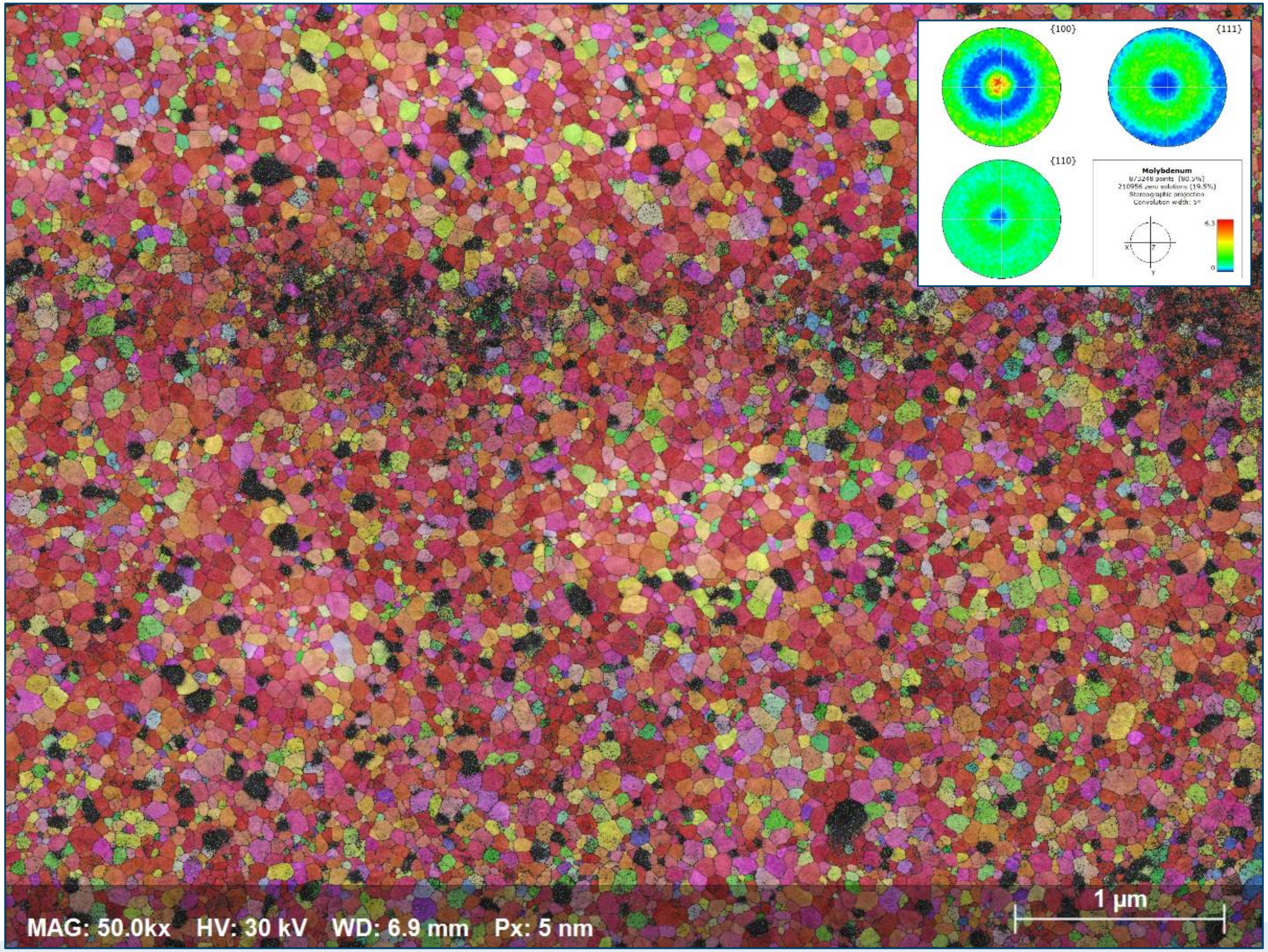


On-Axis TKD in SEM

Orientation mapping of Mo thin film – plane view

- Map size: **+1Mpixels**
- Acquisition speed: **626 fps**
- Measurement time: 28:52 minutes
- **Pixel size: 5 nm**
- Field of view: 6x4.4 μm^2
- IPF map (orientation distribution)
- Map shows raw indexing results
- High indexing rate – no misindexing

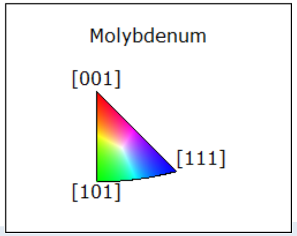


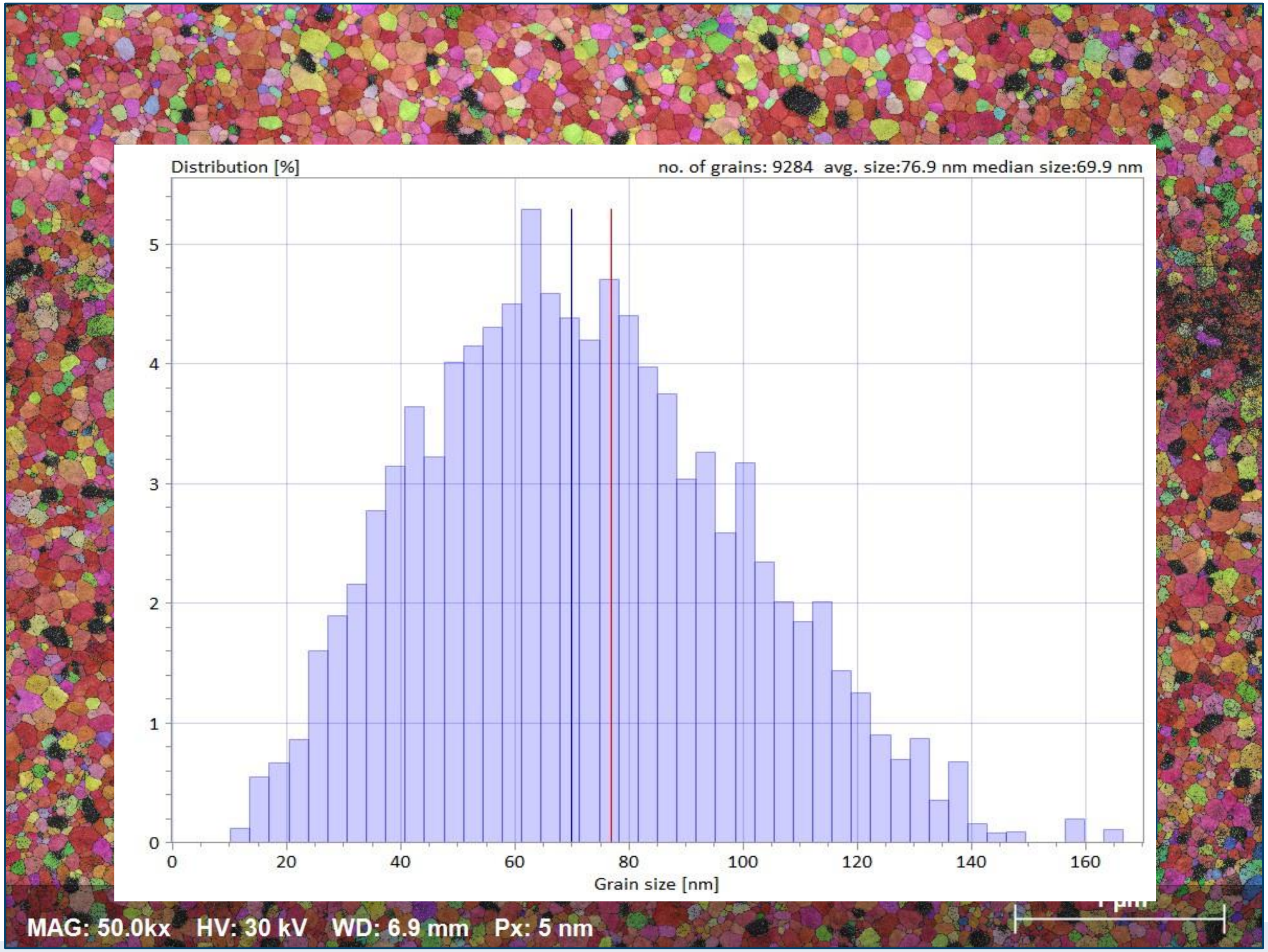


On-Axis TKD in SEM

Orientation mapping of Mo thin film – plane view

- Map size: **+1Mpixels**
- Acquisition speed: **626 fps**
- Measurement time: 28:52 minutes
- **Pixel size: 5 nm**
- Field of view: 6x4.4 μm^2
- **9284 grains**! Average size 70 nm
- Map shows raw indexing results





Summary

- Best spatial resolution period - **Don't miss any detail in your samples**
  - Down to 1.5nm effective spatial resolution*
  - *New* ESPRIT FIL-TKD for orientation mapping using Full Immersion Lens mode on certain e- columns
  - *New* BF-like imaging in mapping position for ideal beam focus optimization
  - *New* Use of high-performance materials for minimized interference with electron beam
- **Understand your samples better** using the best data quality & integrity
  - Maximized SNR during imaging & mapping due to unique on-axis geometry
  - Ideal also for low kV and low probe current applications
  - *New* Improved drift correction due to BF-like imaging in mapping position
  - *New* Sample holders made of high-performance alloys to minimize thermal expansion induced drift

* Dependent on SEM type, vacuum quality & room environment, e.g. vibrations, acoustics, etc.

03

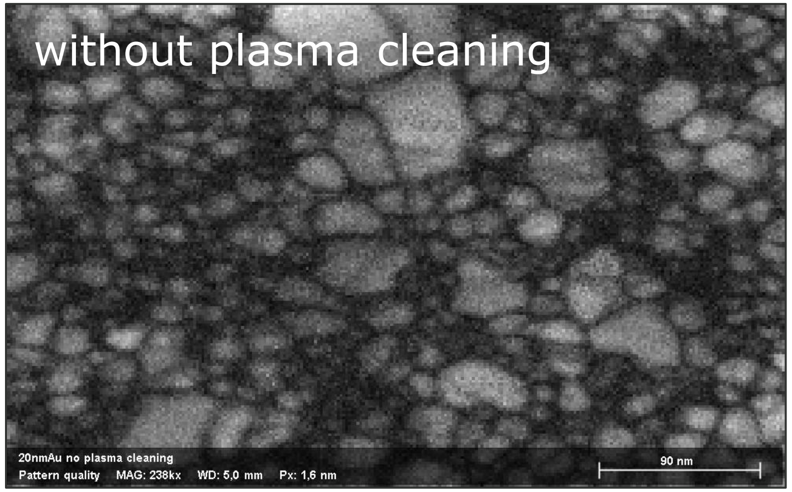
Optimal Sample Preparation for Large-Area TKD

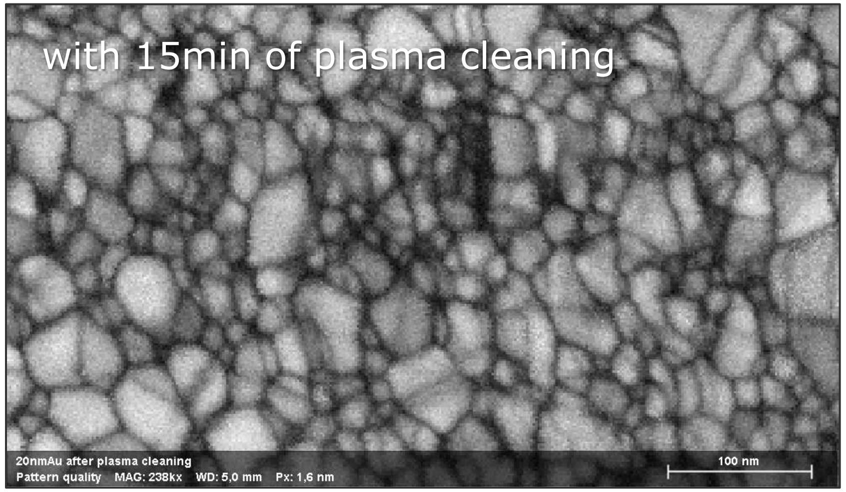
04

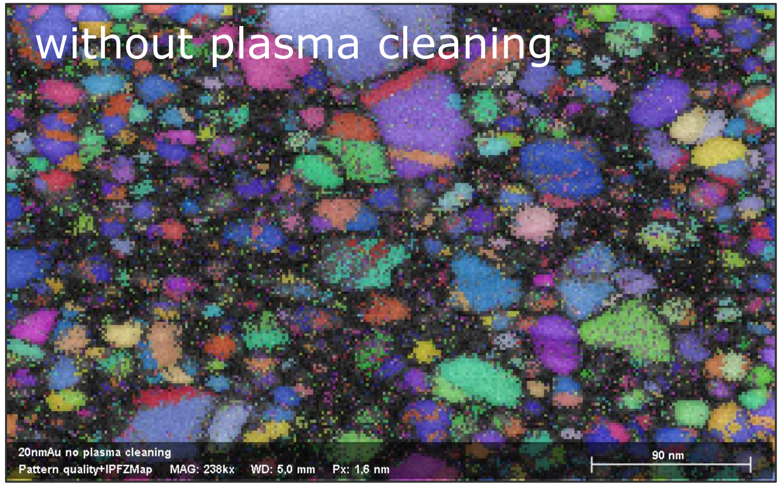
Questions & Answers

please type your questions in the Q&A window and press *SEND*

on-axis TKD in SEM
Effect of plasma cleaning on data quality







20nm Au thin film on Si_3N_4 membrane

- Acquisition at 4ms
- Step size 1.6nm
- e^-Flash^{FS} with OPTIMUS TKD

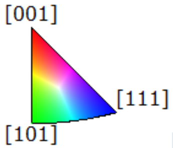
- Beam parameters:

EHT: 30kV / probe current: 1.75nA

- Indexing rate:

46.5% vs. 87.6%

Raw data – no cleaning no processing!

