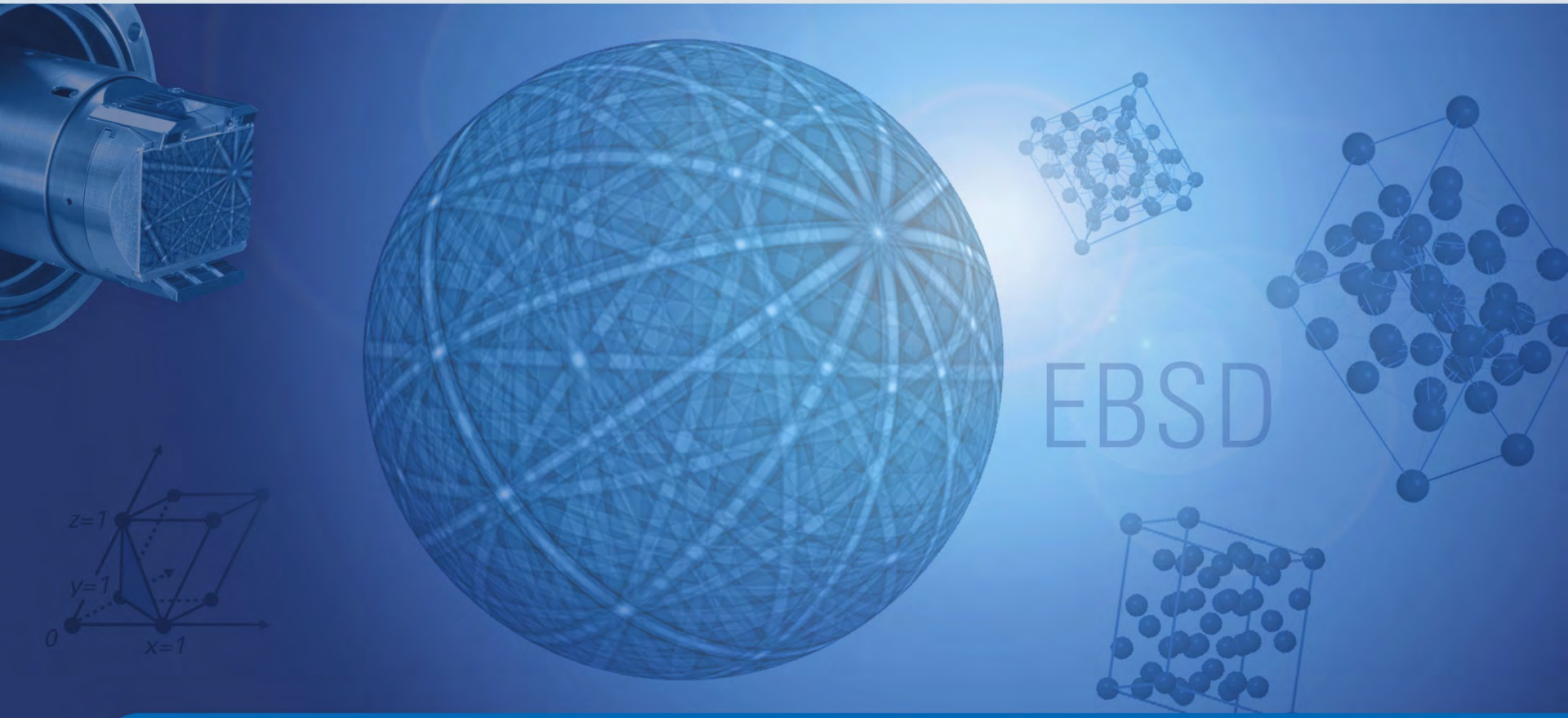
Quantitative Characterization of Nanostructured Materials with Fast TKD Measurement

Bruker Nano, Berlin



Presenters

- **Dr. Daniel Goran**

Product Manager EBSD
Bruker Nano Analytics, Berlin, Germany

- **Dr. Laurie Palasse**

Senior Application Scientist EBSD
Bruker Nano Analytics, Berlin, Germany

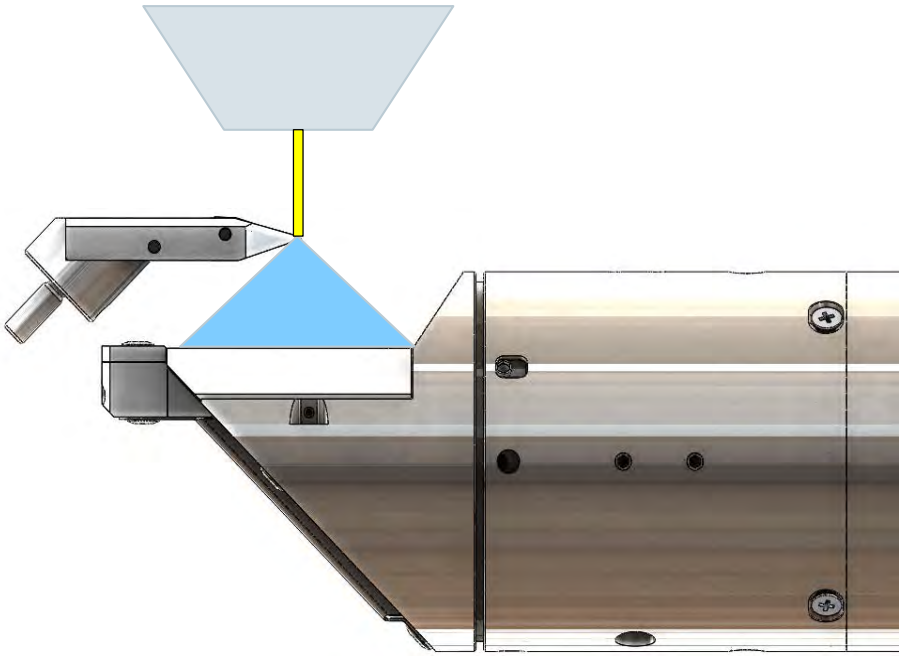
High speed TKD in SEM
outline

- Review of on-axis TKD geometry
- Application examples of fast-TKD: no compromise!
  - Speed
  - Spatial resolution
  - Indexing quality
  - Large area orientation mapping for statistics
- Combined EDS/TKD measurement

High speed TKD in SEM

Optimum sample-detector geometry

Schematic view



Strong signal

- Fast acquisition speeds
  - High efficiency
  - measurements less prone to beam instability
- low probe currents
  - Higher resolution
  - Low carbon contamination
- No pattern distortions
  - Pattern Center is in the middle ⇔ almost no gnomonic projection induced distortions ⇔ better band detection and indexing quality

* LEM3, Metz, France "*Orientation mapping by transmission-SEM with an on-axis detector*", J.-J Fundenberger et all., Ultramicroscopy, 161, 17–22, 2016

High speed TKD in SEM

Optimum sample-detector geometry

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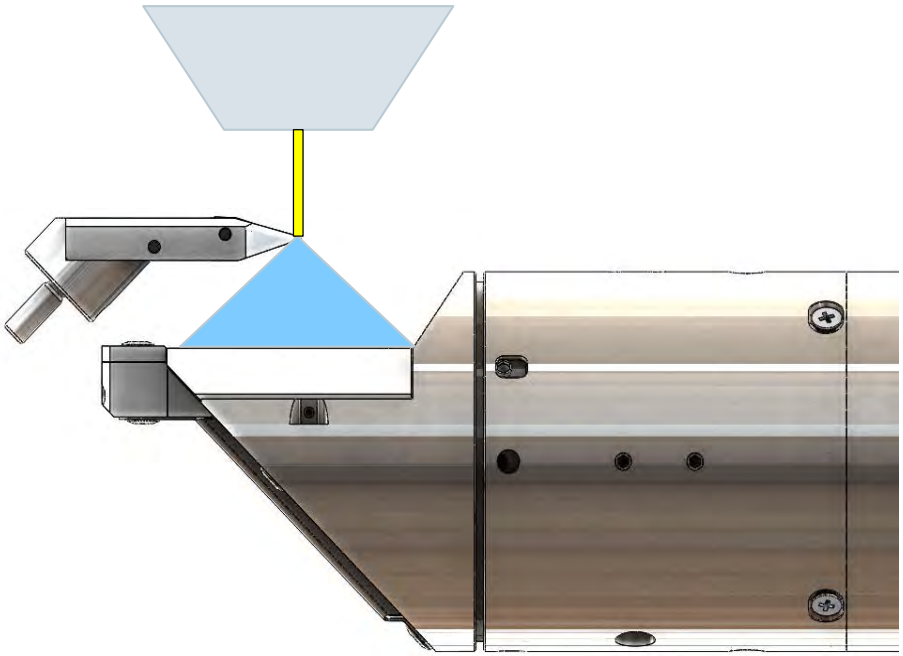

Strong signal

- Fast acquisition speeds
  - High efficiency
  - measurements less prone to beam instability
- low probe currents
  - Higher resolution
  - Low carbon contamination
- Flexibility in DD values
  - active area
  - Adjustment depending on atomic weight/thickness

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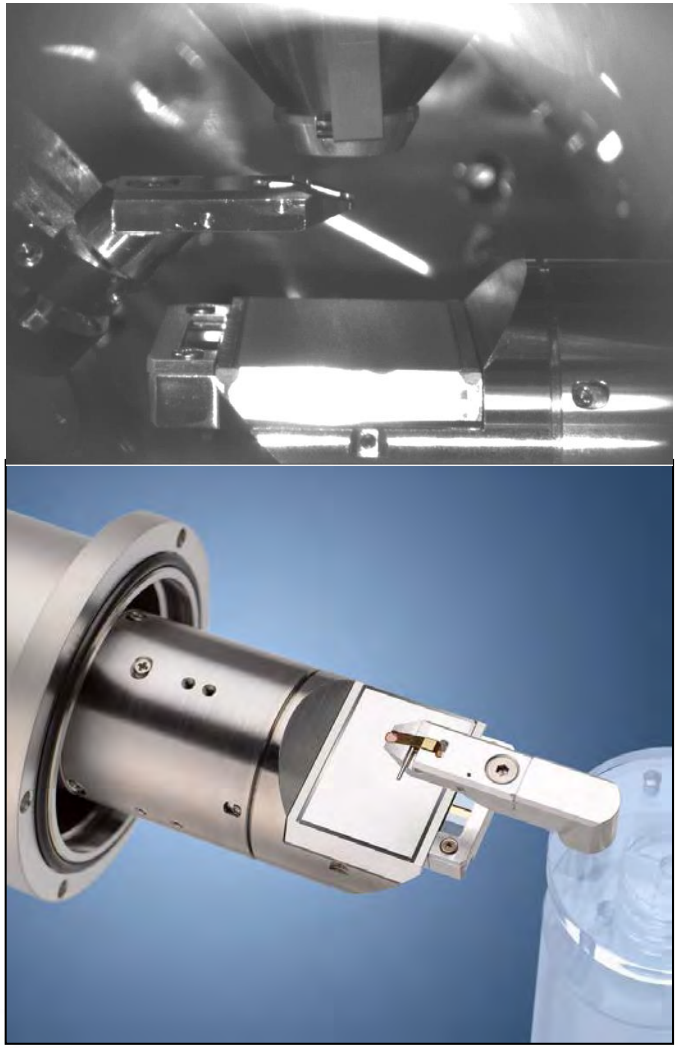
High speed TKD in SEM

Optimum sample-detector geometry



OPTIMUS Main features:

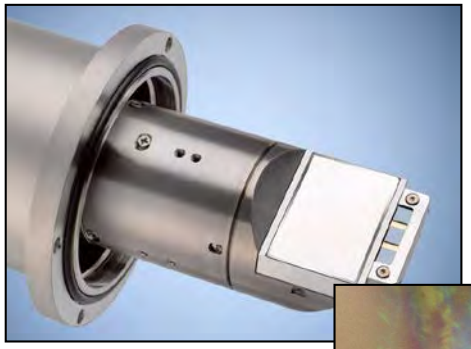
- Horizontal phosphor screen – for capturing the signal around SEM's optical axis
- DF/BF imaging (3x Si diodes)
- Easy to use/switch between EBSD and TKD modes
- Compatible with all existing *e*-Flash detectors

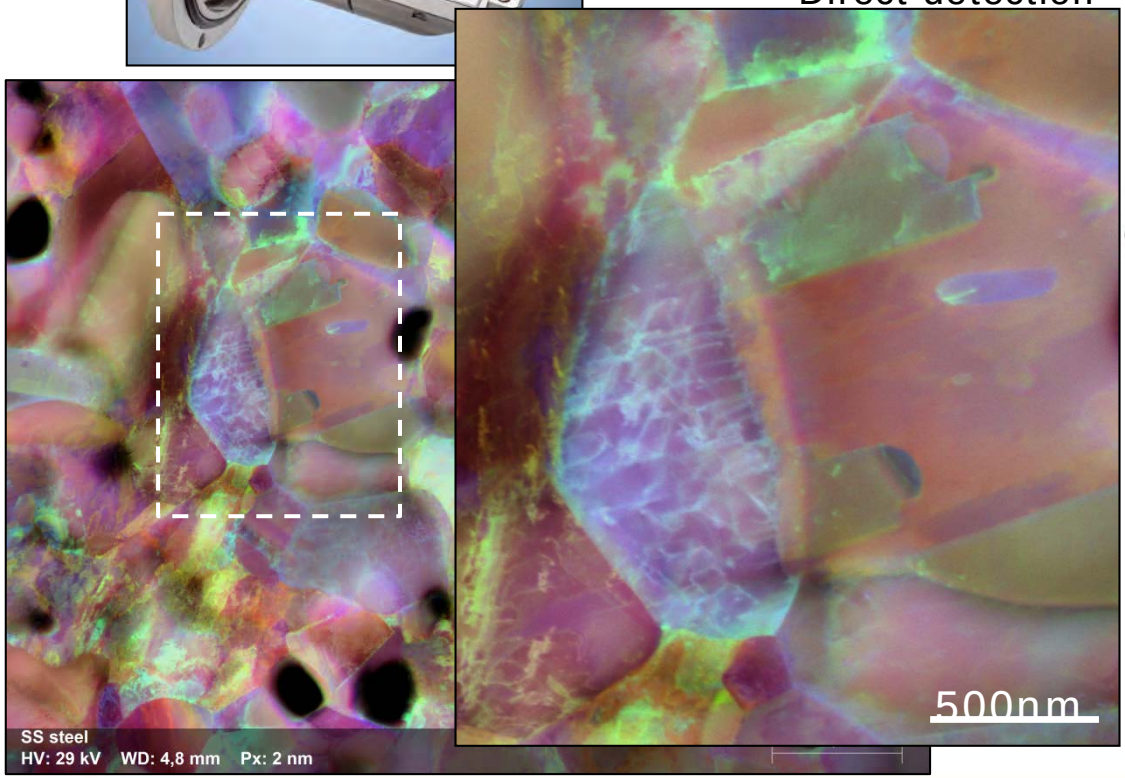
TKD sample holder

Patent EP 2824448A1

High speed TKD in SEM

Integrated ARGUS™ imaging system



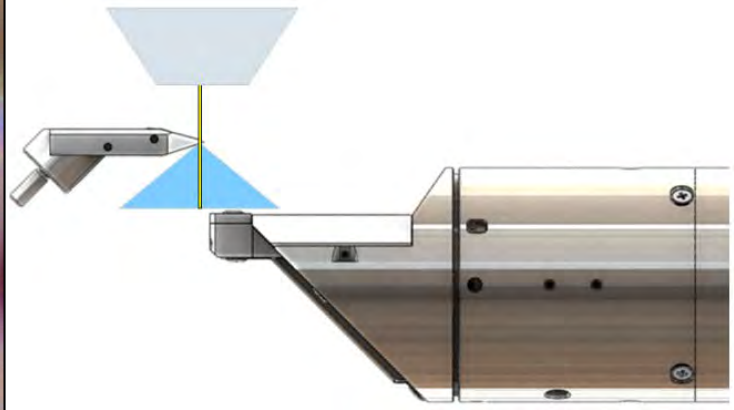


Feature:

- Built-in ARGUS™ imaging system (3x Si diodes)

Benefits:

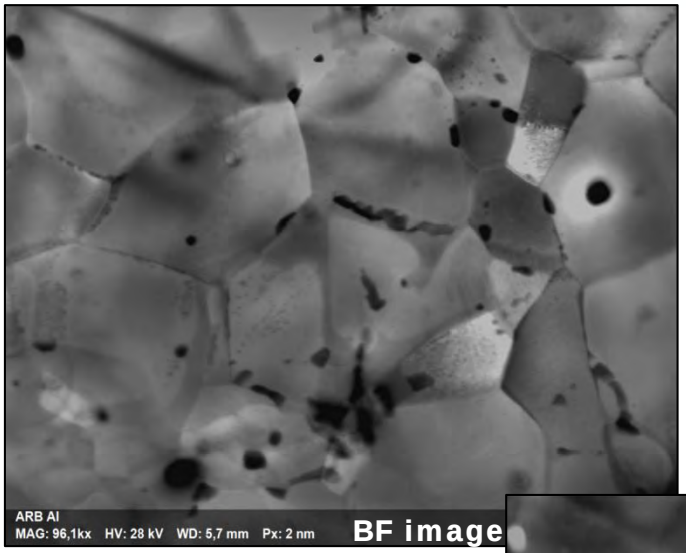
- Color coded Dark Field imaging
- Direct detection – fast & sensitive (up to 125,000 p/s)



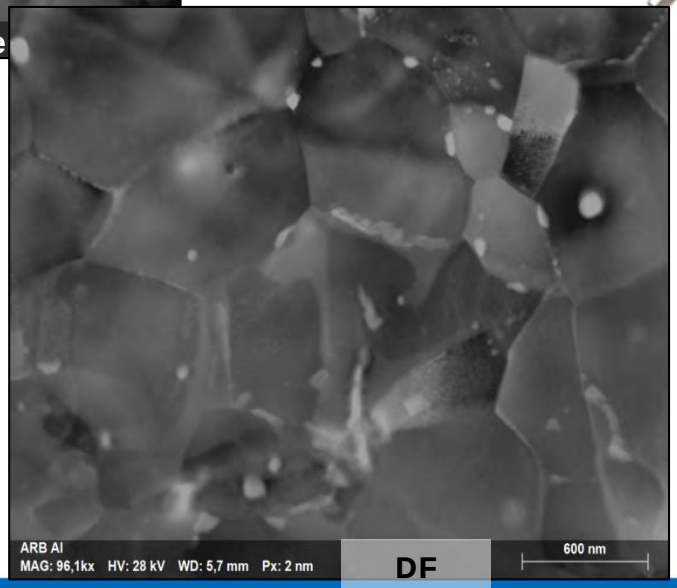
Thanks to Prof. Zeng Yi from Shanghai Institute of Ceramics in China for generously providing the sample

TKD in SEM using on-axis detector

Integrated ARGUS™ imaging system



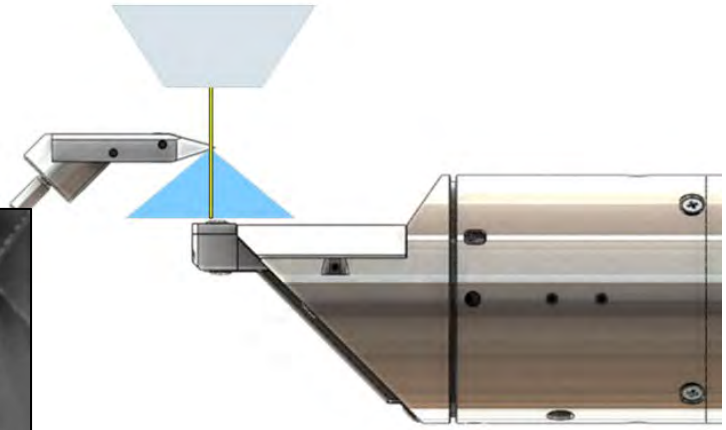
BF image



DF image

Benefits:

- Built-in BF/DF imaging system (3x Si diodes)
- Bright field imaging (middle diode)
- Dark field imaging (side diodes)



TKD in SEM using on-axis detector

Integrated ARGUS™ imaging system

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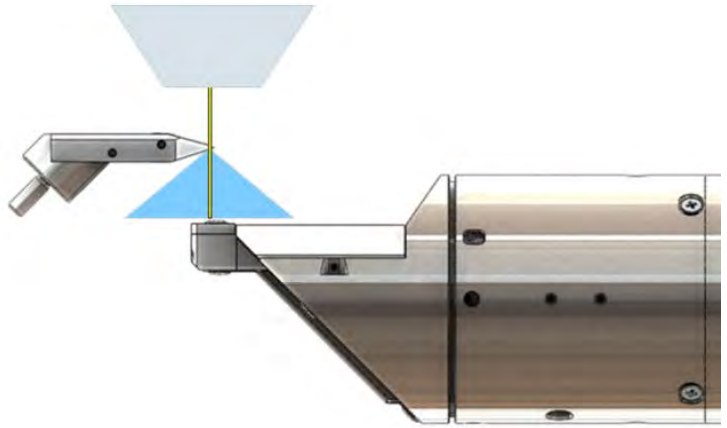
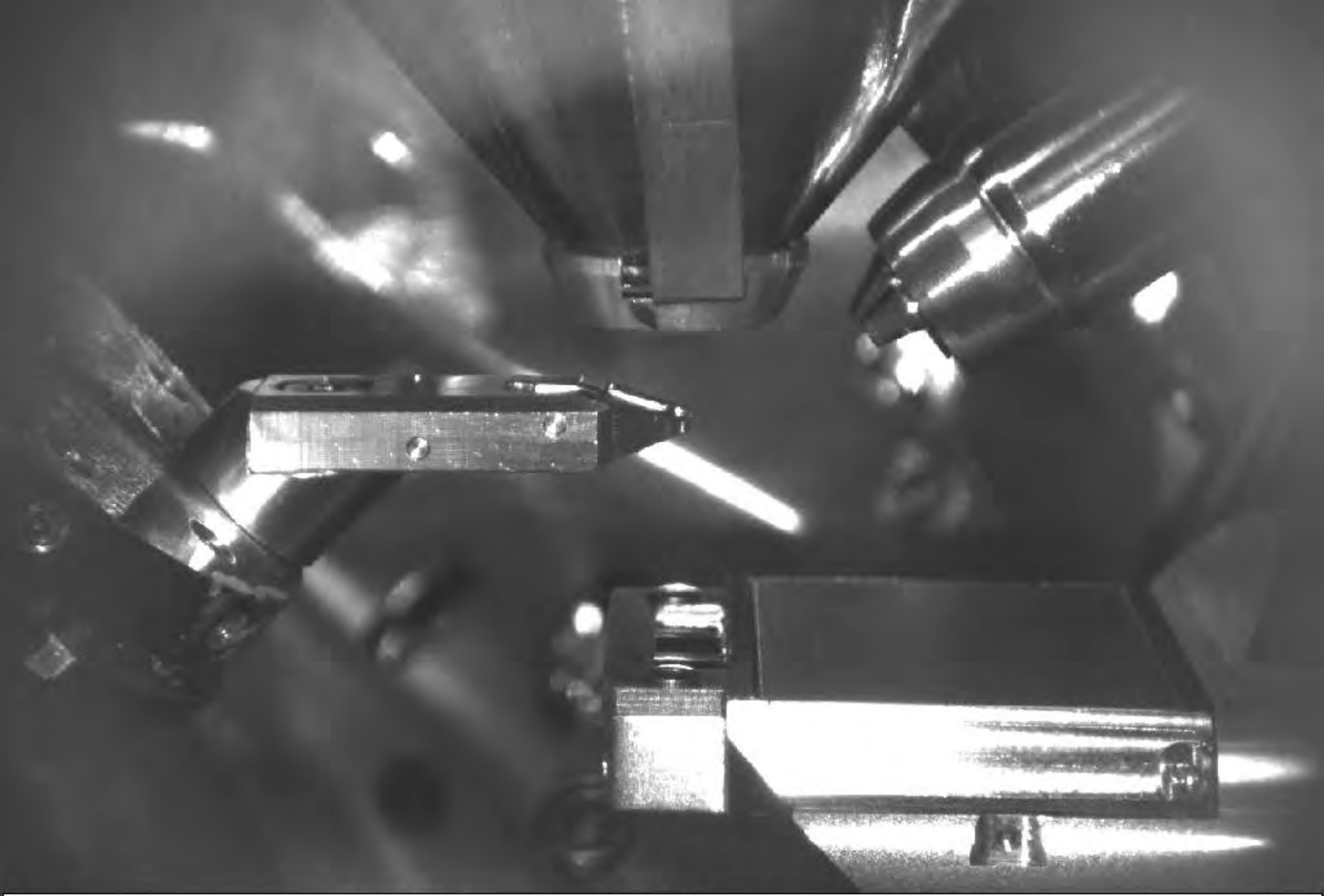
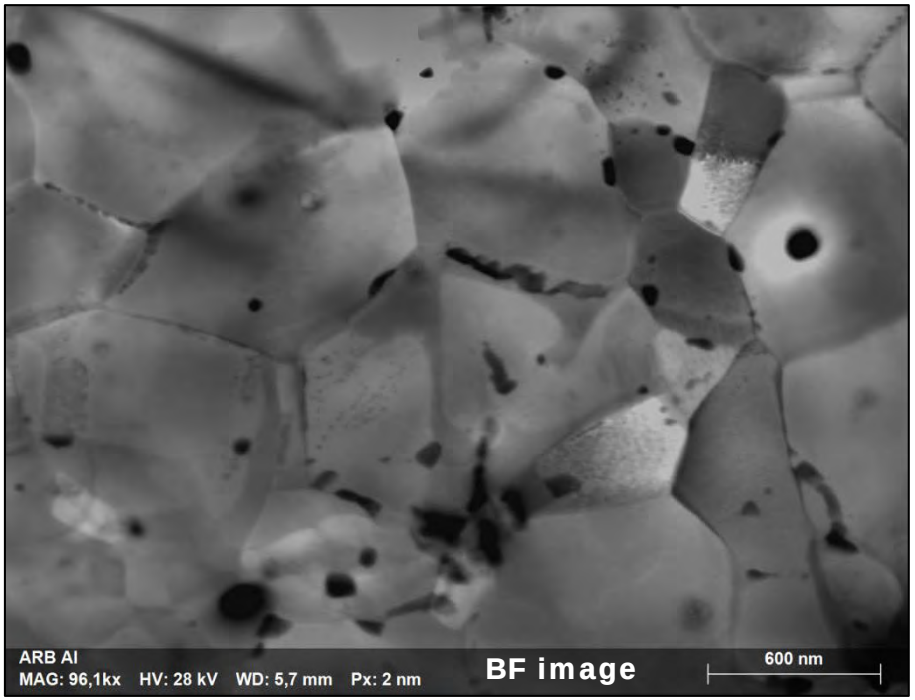



1 µm
EHT = 29.00 kV
WD = 7.1 mm
Signal A = USB TV2
Mag = 41.00 K X
Date :3 Dec 2015
Time :12:02:38
ZEISS

TKD in SEM using on-axis detector

Integrated ARGUS™ imaging system



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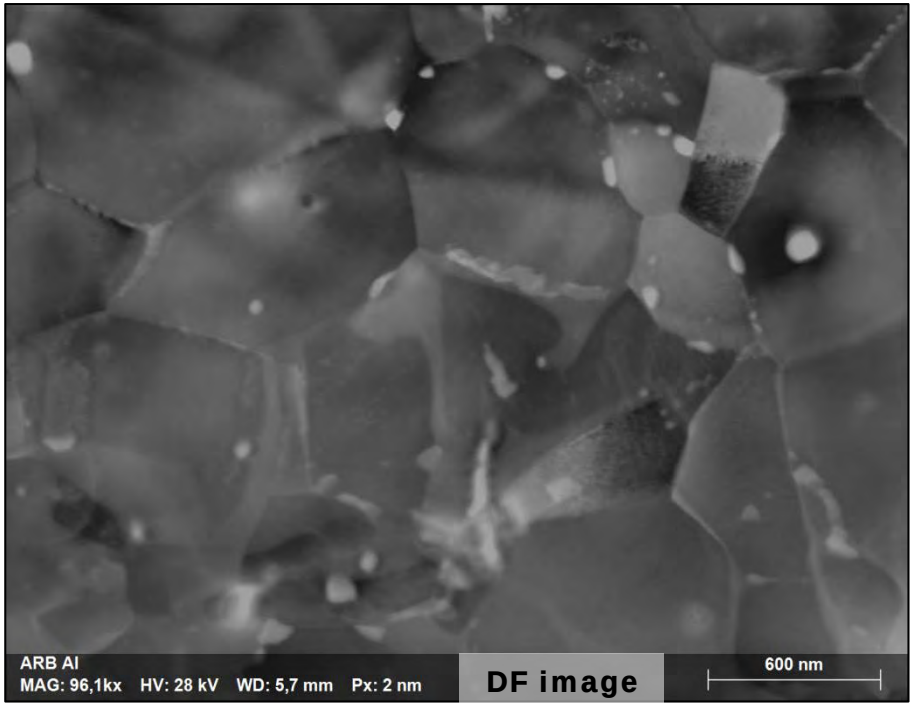


High speed TKD in SEM

Setup

- *e*-Flash^{FS} detector
- OPTIMUS™ TKD head
- Bruker TKD sample holder

Esprit 2.1 : all-in-one software

SW for all analytical techniques: WDS, µXRF, EDS, EBSD, TKD, …

For measurement and post-processing

High speed TKD in SEM

Application examples

large-area orientation mapping at nanoscale

High speed TKD in SEM

high speed measurement, moderate probe current

Partially recrystallized stainless steel

Indexed points: 85.6%

Pattern resolution:

160x120

Speed: **623fps**

Probe current : **7nA**

Map size:

1990x1492pixels

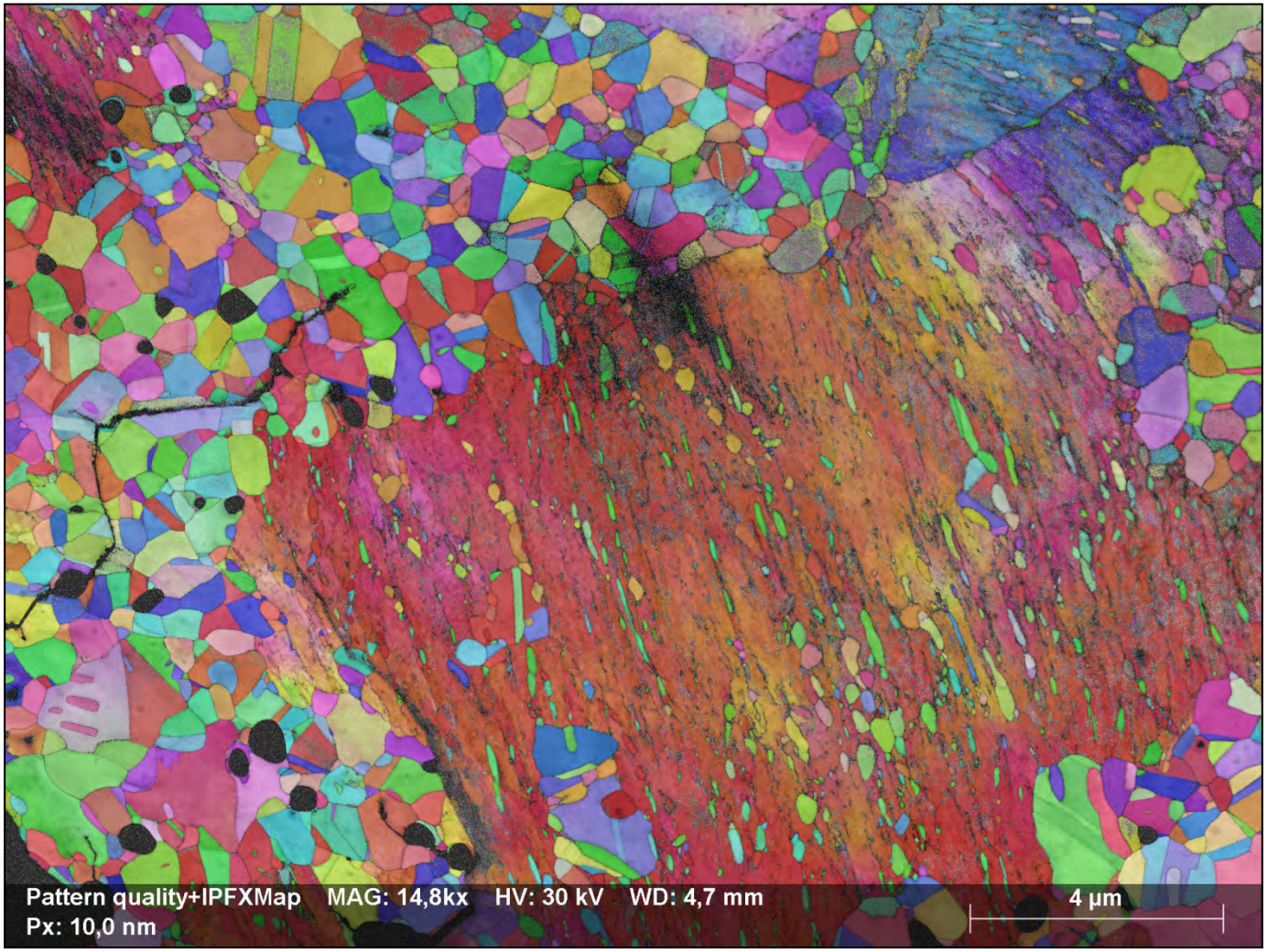
Measurement time:

1:19:30h

Step size: 10nm

High speed TKD in SEM

high speed measurement, moderate probe current



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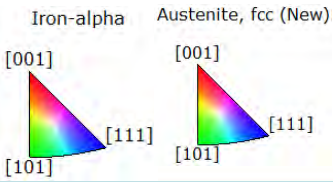
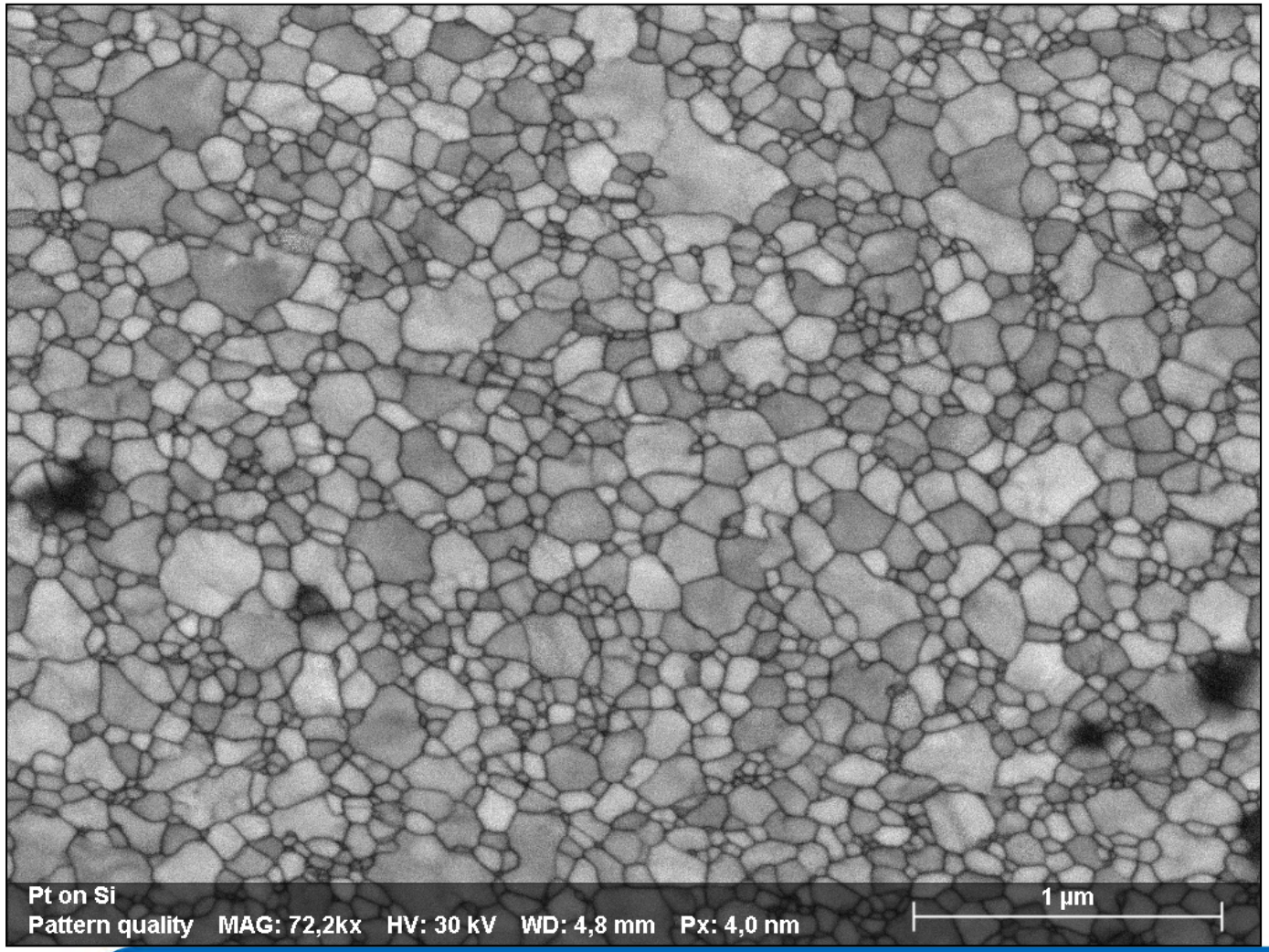


High speed TKD in SEM

higher spatial resolution, low probe current & speed



Pt thin film on Si

Indexed points: 93.1%

Pattern resolution:

160x120pixels

Speed: **328fps**

Probe current :1,8 nA

Map size:

1017x763pixels

Measurement time:

39:27min

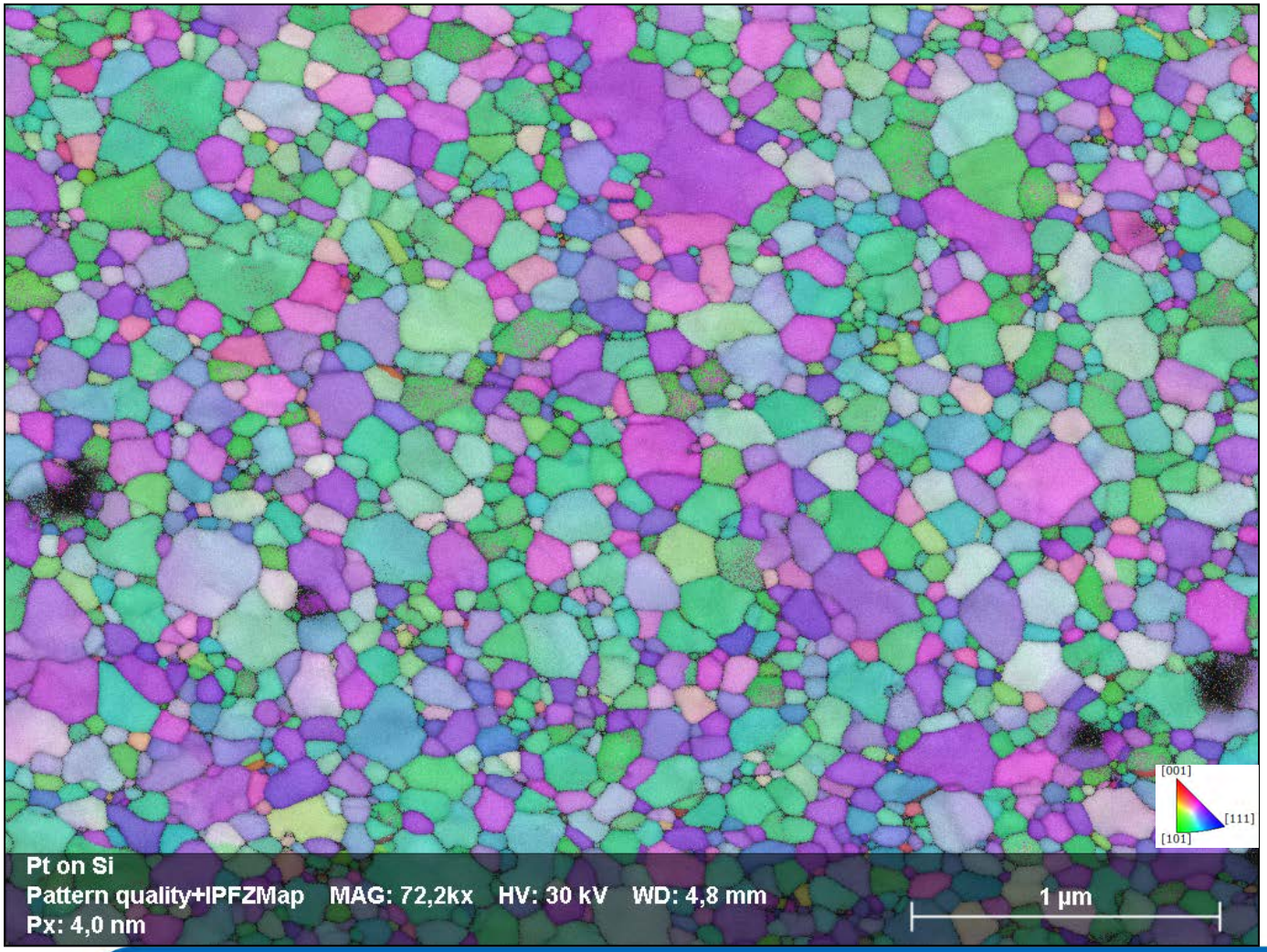
1260 grains

Step size: 4nm

Sample preparation: Wolfgang Grünewald, Leica, Germany.

High speed TKD in SEM

higher spatial resolution, low probe current & speed



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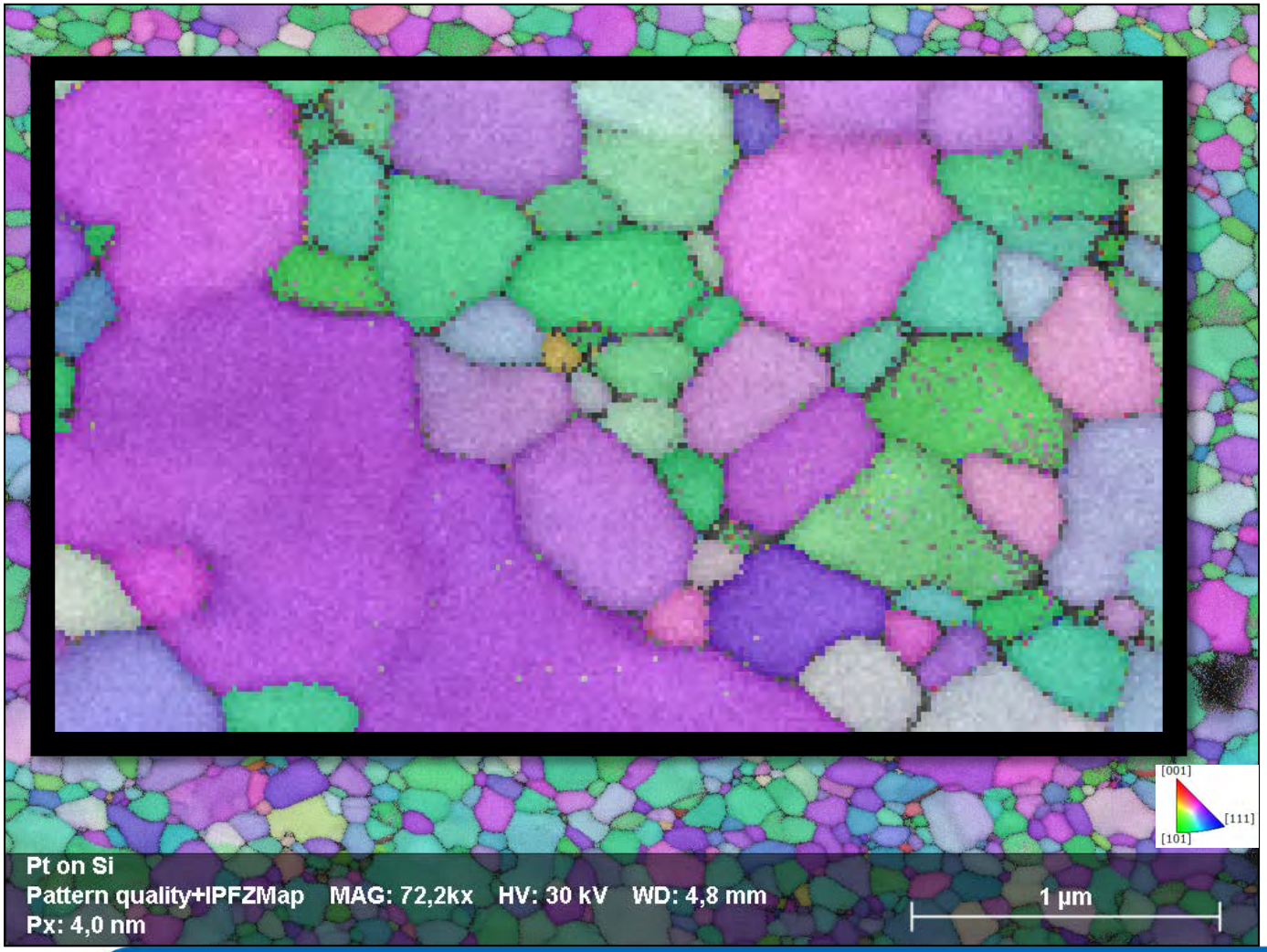

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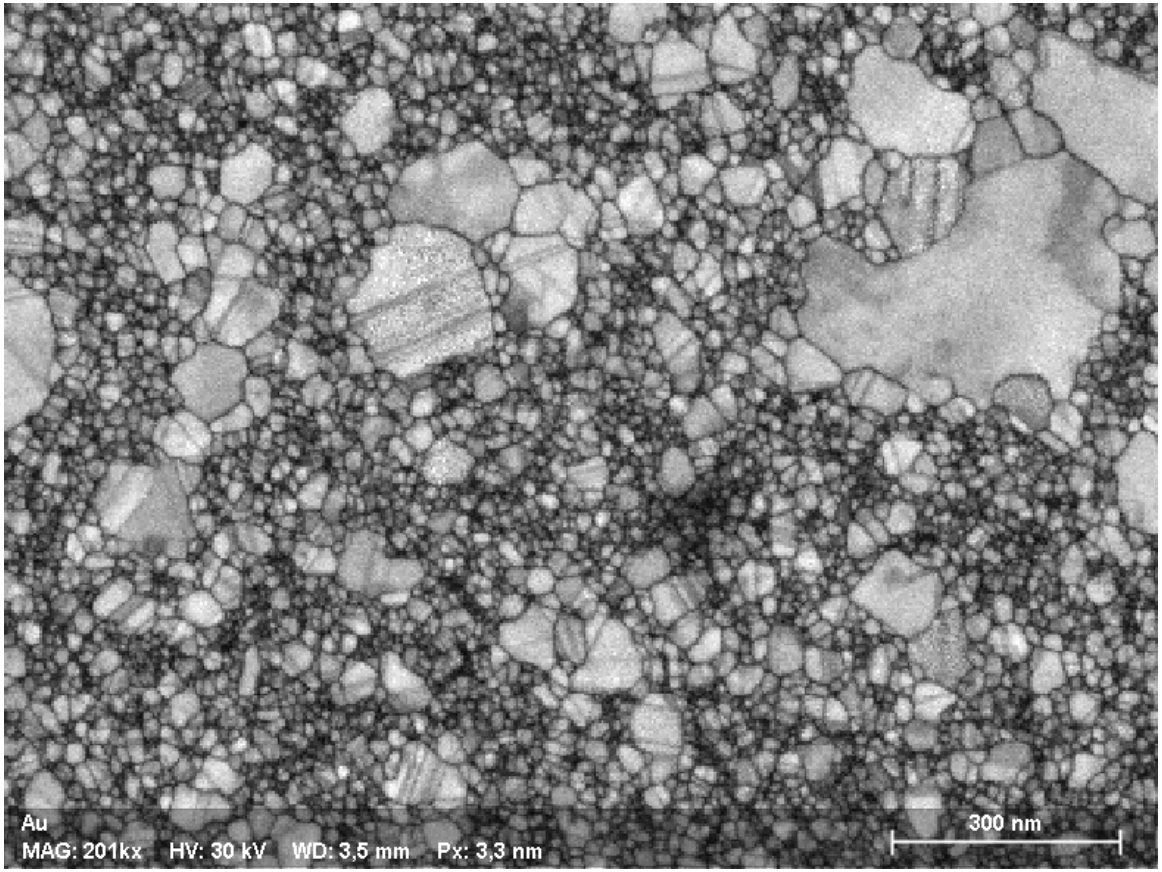
High speed TKD in SEM