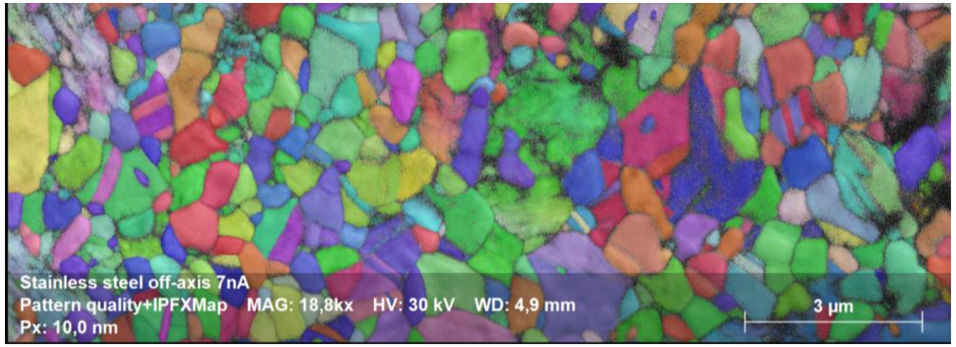


Sample is courtesy of Alice Da Silva Fanta from CEN, DTU, Denmark

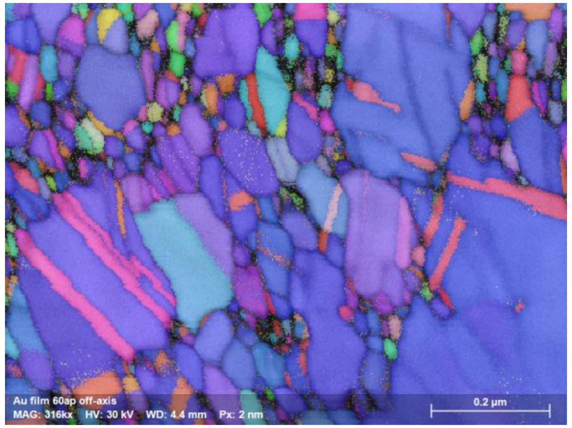
Off-axis TKD vs on-axis TKD

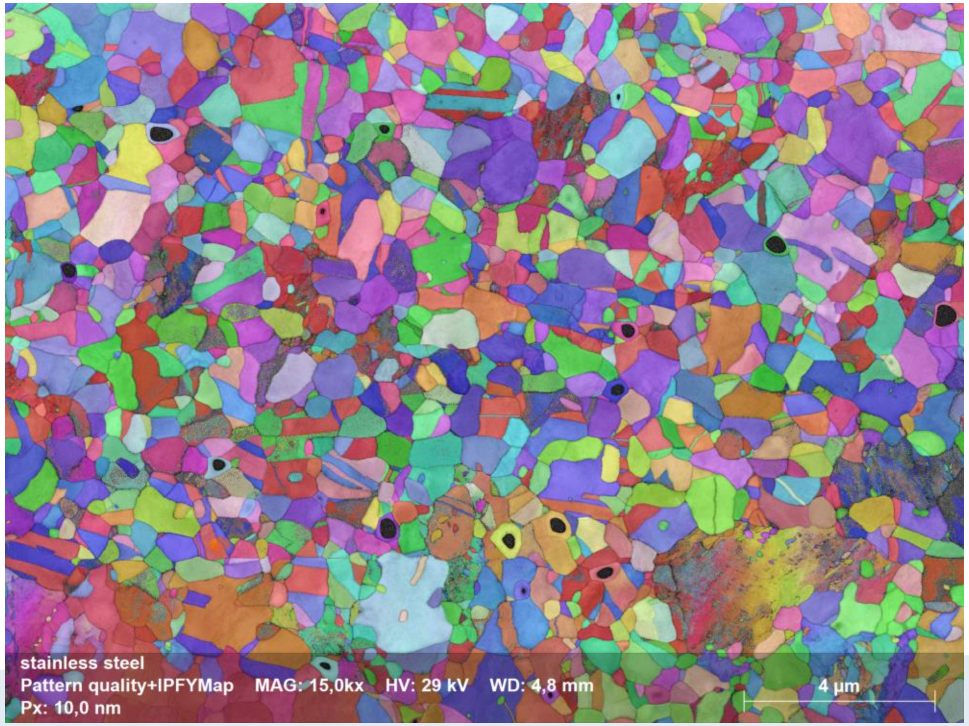
Stainless steel TEM foil (10 nm step, 6nA)

Au thin film (2nm step, 1,6nA)



< OFF-AXIS TKD >





< ON-AXIS TKD >

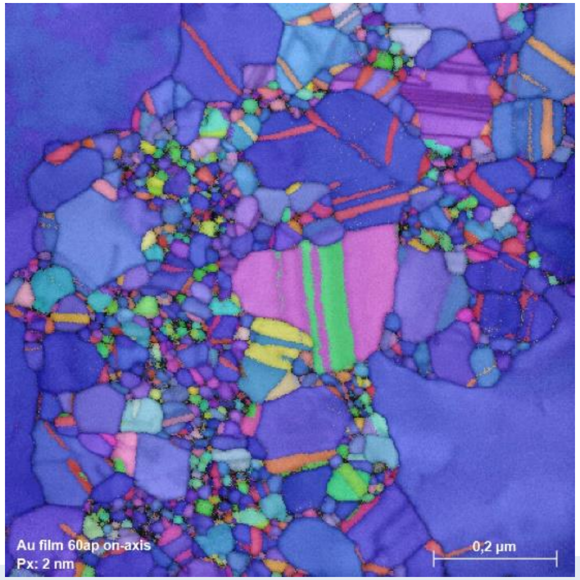
Sharp grain/twin boundaries

Almost no misindexing

Higher indexing rate

Faster speed>>less beam instabilities

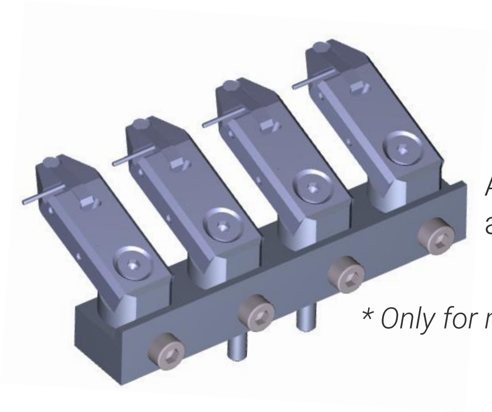
better data quality in less time!



Productivity boost
TKD sample holders

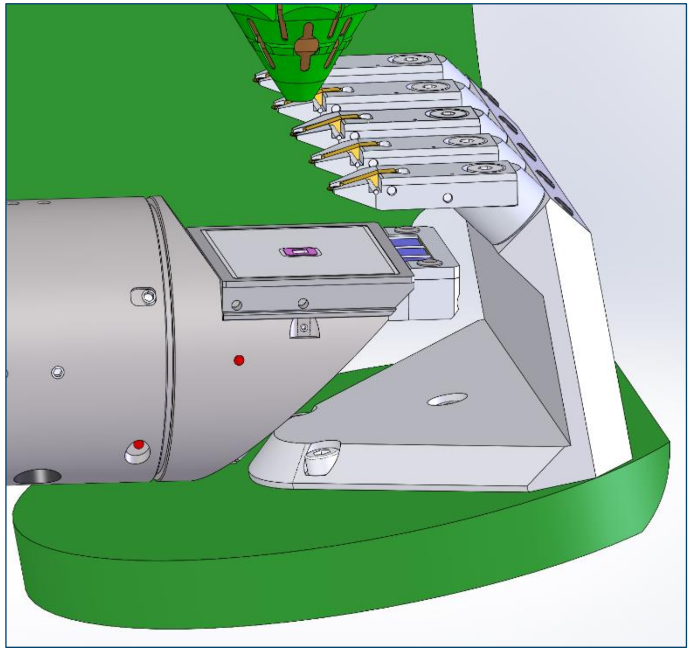
TKD holder (EP000002824448B1) in various configurations:

- Designed for increased productivity & data integrity
- Various concepts fitting most SEM models and airlocks
- Flexible design to allow quick upgrades based on customer requirements

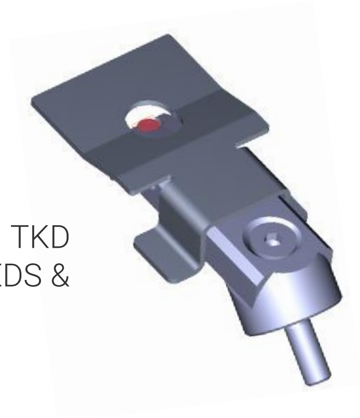


Adapter for Zeiss, TFS* and TESCAN SEMs

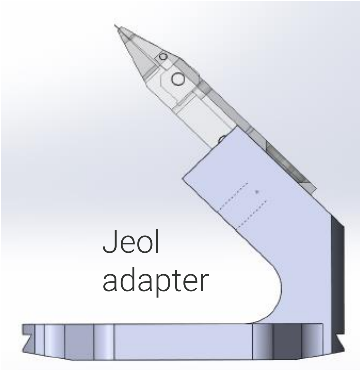
** Only for models with standard DC stages*



Adapter for TFS SEMs with 6in Piezo stages

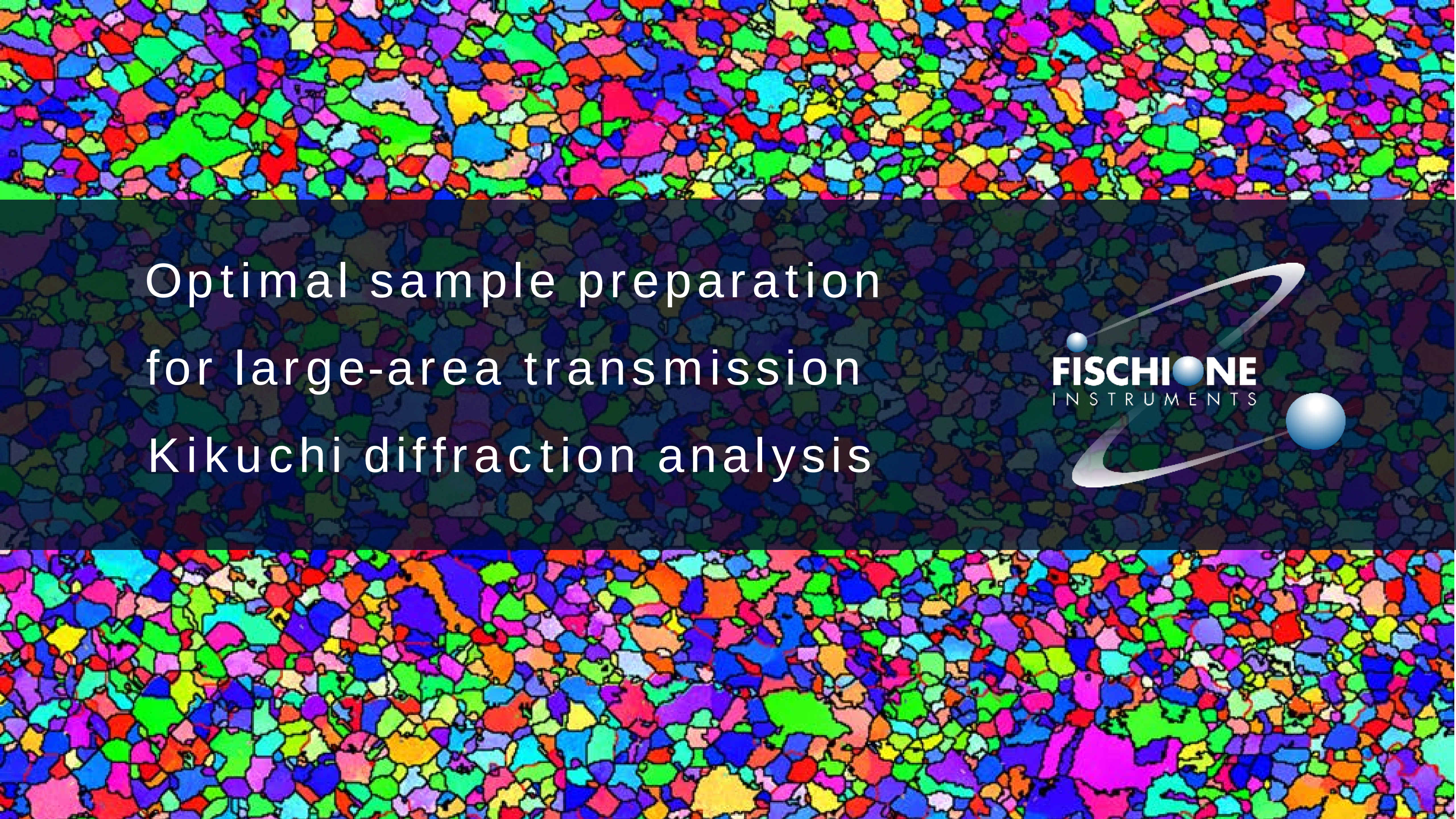


X-Rella mask mounted on TKD holder for simultaneous EDS & TKD mapping



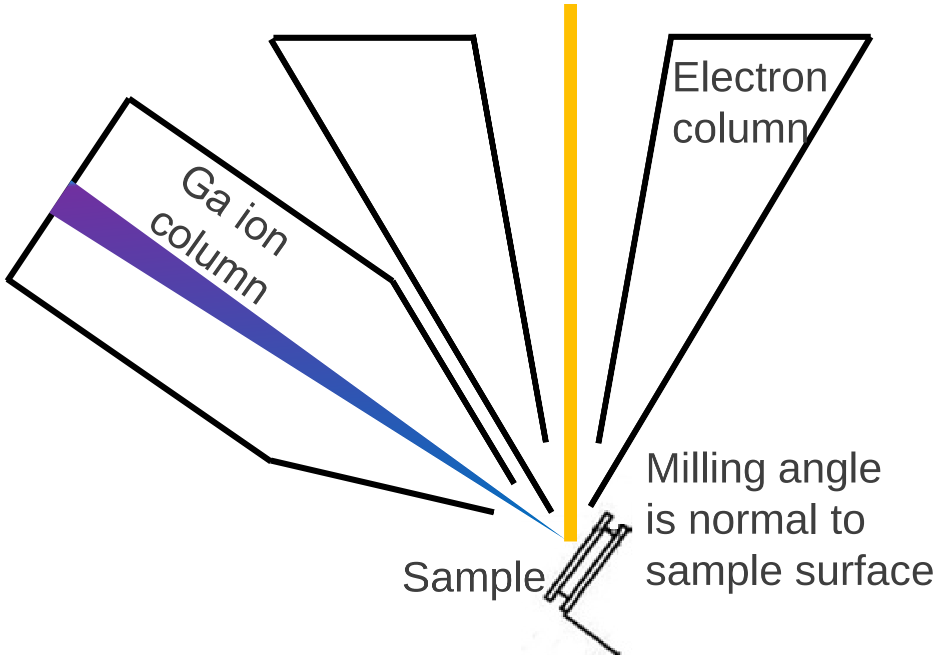
Jeol adapter

Innovation with Integrity


Optimal sample preparation
for large-area transmission
Kikuchi diffraction analysis
FISCHIONE
INSTRUMENTS

Introduction
FIB sample preparation

Focus ion beam (FIB)



FIB milling is a versatile, powerful technique for electron-transparent sample preparation

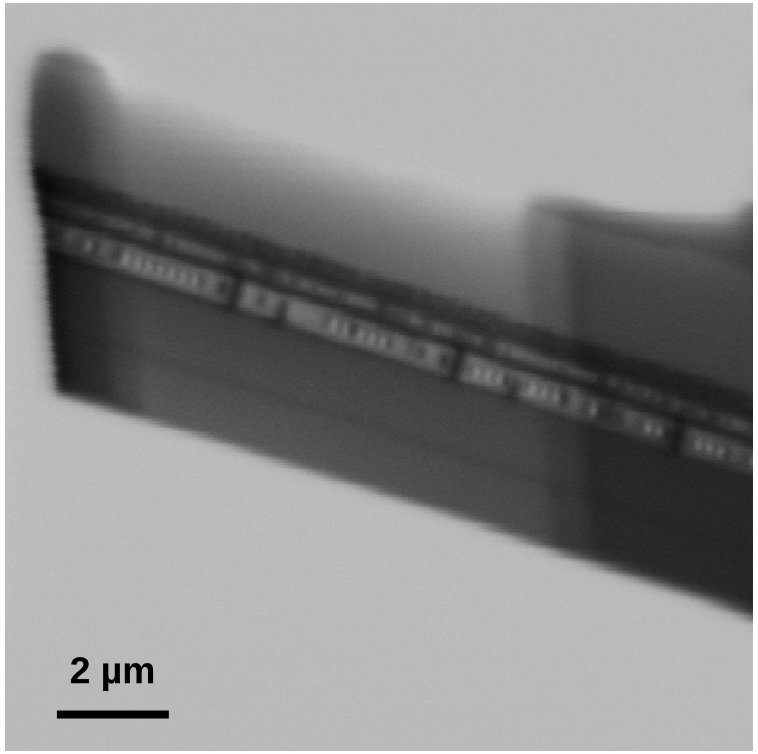
Disadvantages

- Requires significant polishing of the sample surface to remove:
  - Amorphization
  - Ga implantation
  - Structural damage
- Control of sample thickness can be complex and time consuming

Introduction

FIB sample preparation disadvantages

Excessive FIB sample thinning can lead to lamella banding

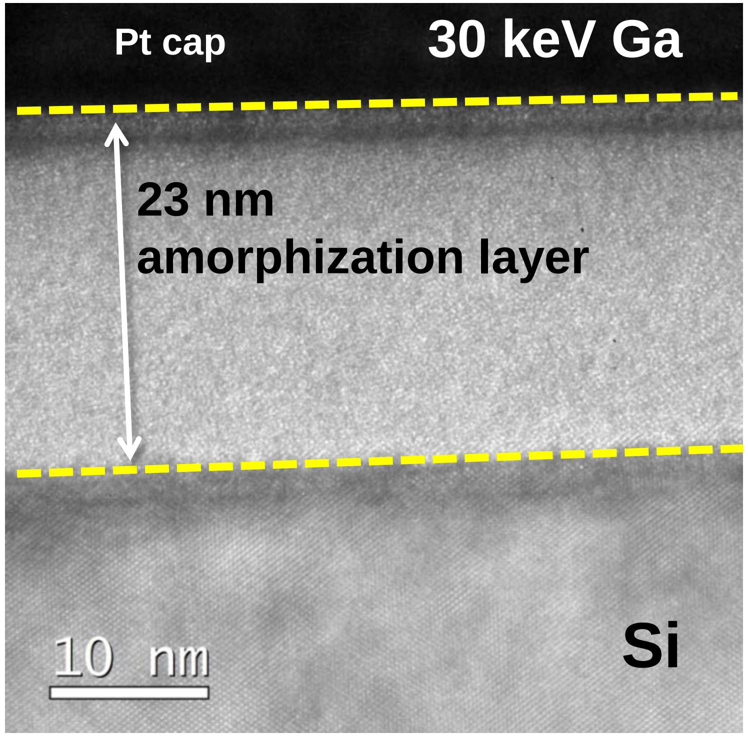


Non-uniform sample thickness. Sample very thin on front side and thick on back side.

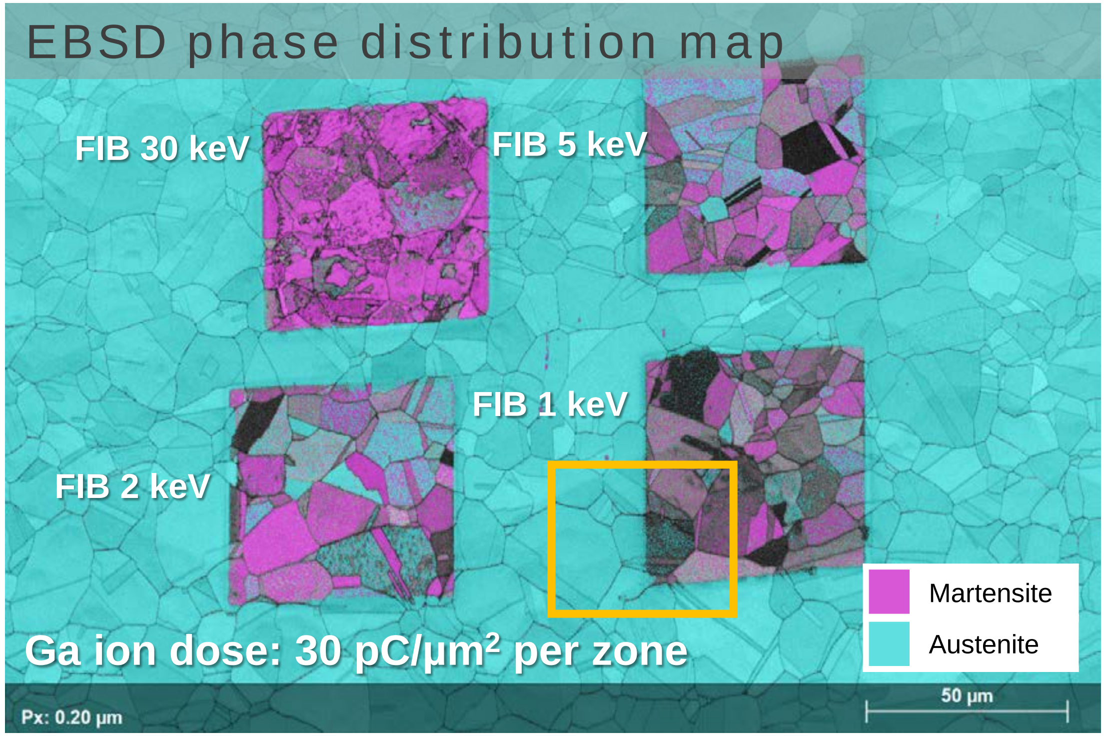
Neither situation is ideal for large-area TKD acquisition

Introduction

FIB-induced structural damage



FIB-induced amorphization and ion implantation.*



FIB milling introduced enough atom displacement and vacancies to cause dynamic strain-induced phase transformation of austenite to martensite. Ga ions can penetrate austenite steel up to 300 nm at 30 keV, depending on grain crystal orientation.†

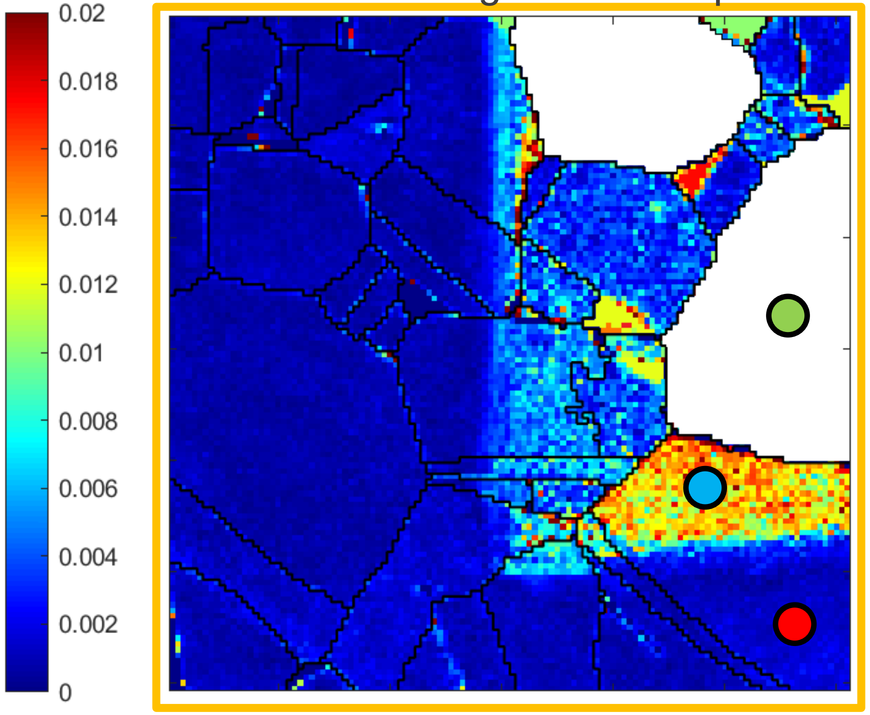
*Bonifacio, C., ISTFA Proceedings, ASM (2018)

†R. Prasath Babu, et al. *Acta Materialia* 120 (2016)

Introduction

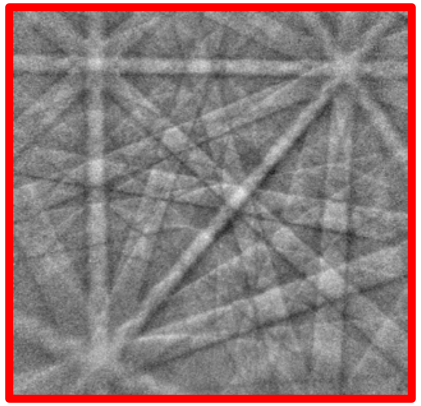
FIB-induced structural damage

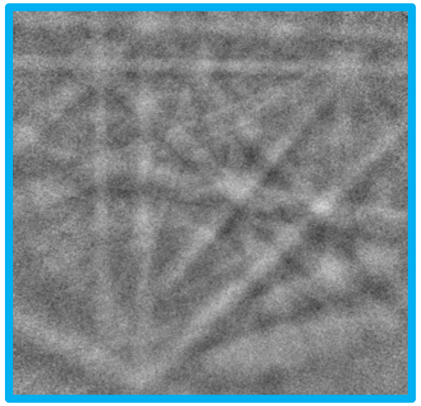
HR-EBSD average strain map

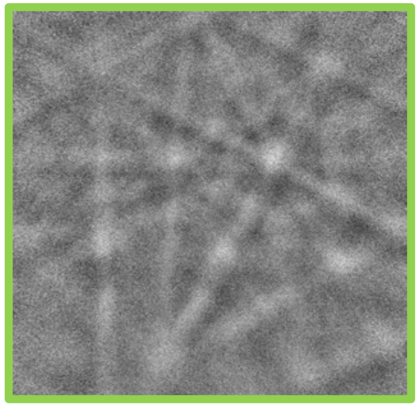


Ga ion penetration causes damage in Fe- γ, creating lattice atom displacements

Sample damage after 1 keV Ga FIB







In the area affected by the Ga ion beam, the EBSD pattern is of very poor quality (Kikuchi bands shift and overlap)

P. Nowakowski, et al. *Microsc. Microanal* 28 (2022)

To accurately characterize a material, a sample preparation technique that preserves the microstructure of the material in its native state is necessary.

Model 1040 NanoMill® TEM specimen preparation system

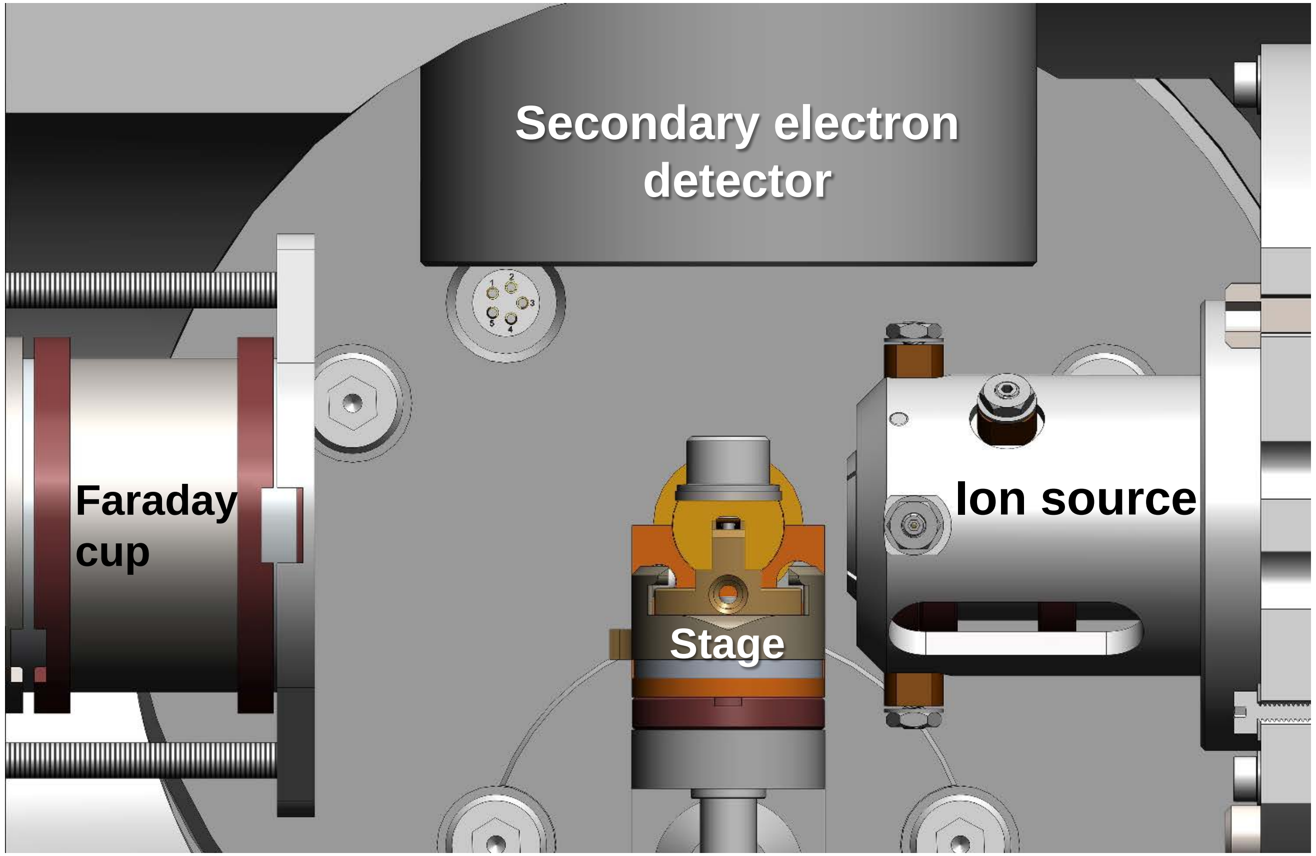
Post-FIB sample preparation system

Low-energy, inert, concentrated ion beam avoids sample damage

- Inert gas (Ar)
- Ultra-low ion energies (~50 eV)
- Small spot size (1 µm)
- Targeted ion beam positioning
- Imaging of ion-induced secondary electrons