e-Flash^{FS} and OPTIMUS – resolution & speed

20nm Au film on 5nm Si_3N_4 membrane



Step size: 3.3nm

Acq. speed: 316 fps

Probe current : 1,8 nA

Total acquisition time: 08:22 min

Map size: 158,700 pixels

Pattern resolution: 160x120 pixels

Zero sol.: 12.5%

2662 grains

High speed TKD in SEM

e-Flash^{FS} and OPTIMUS – resolution & speed

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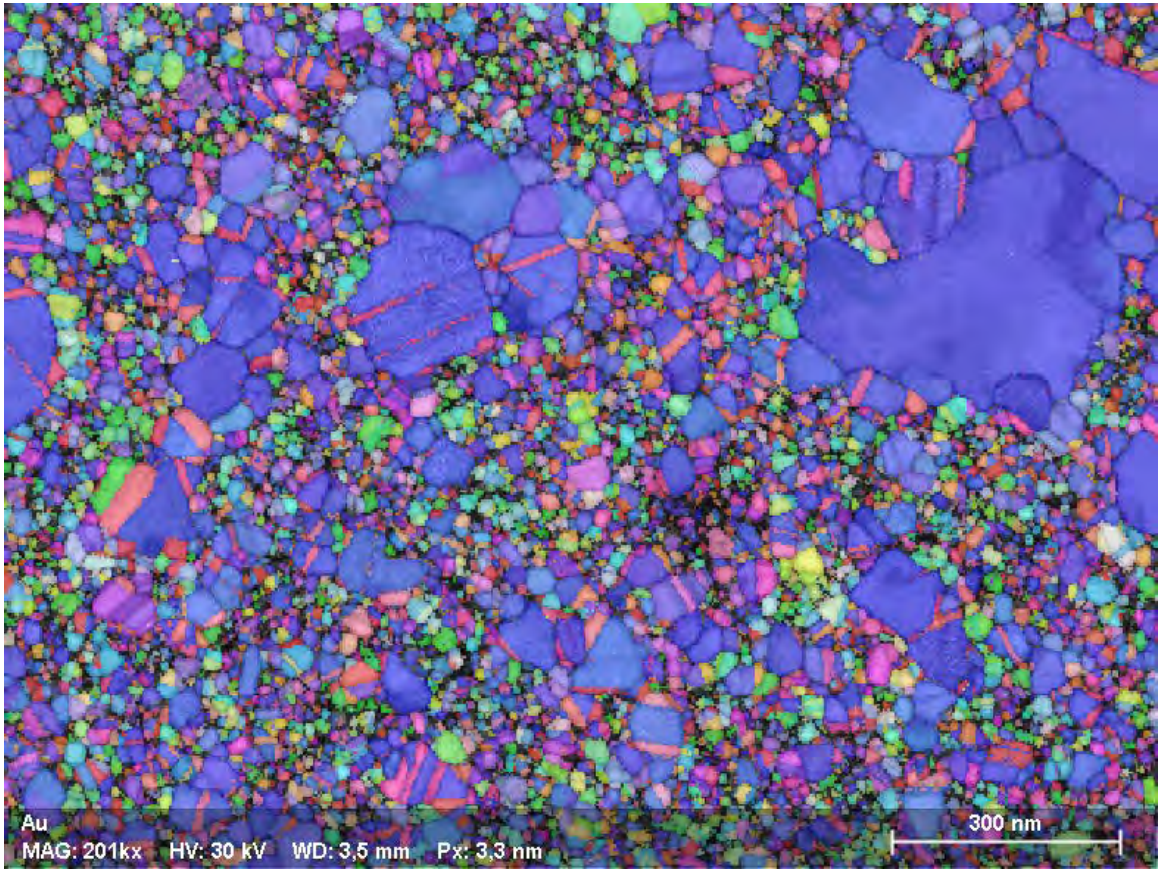

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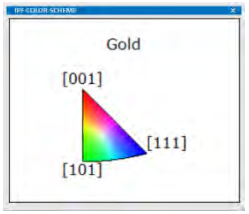

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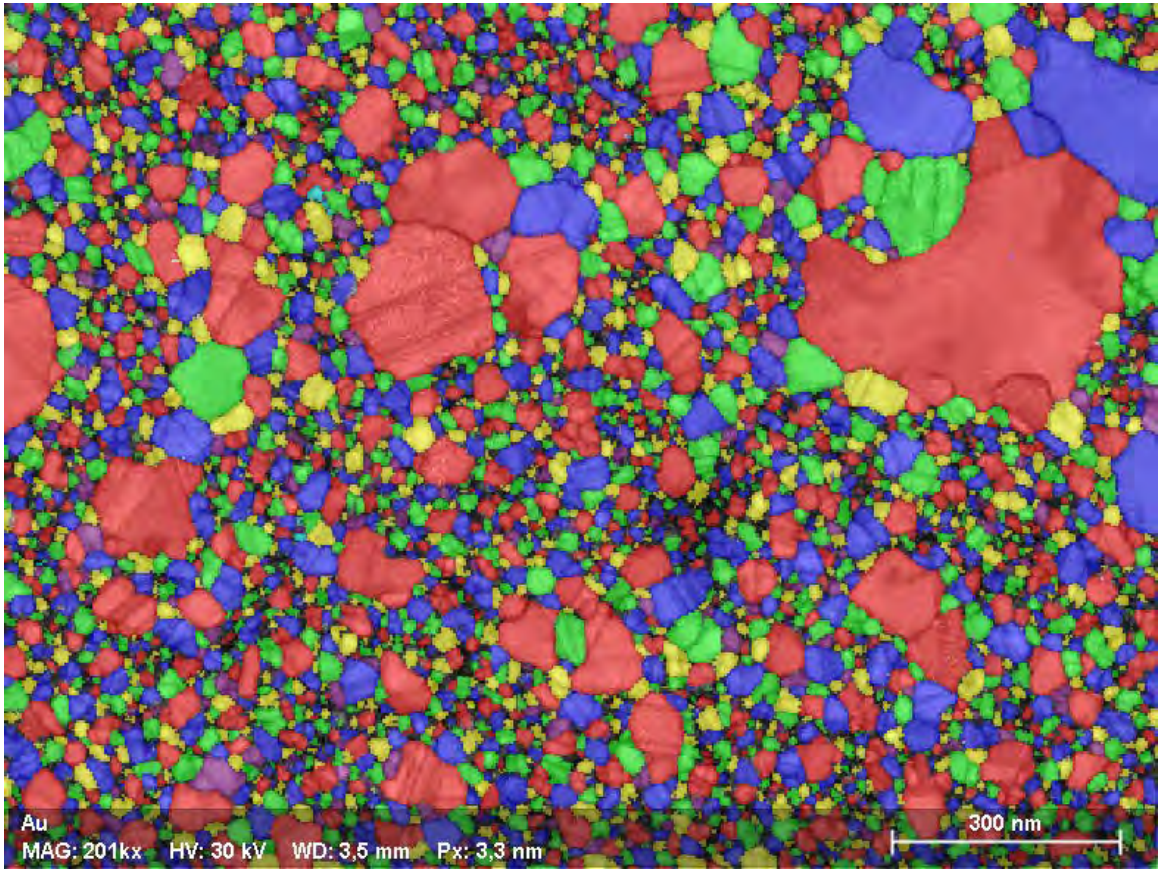

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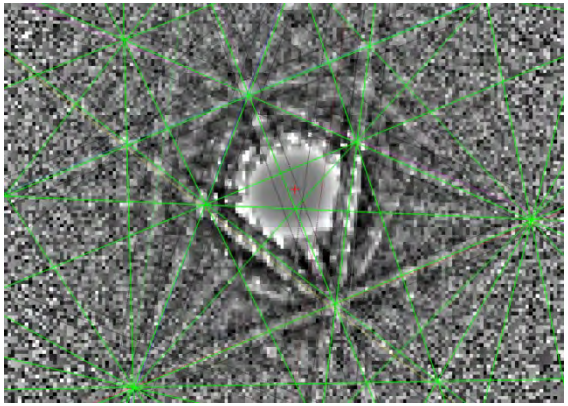

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e-Flash^{FS} and OPTIMUS – resolution & speed

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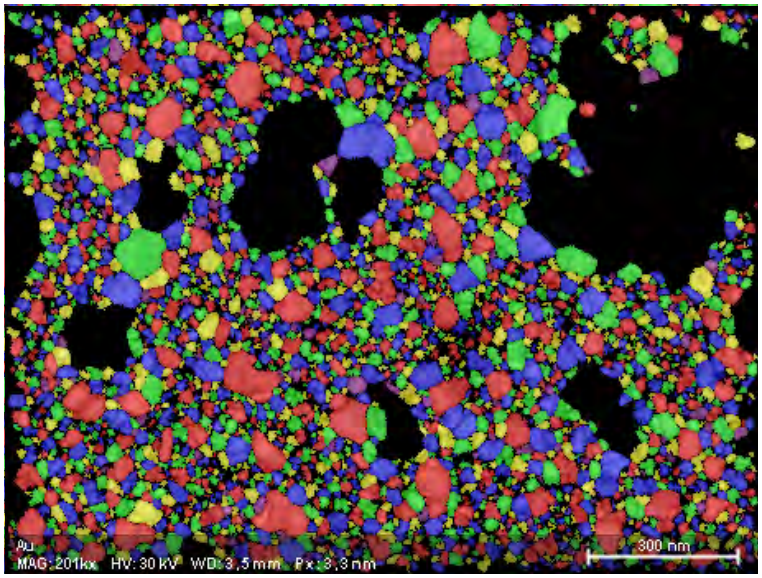
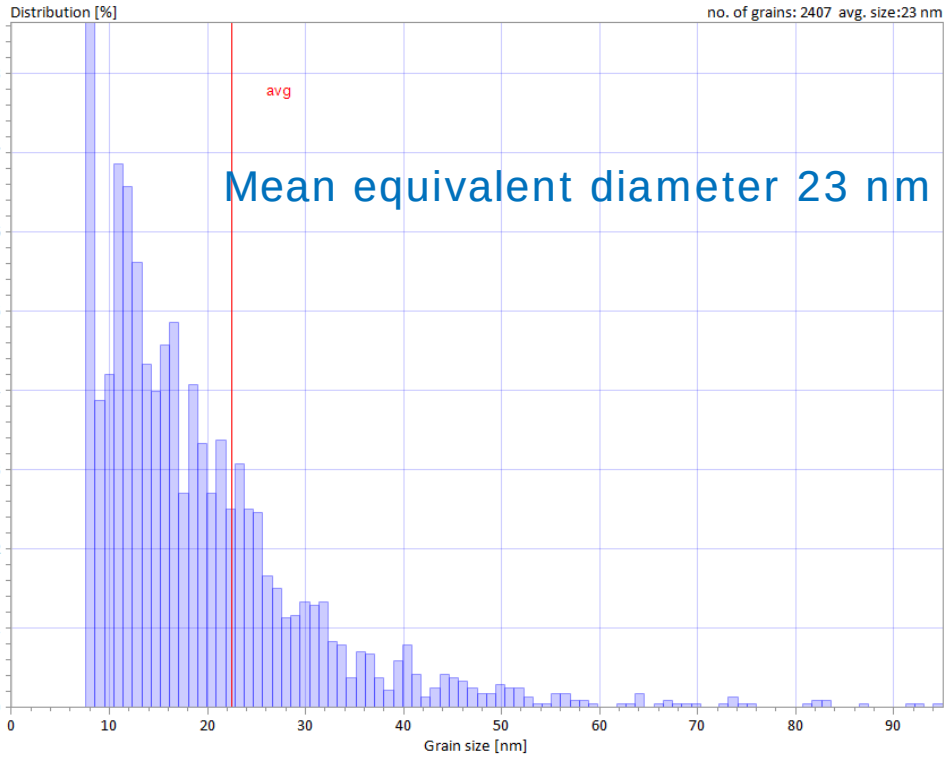
2407 grains <100nm grain size



grain size distribution not weighted by area fraction :