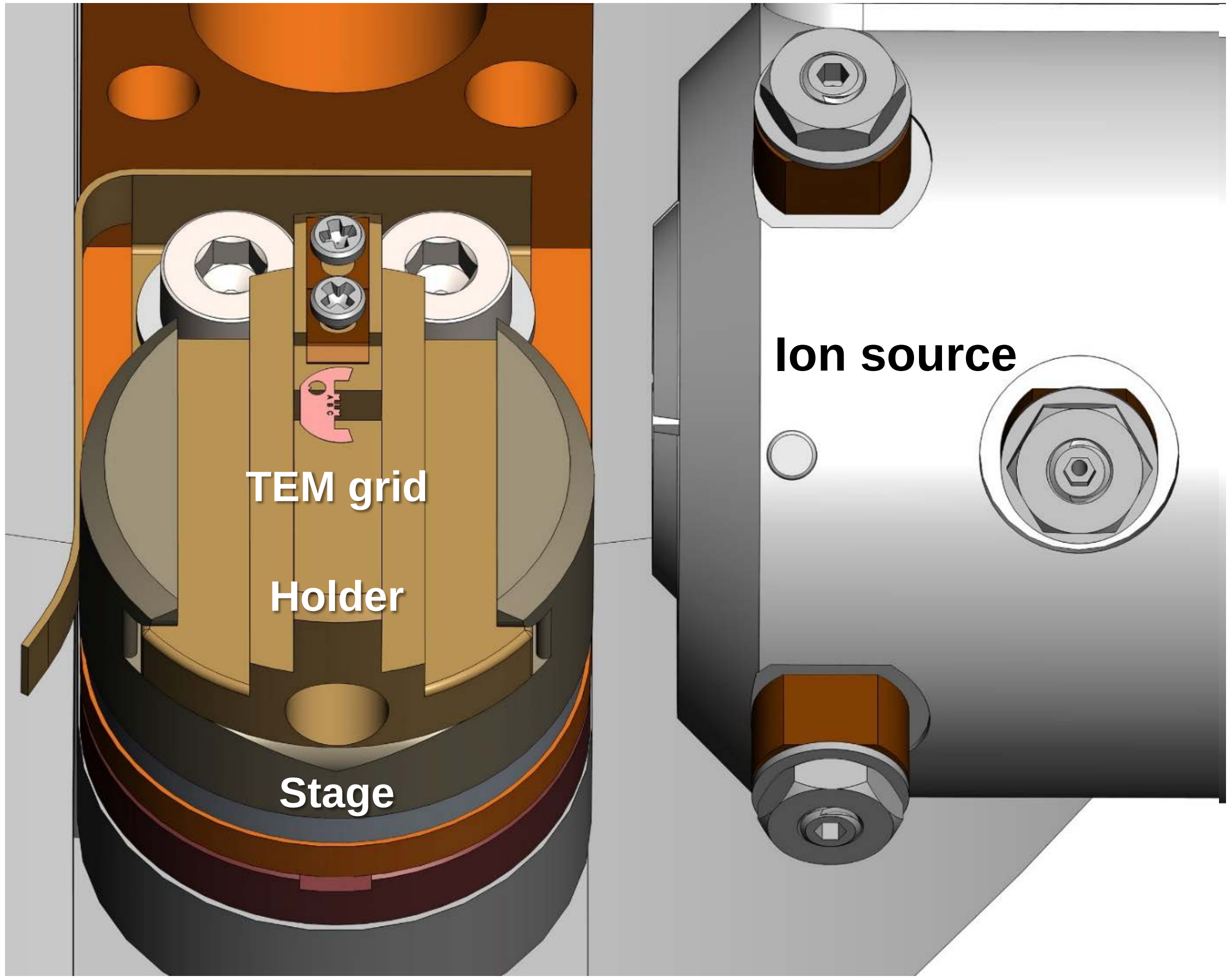
Application of low ion beam energies and currents allow improvement of FIB-prepared specimens for electron microscopy applications

NanoMill system



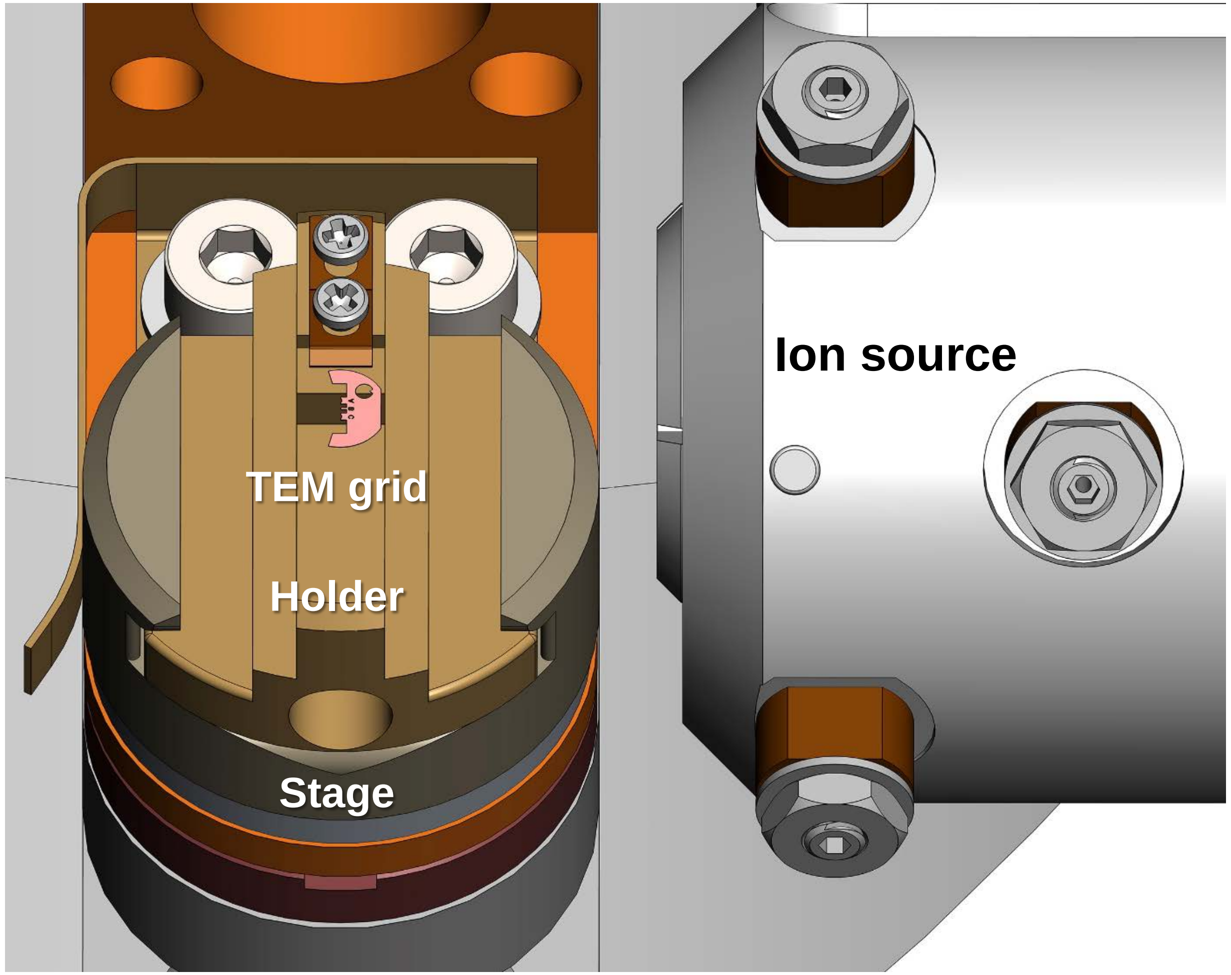
NanoMill system

Milling geometries



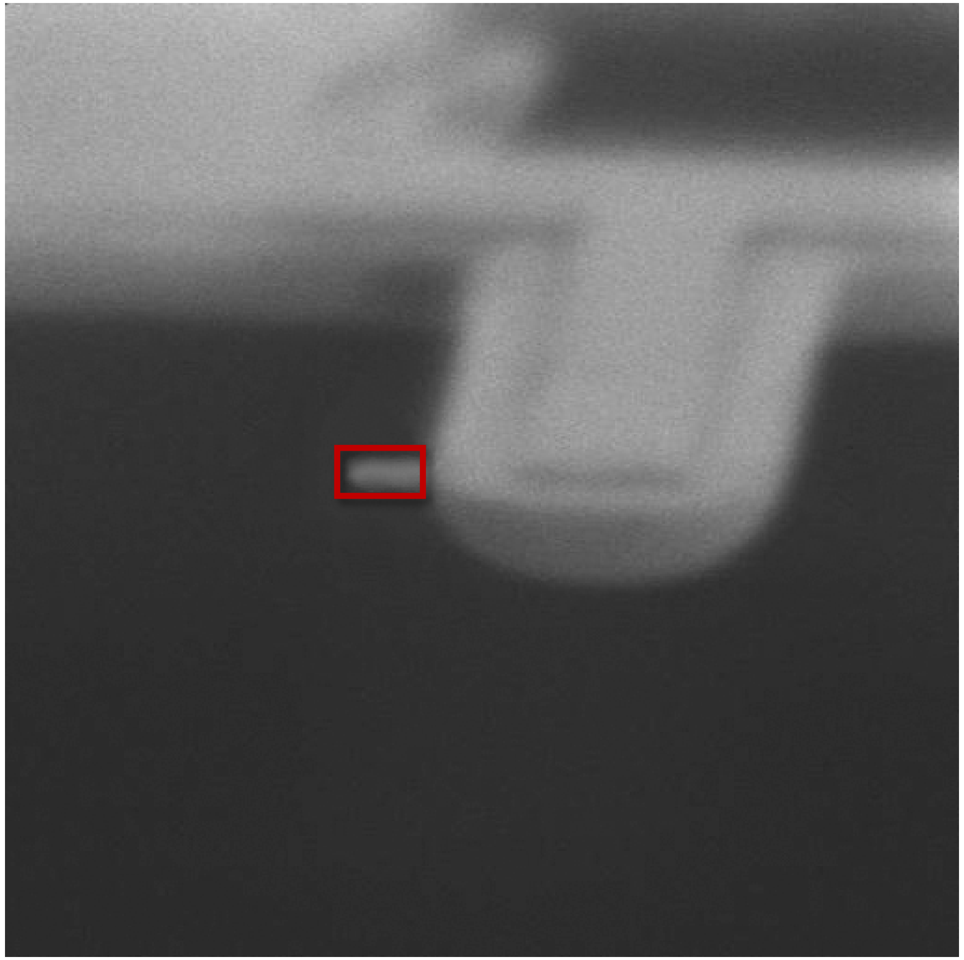
- Ion milling at low energy (50 eV to 2 keV) and low beam current
- Thinning of FIB lamella from the front side

NanoMill system
Milling geometries



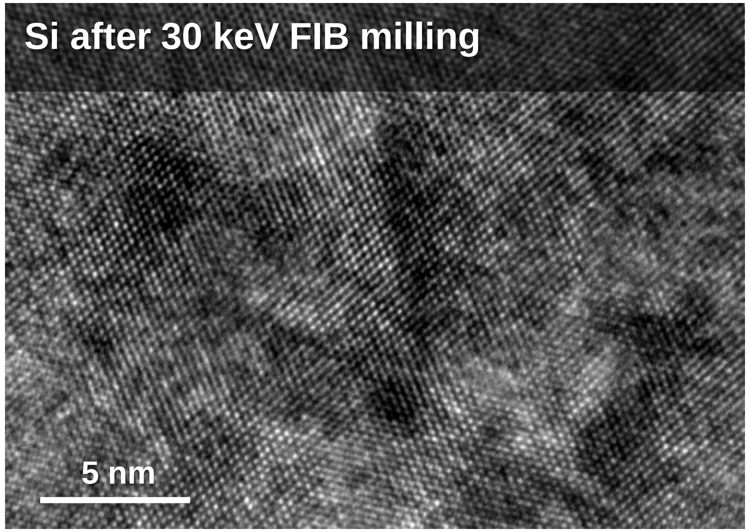
- Thinning of FIB lamella from the back side
- Milling angles up to 30°

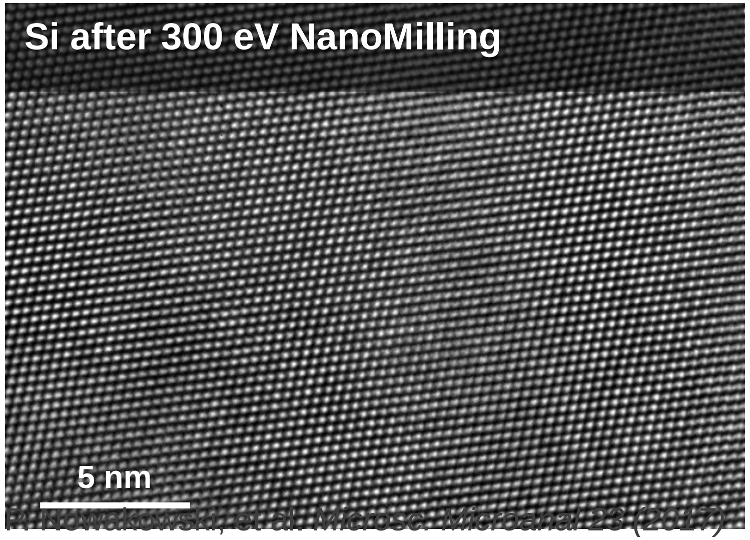
NanoMill system



- Specimen visualization using induced secondary electrons
- Region of interest is precisely targeted by the user
- Micron-sized beam moves in a raster pattern

NanoMill system





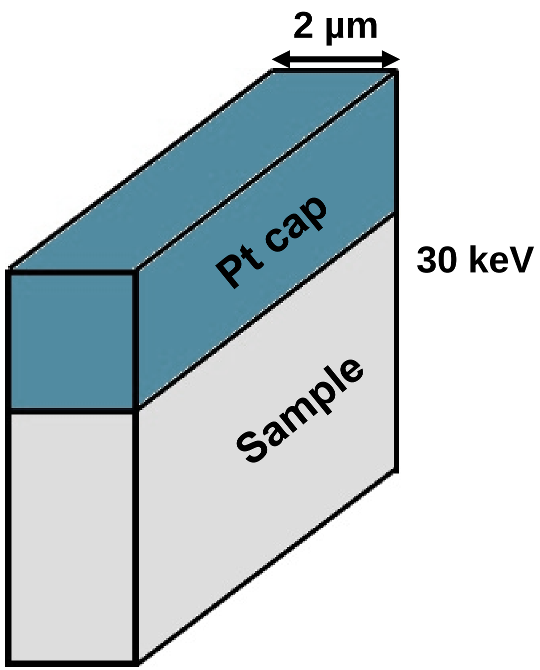
P. Nowakowski, et al. *Microsc. Microanal* 23 (2017)

- Removal of implanted Ga ions
- Removal of FIB-induced amorphization
- Controllable sample thinning

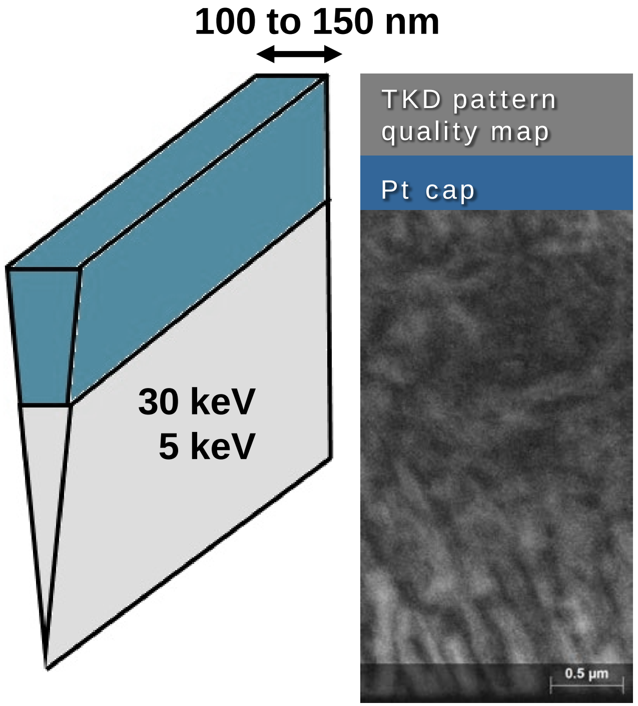
NanoMill system

Large-area TKD sample preparation workflow

FIB sample preparation

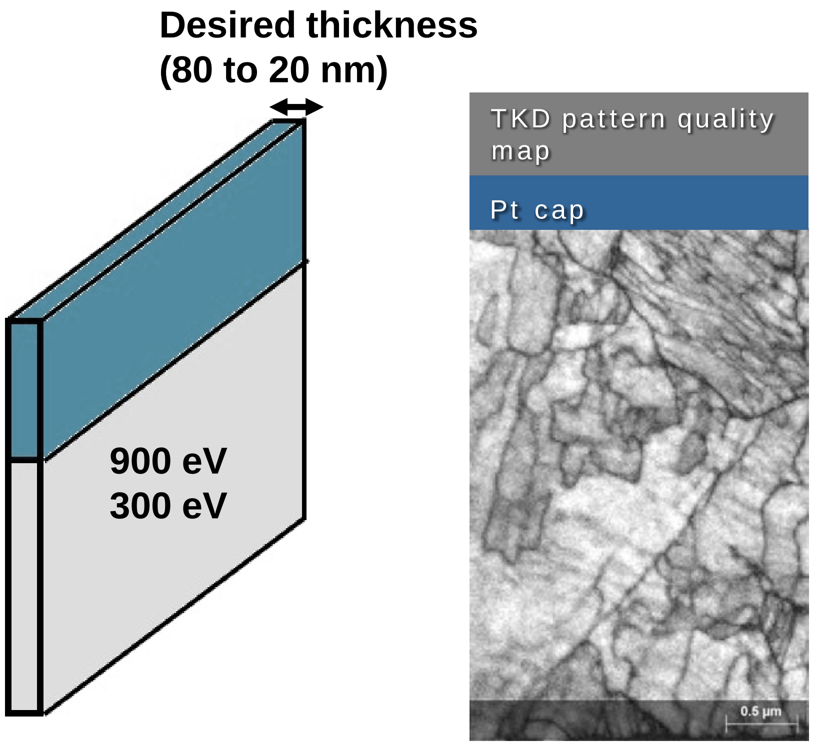


Lamella after standard lift-out



Wedge-shaped FIB lamella milled at high angle from 7 to 2°

NanoMill system

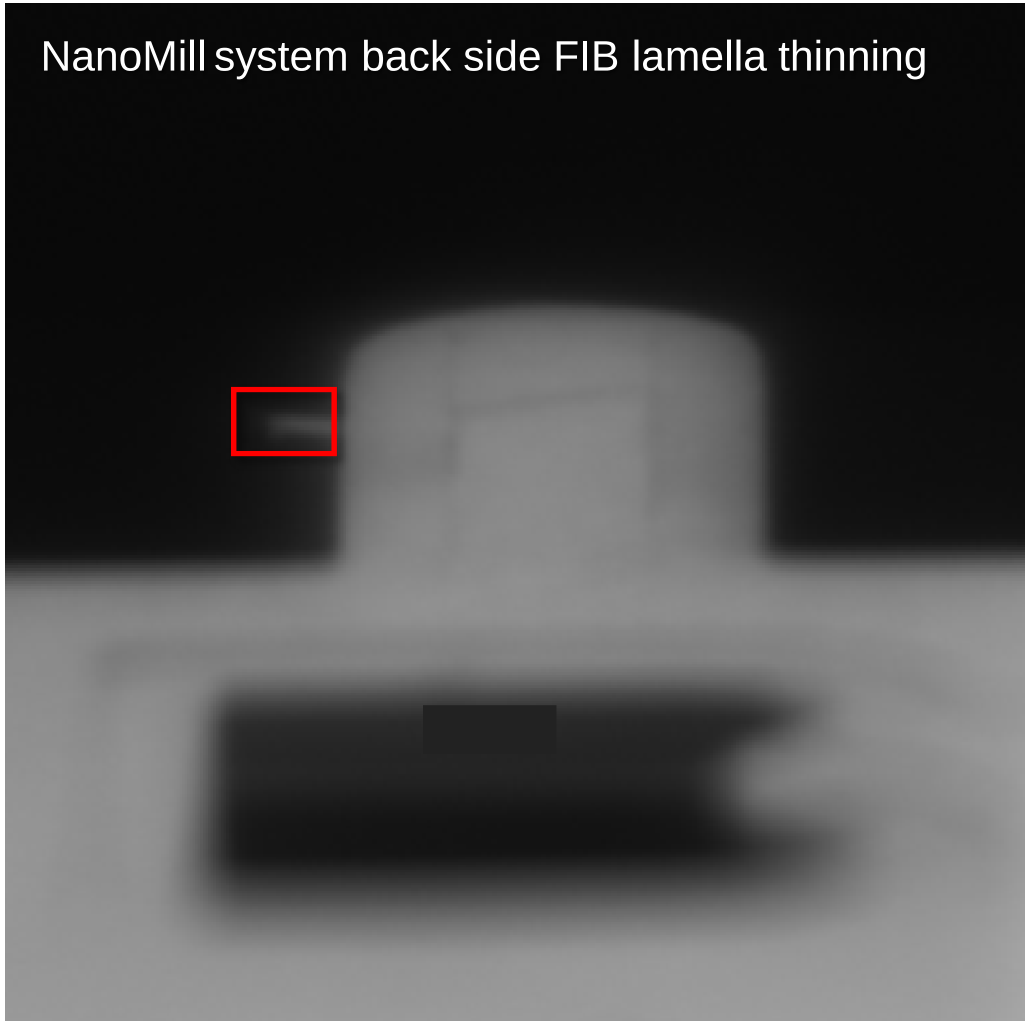


NanoMill system back side thinning at high angle from 30 to 15°

P. Nowakowski, et al. *Microsc. Microanal 29 (2023)*

NanoMill system

Large-area TKD sample preparation workflow

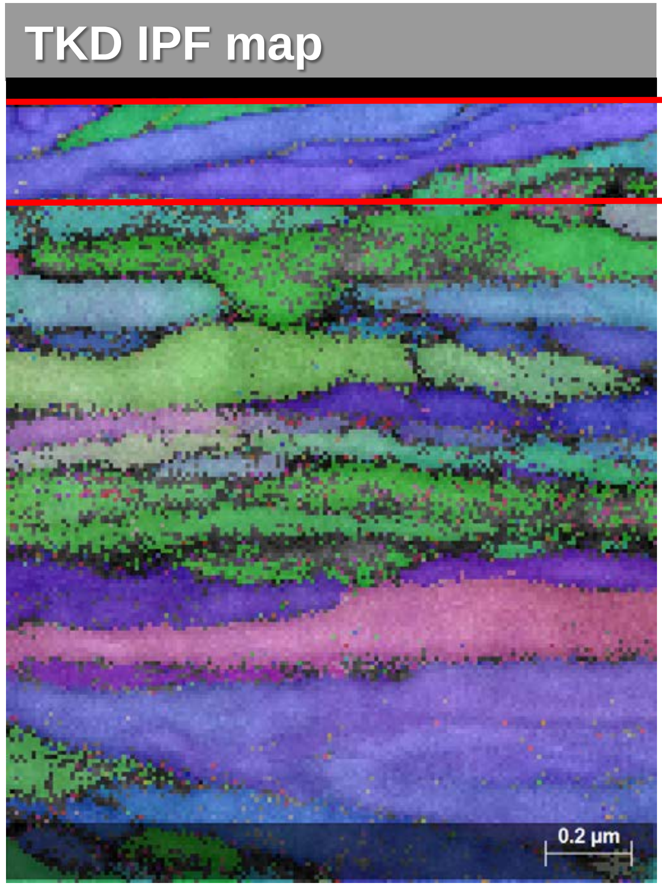


- Ion milling at:
  - low energy (900 eV – 300 eV) and
  - low beam current (100 pA)
- Lamella is thinned from the back side
- Region of interest is precisely targeted; user-defined milling box is 10 x 3 µm
- Specimen is tilted up 30°

Highly deformed Ni alloy (cold rolled; total sheet thickness reduced by 95%)

FIB tool vs. NanoMill system

After 5 kV FIB milling
standard preparation

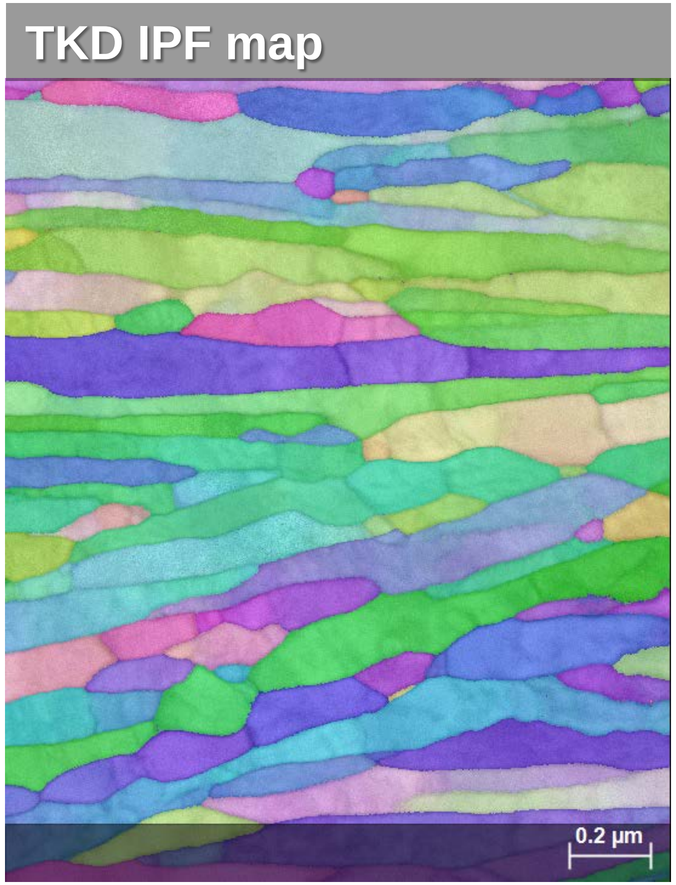


200 nm

Only the front of the lamella is of sufficient quality for accurate TKD acquisition

Indexing rate: 77%

After 500 eV high-angle
back side NanoMilling

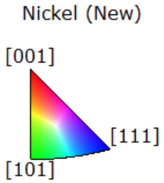


Large area can be easily and accurately measured by TKD

Indexing rate: 97%

Acquisition conditions

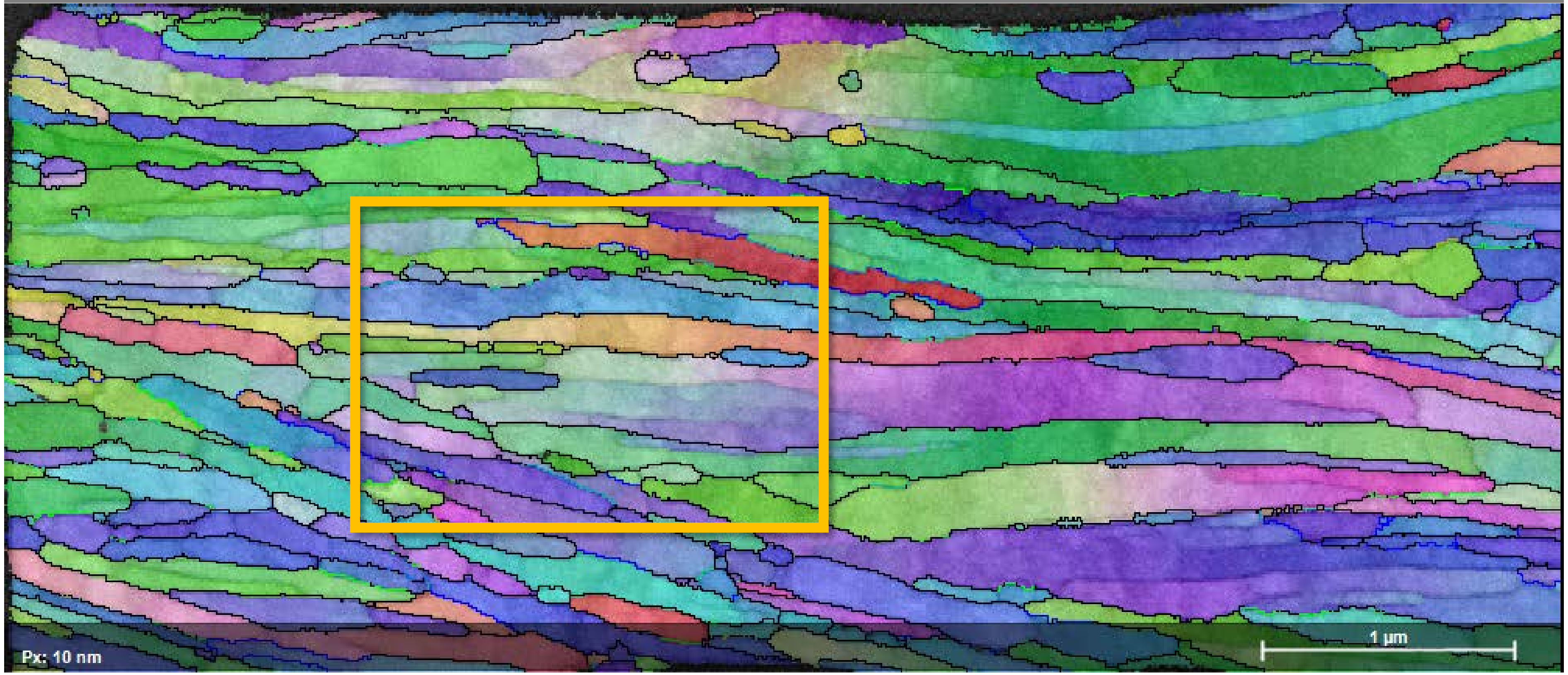
- 30 kV acceleration voltage
- 6.4 nA beam current
- 5 nm step size