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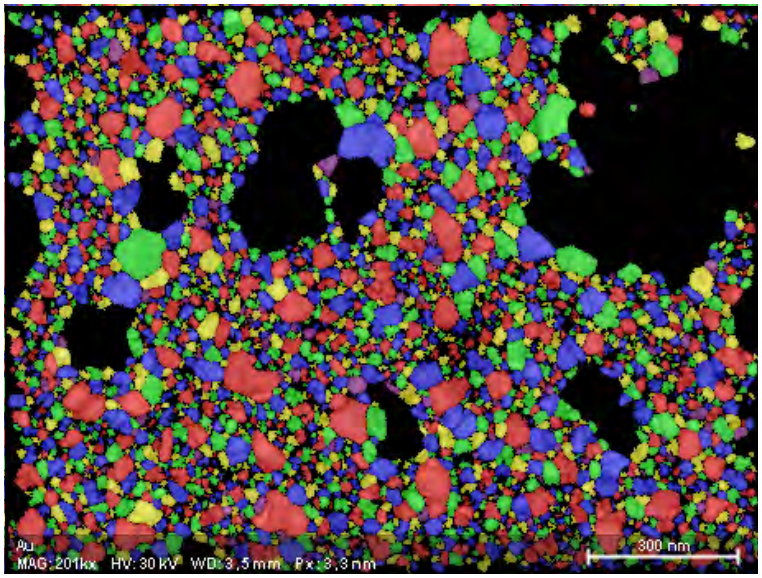

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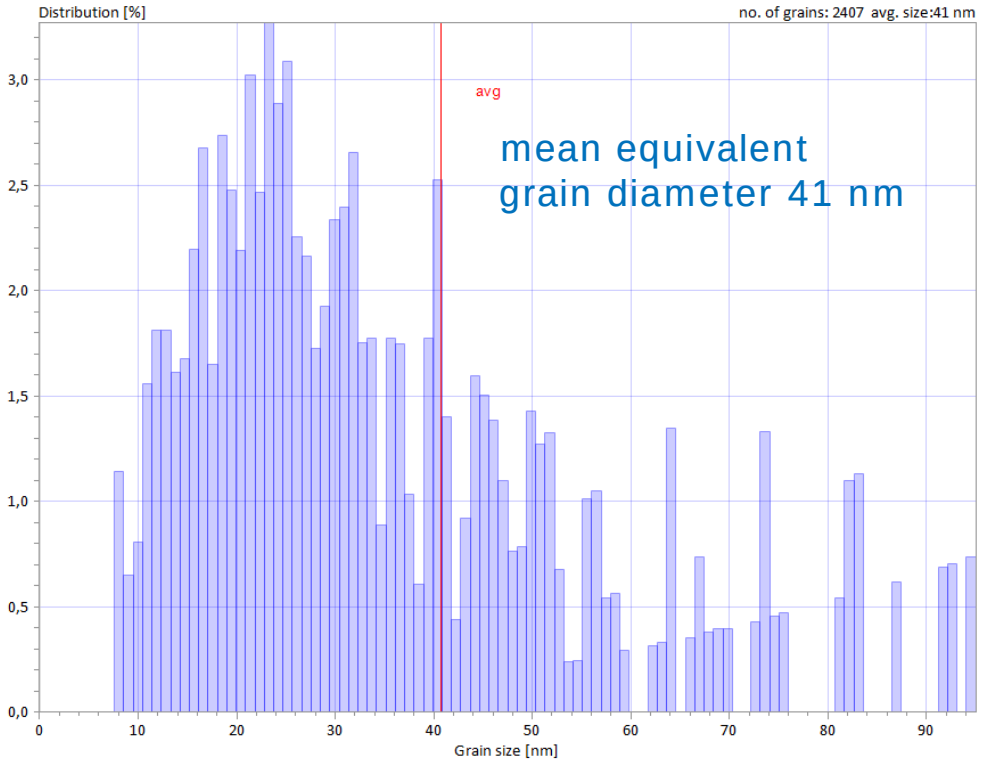
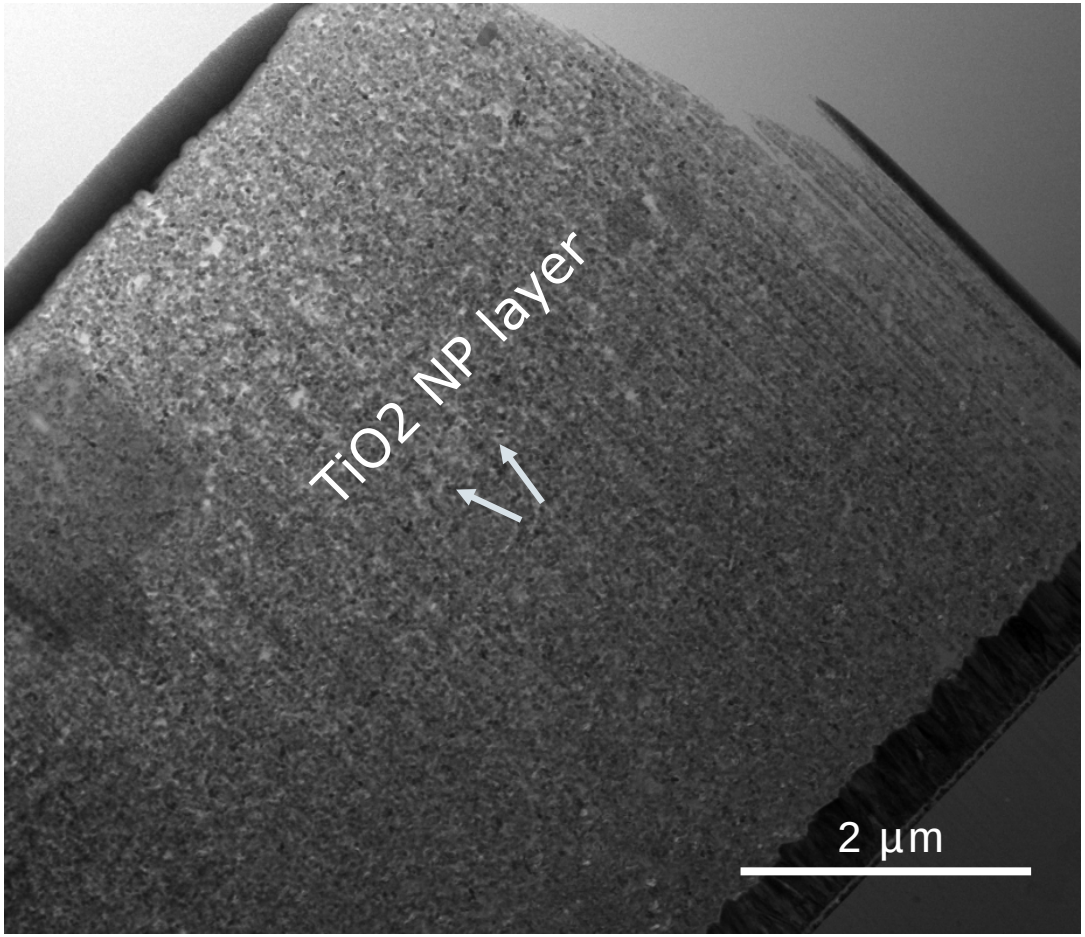
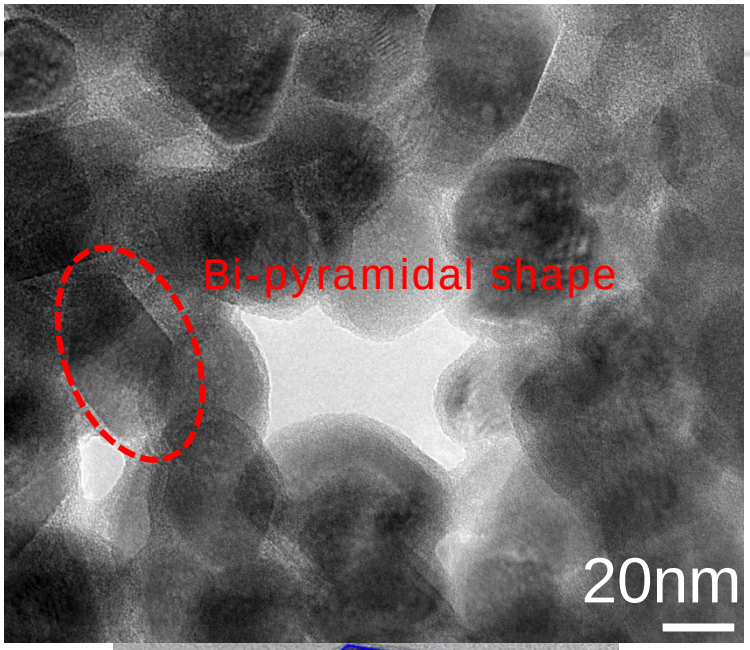