Highly deformed Ni alloy

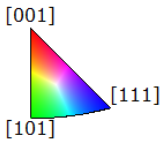
Entire lamella is milled uniformly

TKD IPF map



Acquisition conditions

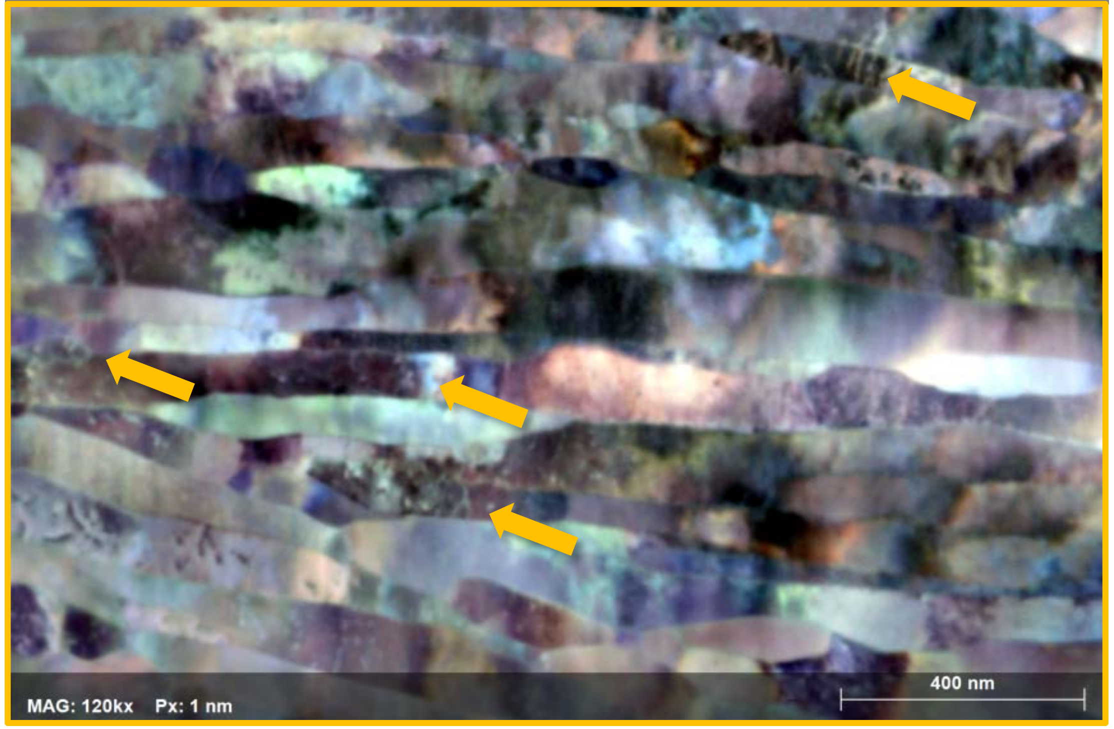
- 30 kV acceleration voltage
- 6.4 nA beam current
- 10 nm step size
- 97 % indexing rate



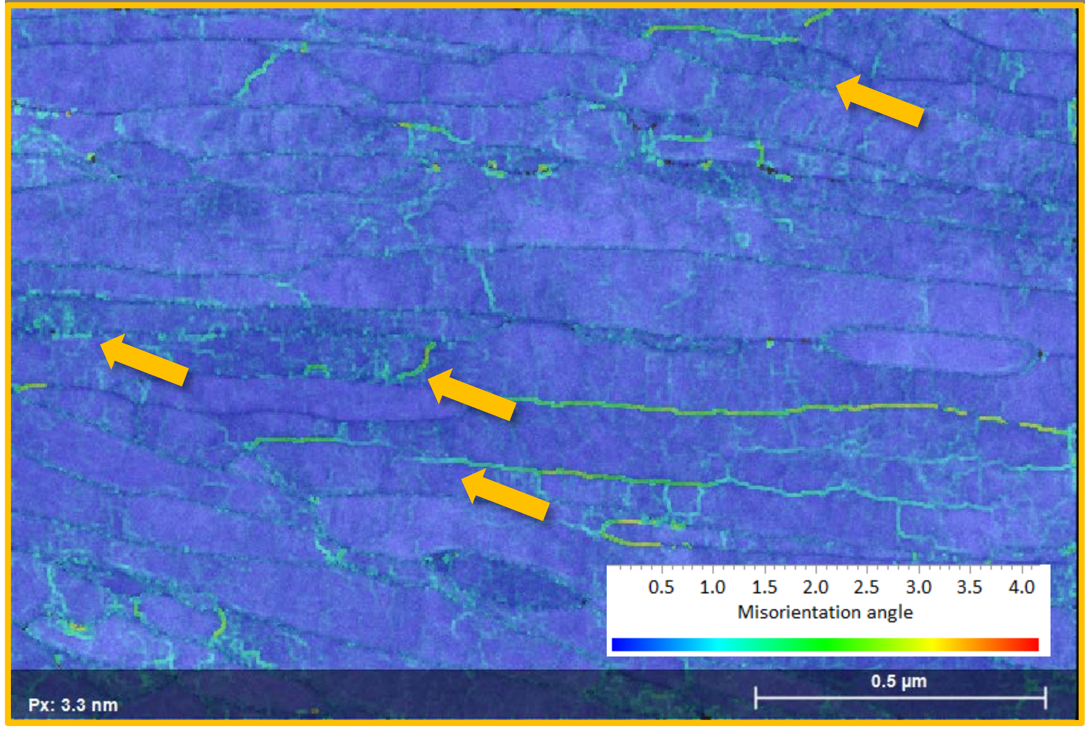
P. Nowakowski, et al. *Microsc. Microanal 27 (2021)*

Highly deformed Ni alloy

Dark field electron contrast image



TKD KAM map



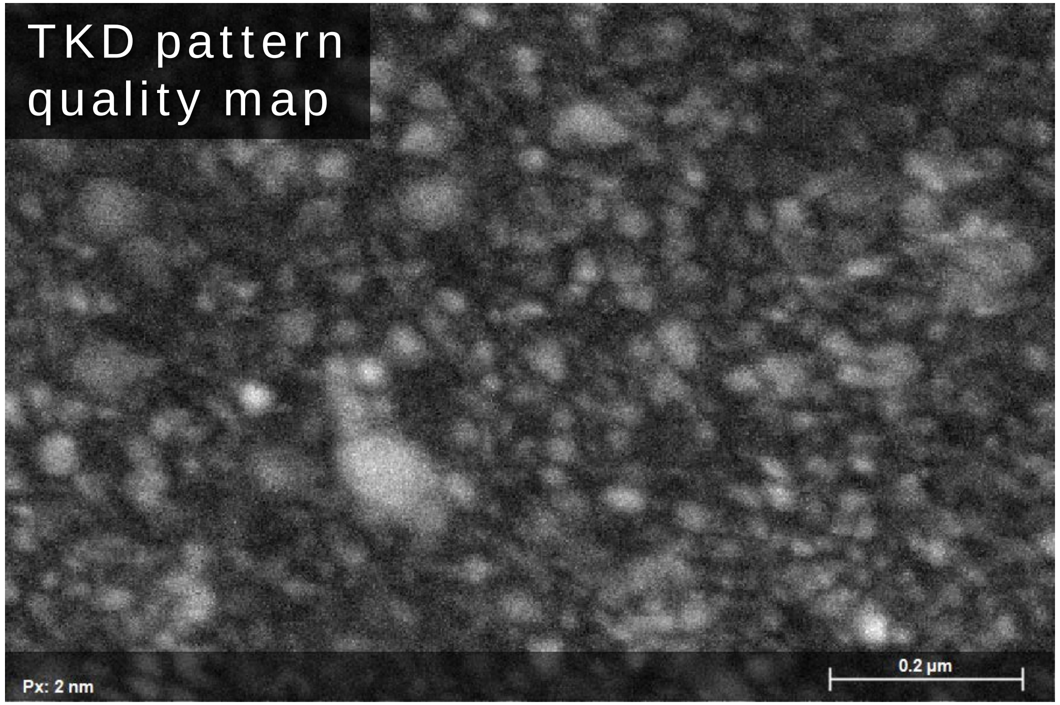
Deformation defects can be observed and correlated with kernel average misorientation TKD measurements

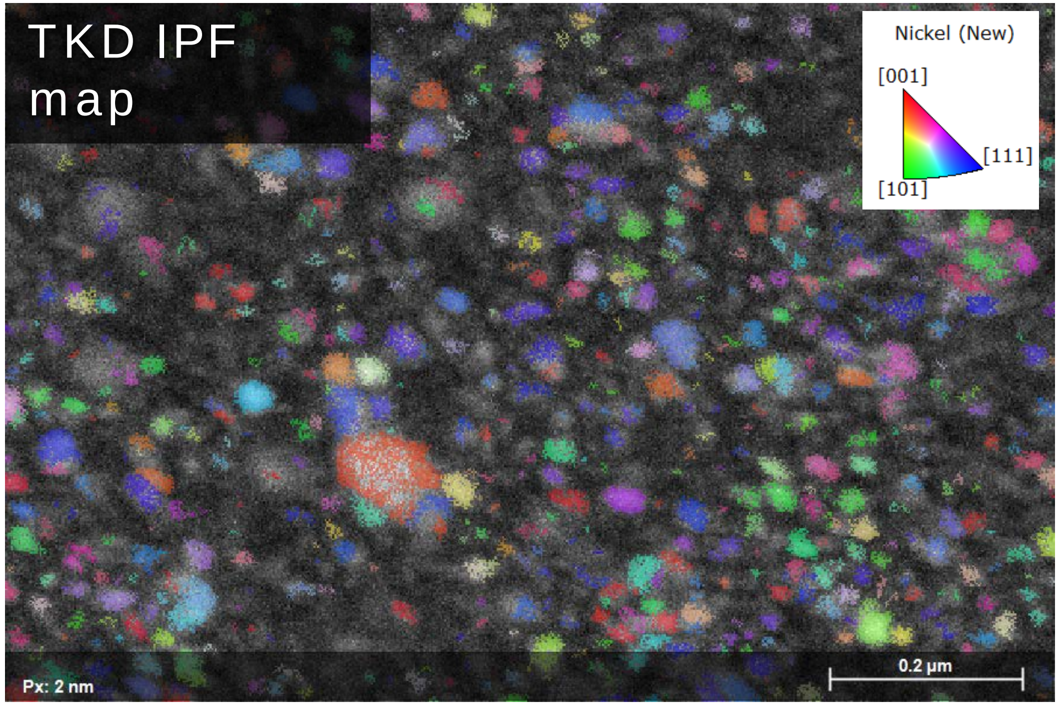
P. Nowakowski, et al. *Microsc. Microanal 27 (2021)*

Nanostructured Ni alloy

Front side standard FIB 5 keV preparation

Sample thickness ~60 nm





- The analysis challenge is the exceptionally fine grain size – significantly less than 50 nm.
- Only grains > 100 nm are indexed.

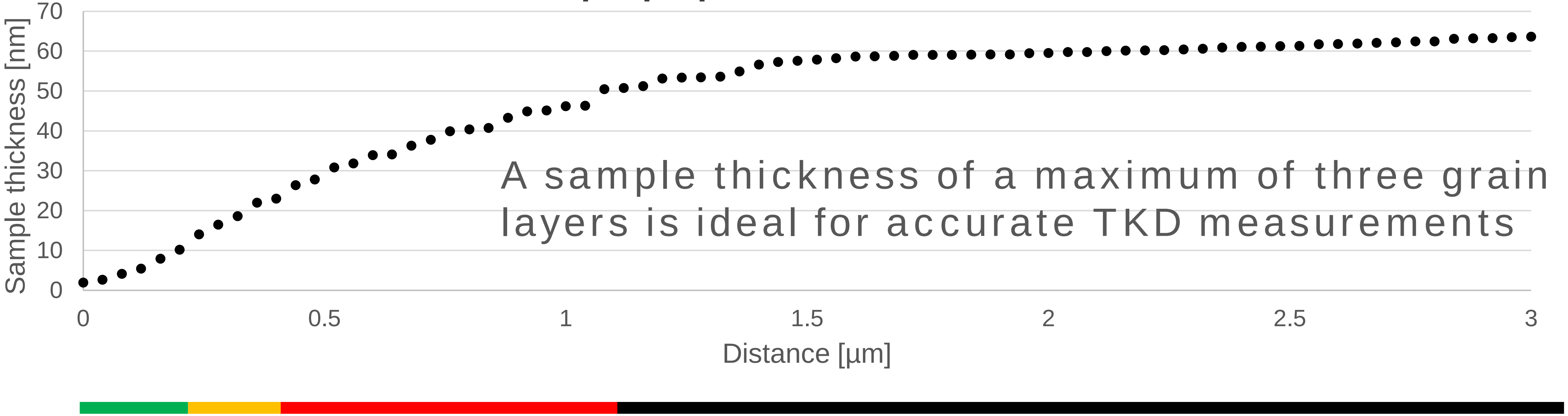
Acquisition conditions

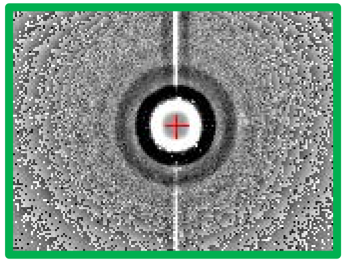
- 30 kV acceleration voltage
- 1.6 nA beam current
- 2 nm step size

Nanostructured Ni alloy

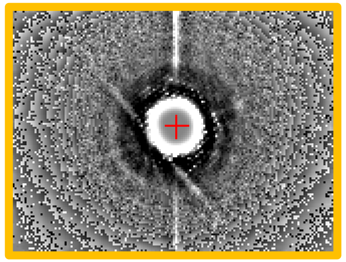
Optimum sample thickness for TKD measurements

Front side traditional FIB sample preparation

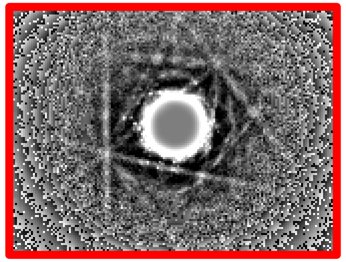




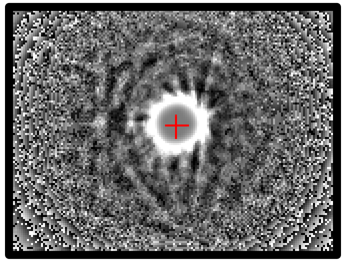
0 to 10 nm



10 to 20 nm



20 to 50 nm

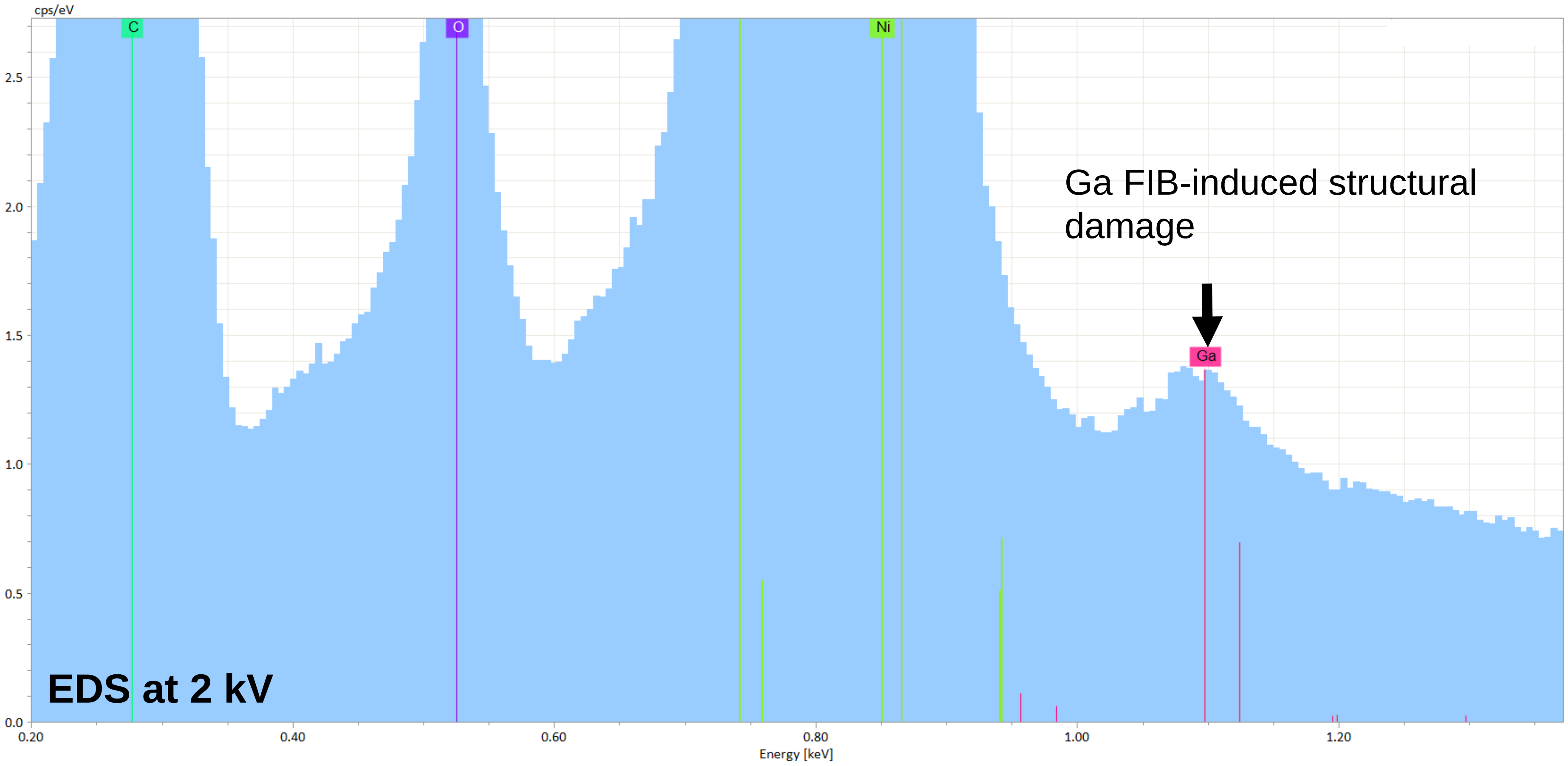


50 to 100 nm

Nanostructured Ni alloy

Structural damage by Ga ion implantation

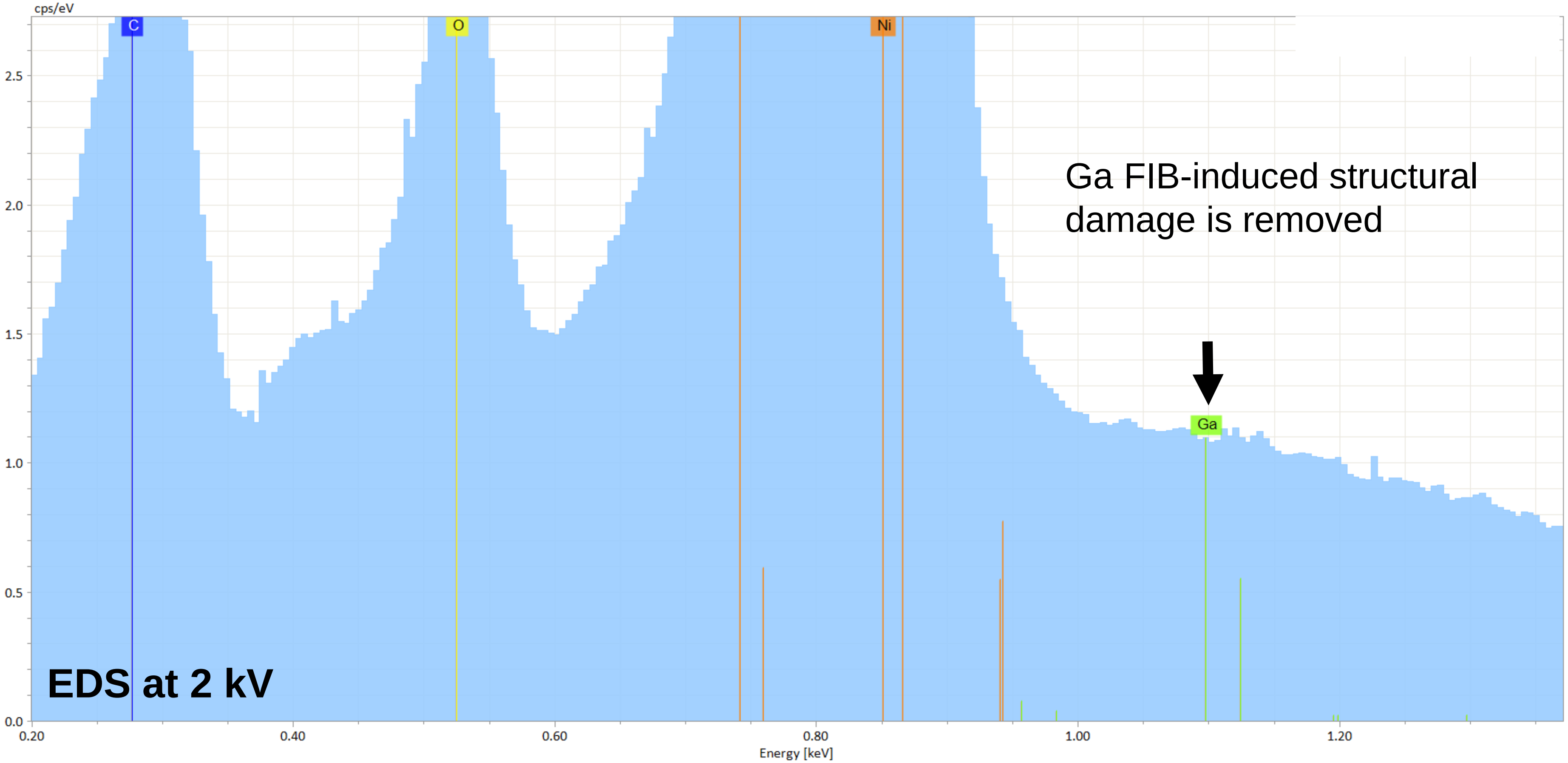
Sample milled by FIB at 30 and 5 keV



Nanostructured Ni alloy

Ga ion implantation / damage is removed after NanoMilling

Sample back side milled by NanoMill system at 500 eV

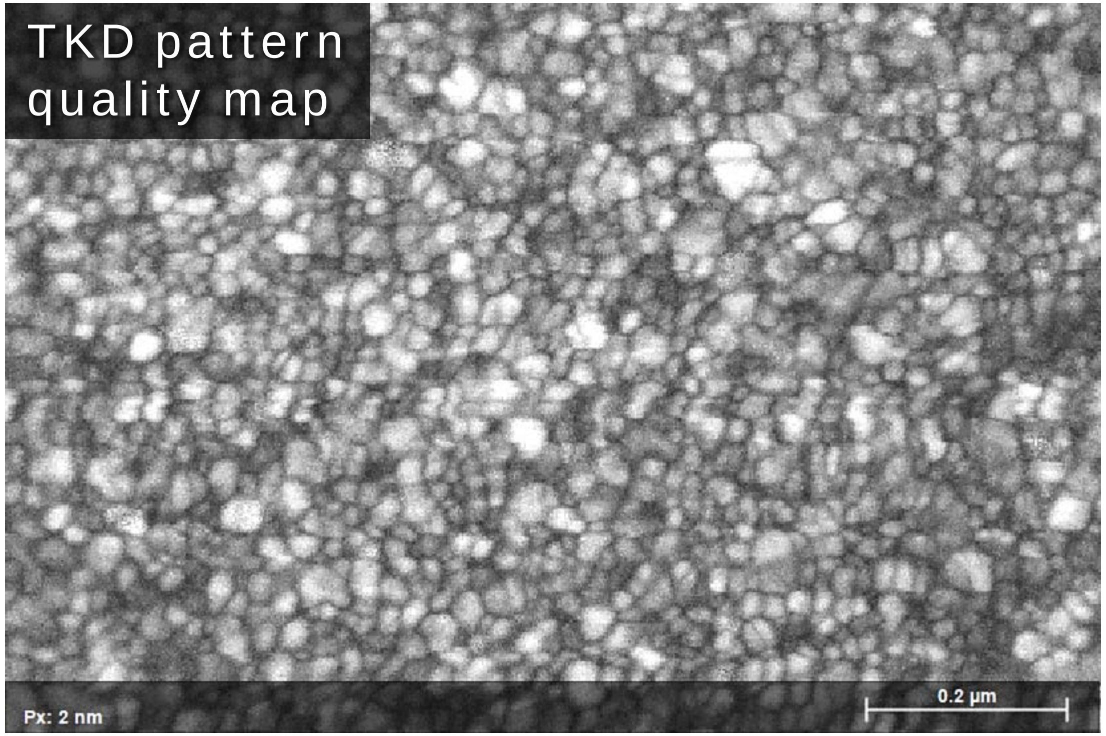


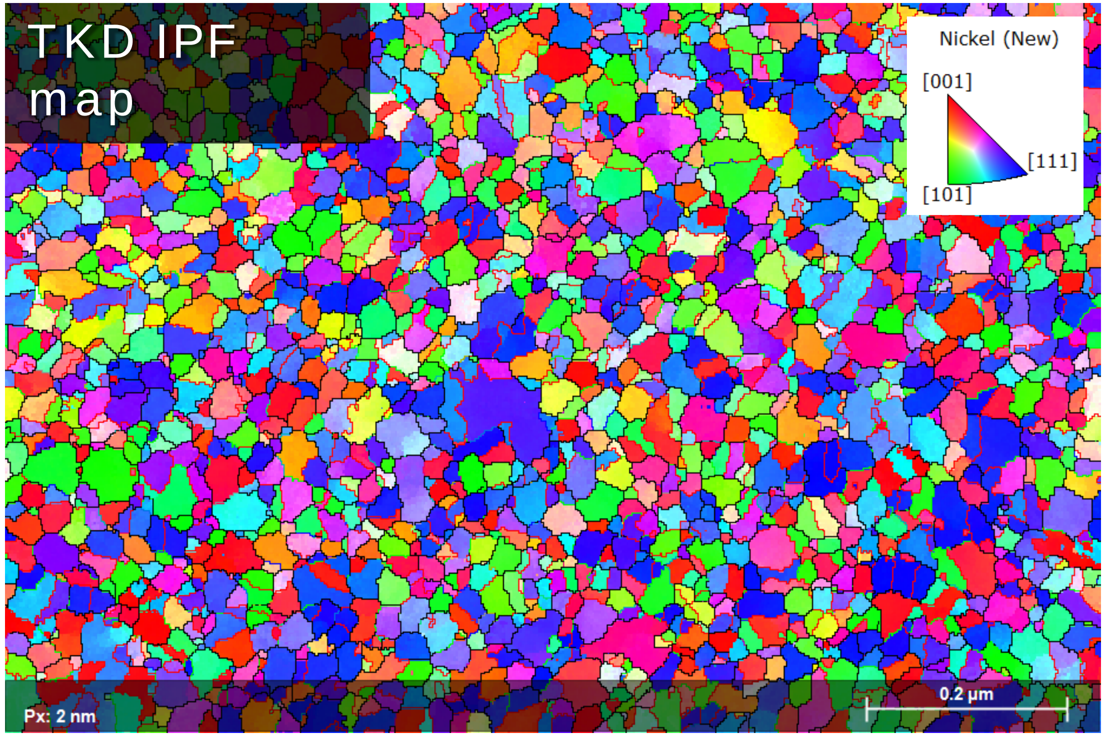
Thickness is not the only concern. Thin lamella must also be free from structural damage for nanostructural material characterization.

Nanostructured Ni alloy

Optimal sample preparation for TKD analyses

Sample NanoMilled at 500 eV; final thickness ~40 nm





Very fine Ni structure is revealed after NanoMilling

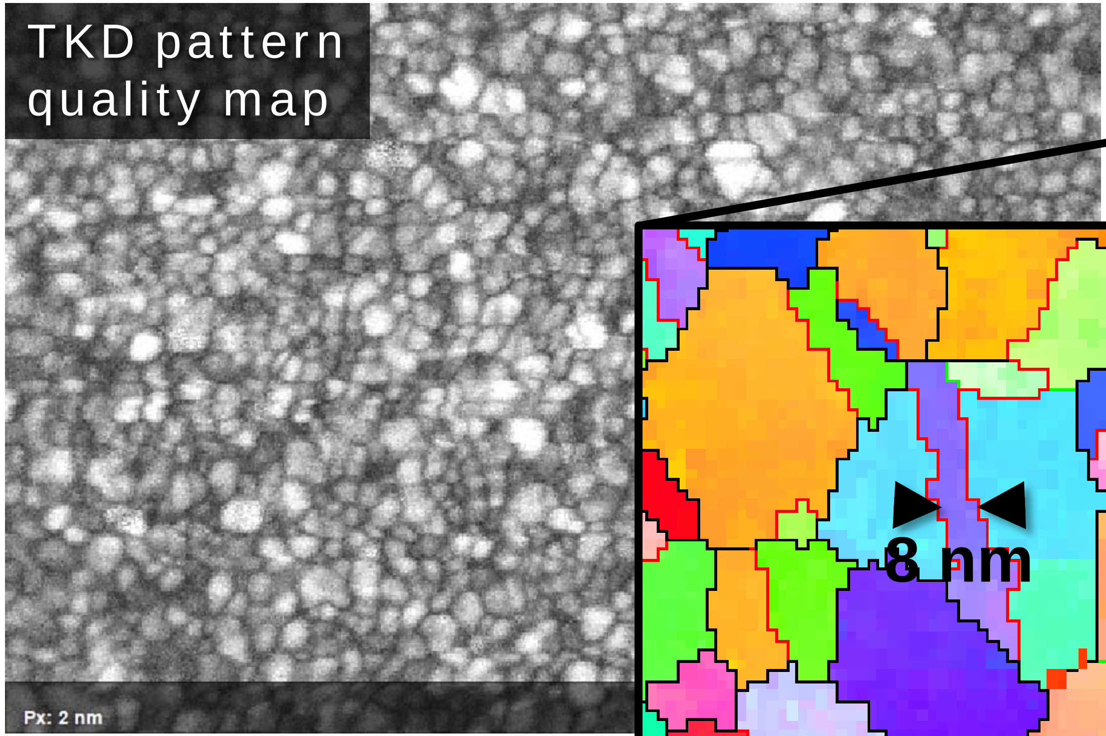
Acquisition conditions

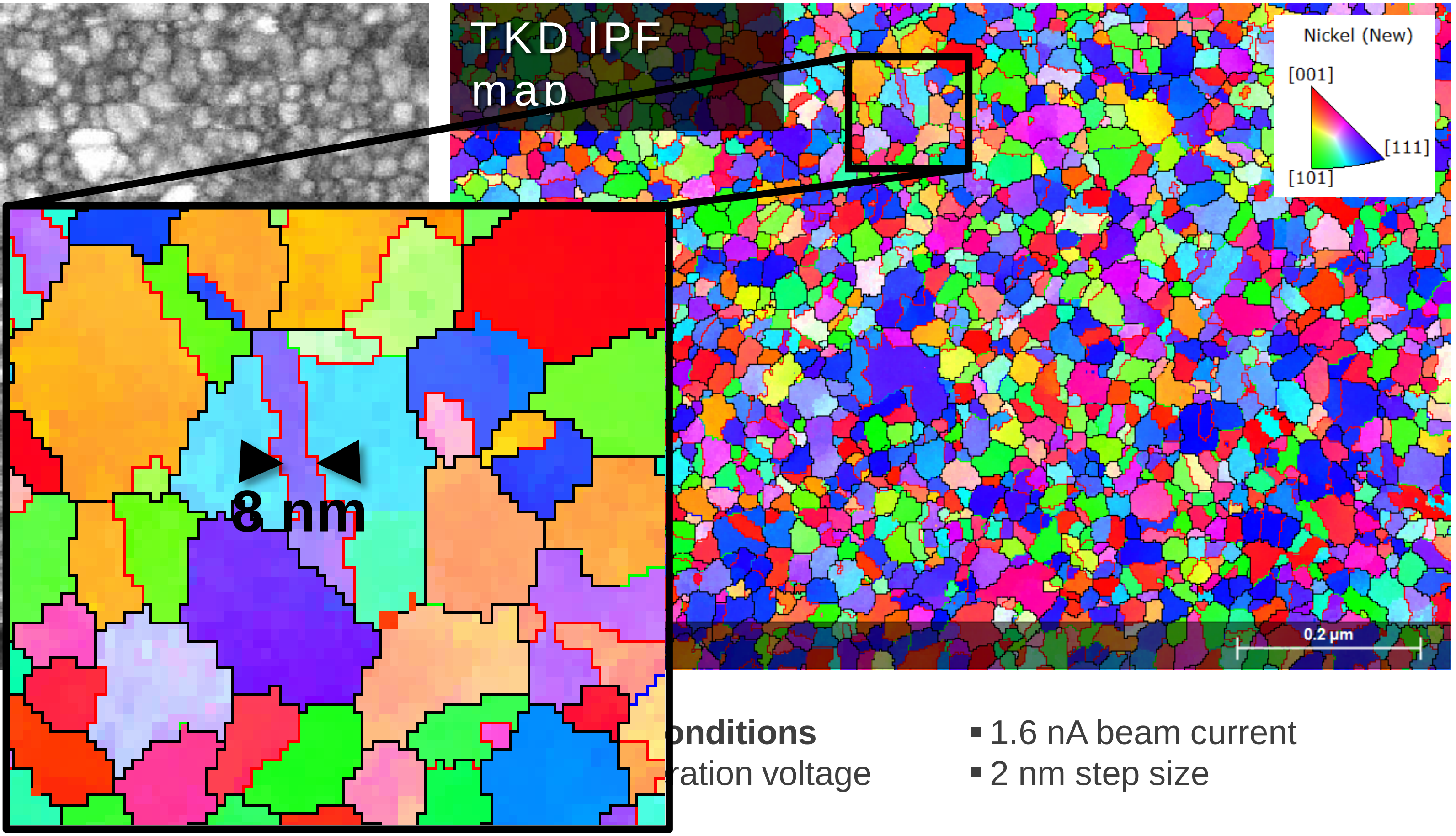
- 30 kV acceleration voltage
- 1.6 nA beam current
- 2 nm step size

Nanostructured Ni alloy

Optimal sample preparation for TKD analyses

Sample NanoMilled at 500 eV; final thickness ~40 nm





8 nm Σ3 <111>60° twin boundaries are observed

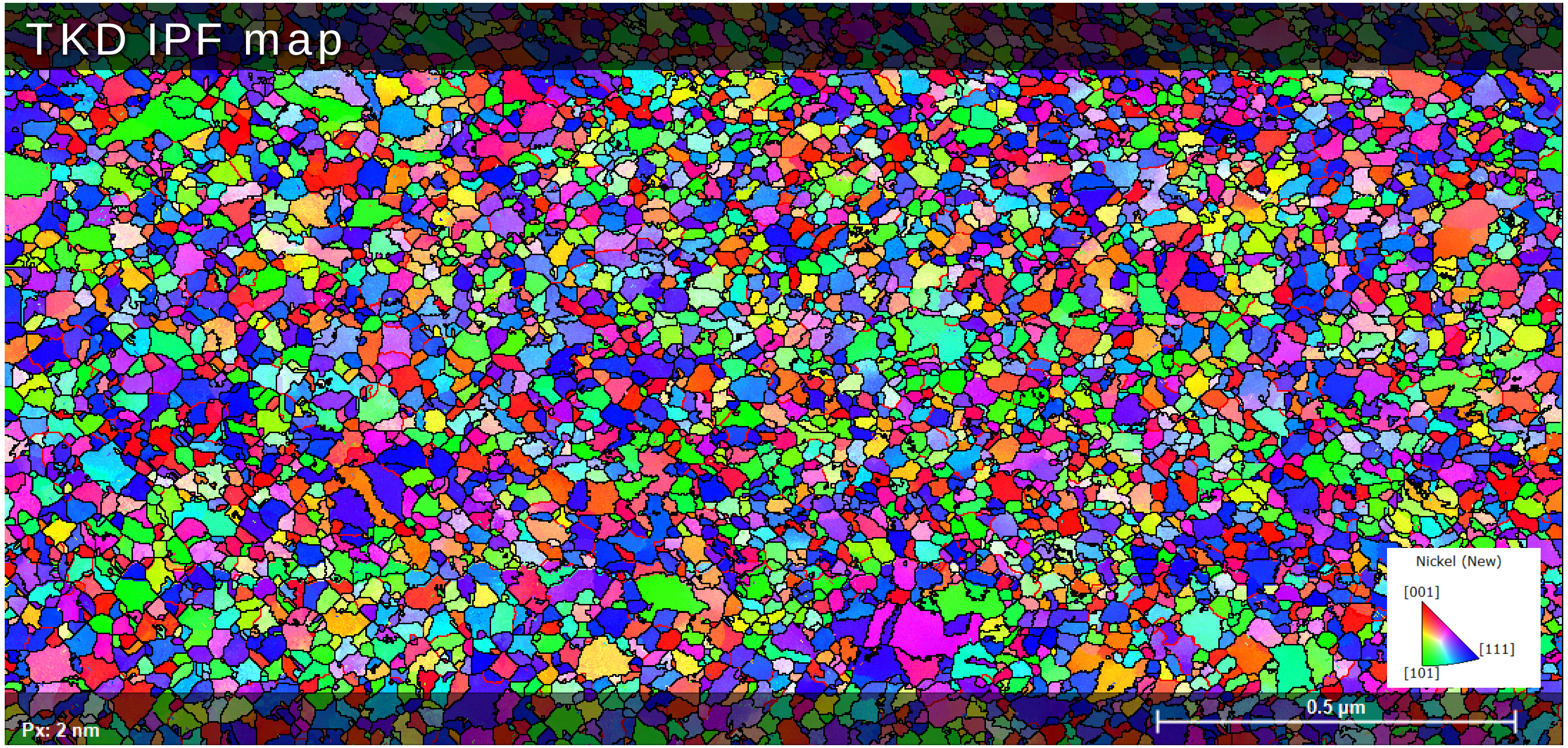
onditions
ration voltage

- 1.6 nA beam current
- 2 nm step size

Nanostructured Ni alloy

Sample NanoMilled at 500 eV; final thickness ~40 nm

The NanoMill system enables large area, electron-transparent sample preparation for accurate TKD analyses



- Average grain size: 30 nm
- 10% Σ3 <111>60° twin boundary

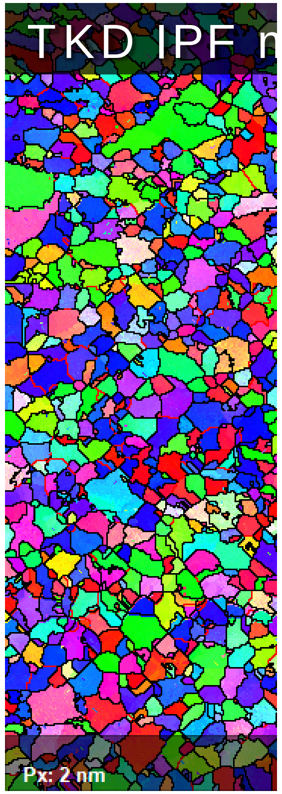
Acquisition conditions

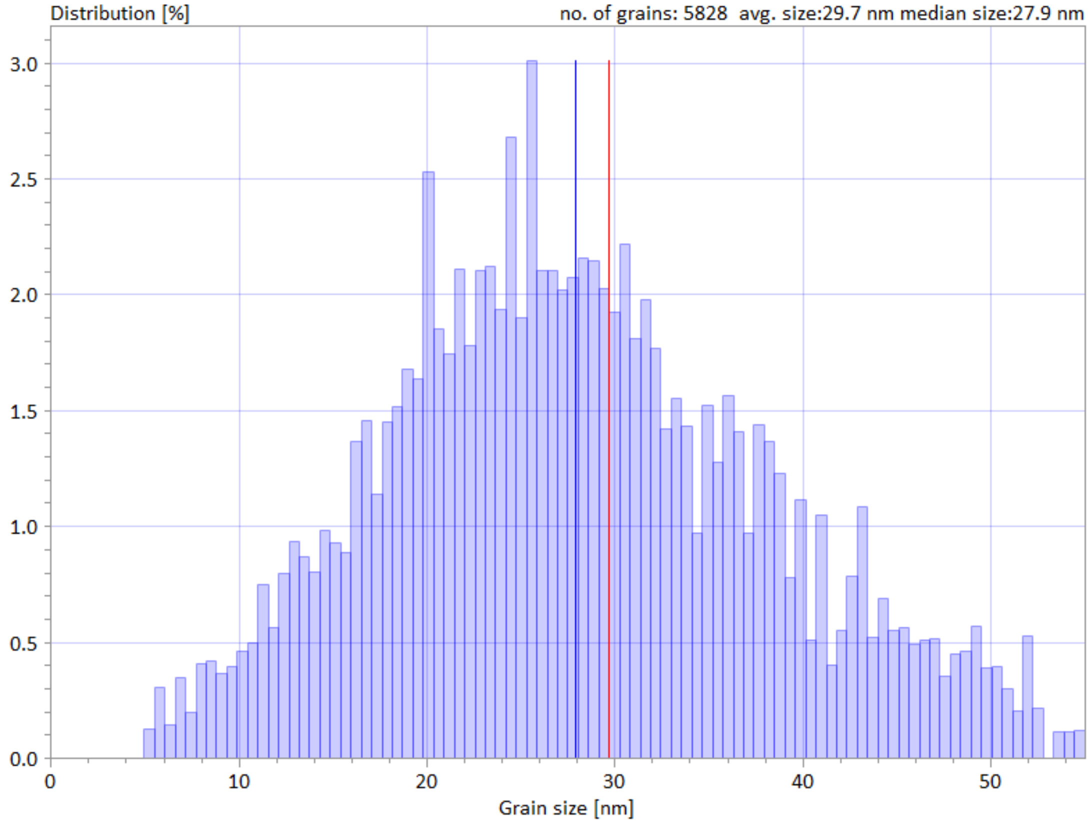
- 30 kV acceleration voltage
- 1.6 nA beam current
- 2 nm step size

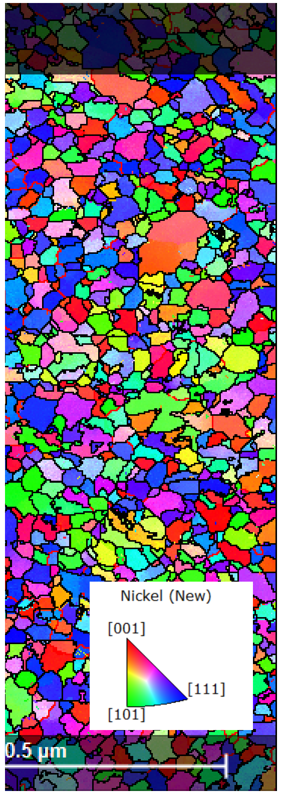
Nanostructured Ni alloy

Sample NanoMilled at 500 eV; final thickness ~40 nm

The NanoMill system enables large area, electron-transparent sample preparation for accurate TKD analyses







- Average grain size 30 nm calculated over 5828 grains
- 70% Σ3 <111>60° twin boundary

P. Nowakowski, et al.
Microsc. Microanal 29 (2023)

Conclusions

- A new approach for large area electron-transparent samples for TKD measurement was demonstrated
- Proposed workflow allows:
  - Significantly reduced FIB milling time
  - Removal of FIB-induced structural damage
  - Control of sample thickness
  - Improved overall TKD signal and indexing rate

Accurate sample preparation makes TKD analyses easier and provides improved results

E.A. Fischione Instruments, Inc.

9003 Corporate Circle, Export, PA 1563 USA
Tel **+1 724.325.5444** Fax **+1 724.325.5443**
www.fischione.com