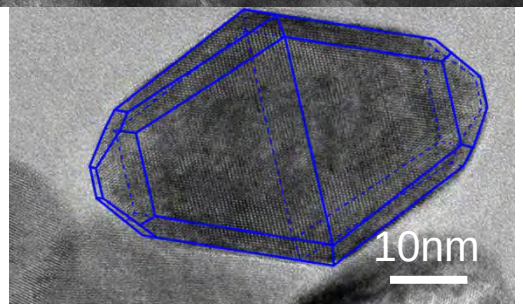
High speed TKD in SEM

characterisation of porous TiO_2 layer (DSSC)

SETNanoMetro project - Dye sensitized solar cells (DSSC)







* In collaboration with Nicole Wollschläger BAM, Berlin.
Sample preparation:FIB then Nanomill (Fischione)

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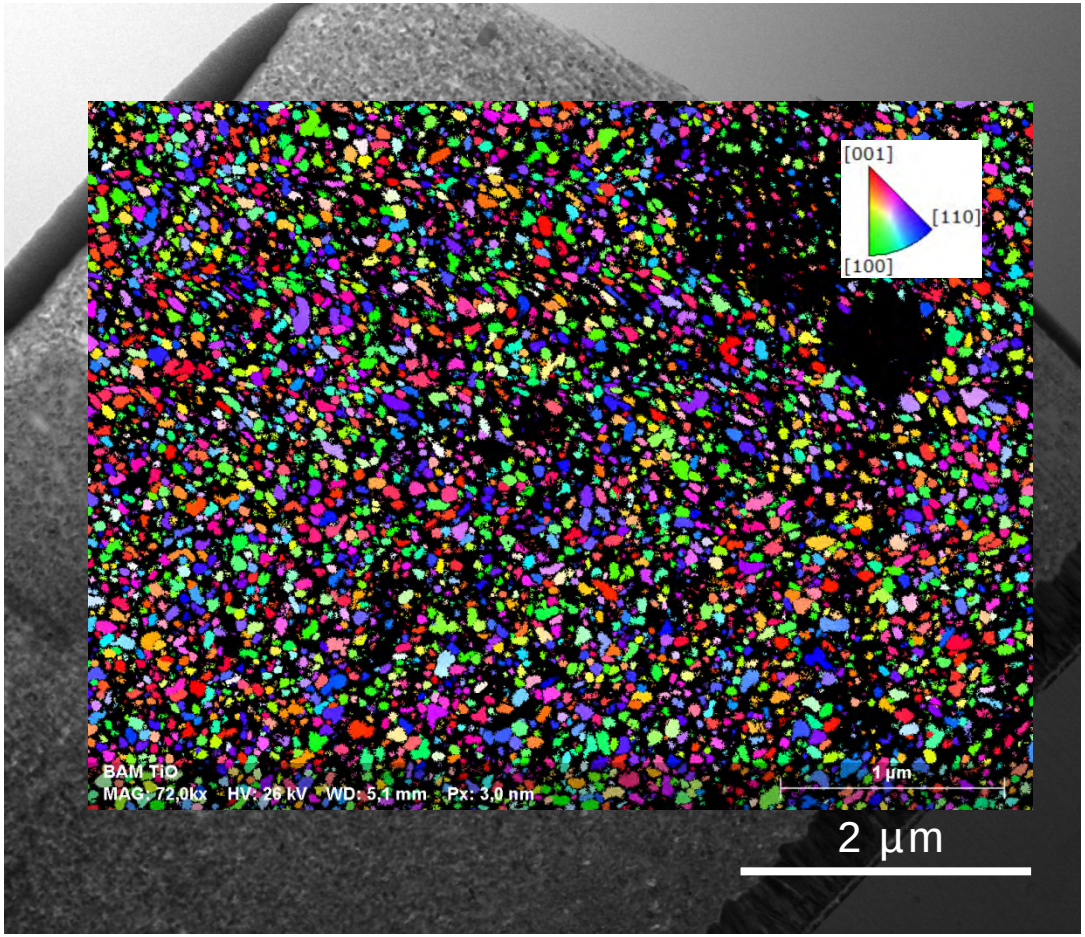


Indexed points: 58.1% (incl. pores)

Pattern resolution:

320x240 pixels

Speed: **243 fps**

Probe current : 7 nA

Map size: 1406*1055 pixels (~1,5M)

>6600 grains

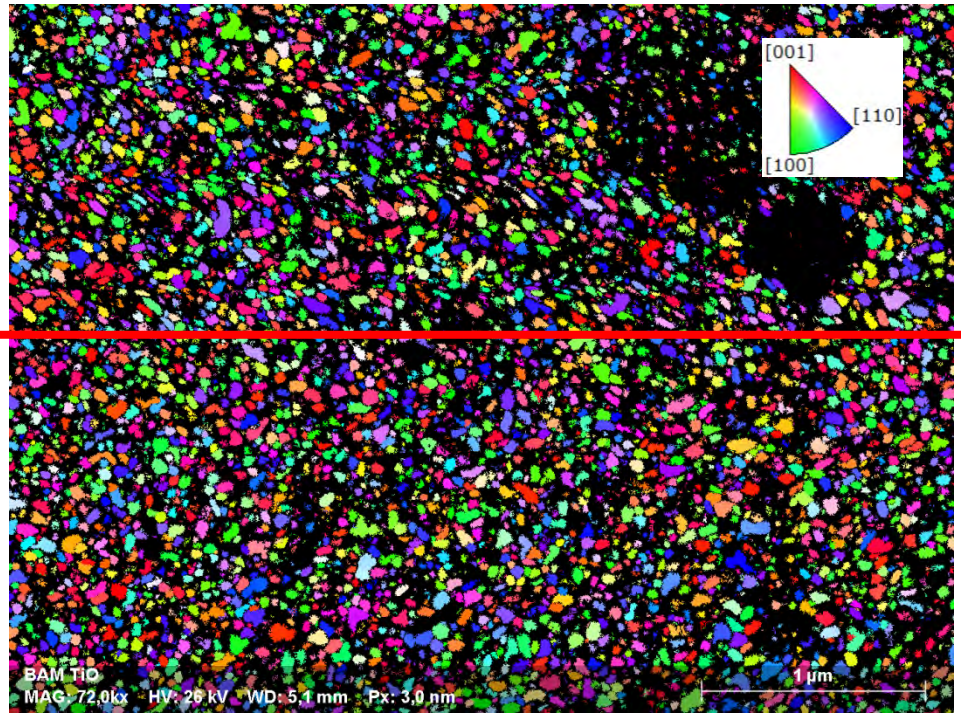
Step size: 3 nm

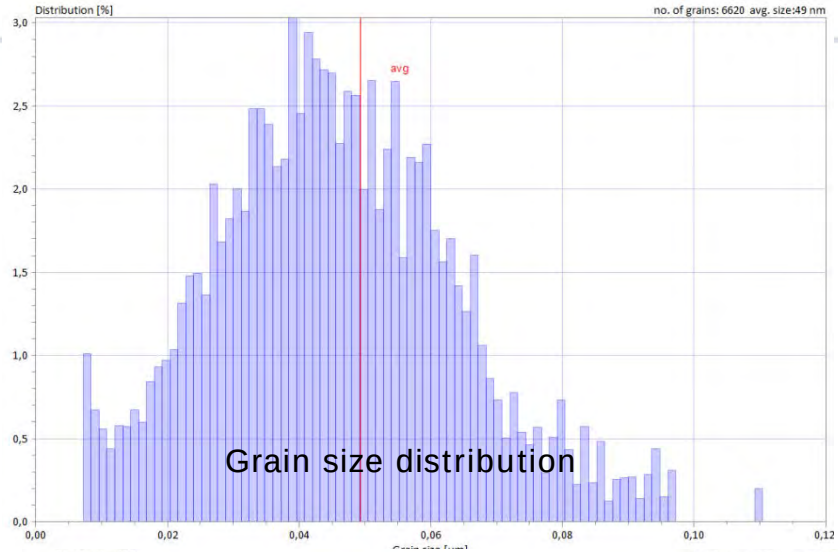
* In collaboration with Nicole Wollschläger BAM, Berlin.
Sample preparation:FIB then Nanomill (Fischione)

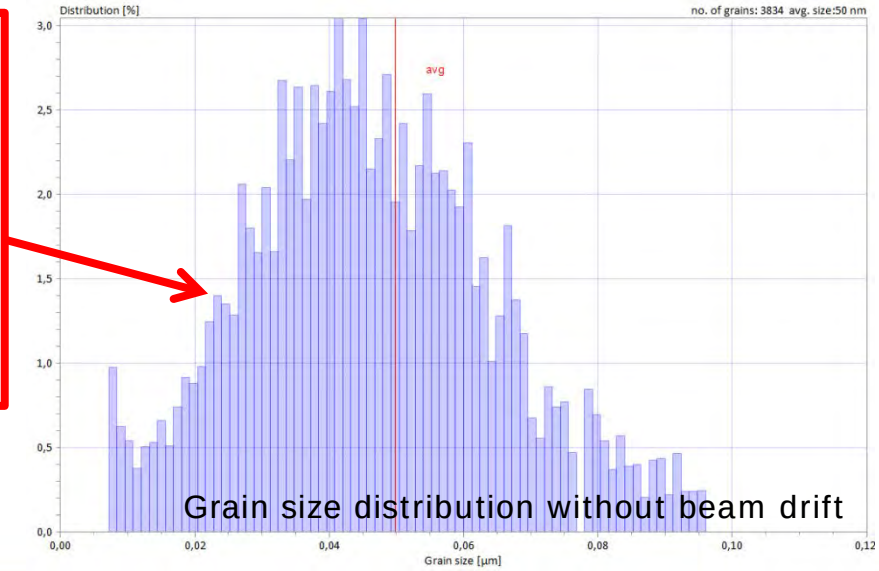
High speed TKD in SEM

characterisation of porous TiO_2 layer (DSSC)

Grain size distribution







High speed TKD in SEM

characterisation of porous TiO_2 layer (DSSC)

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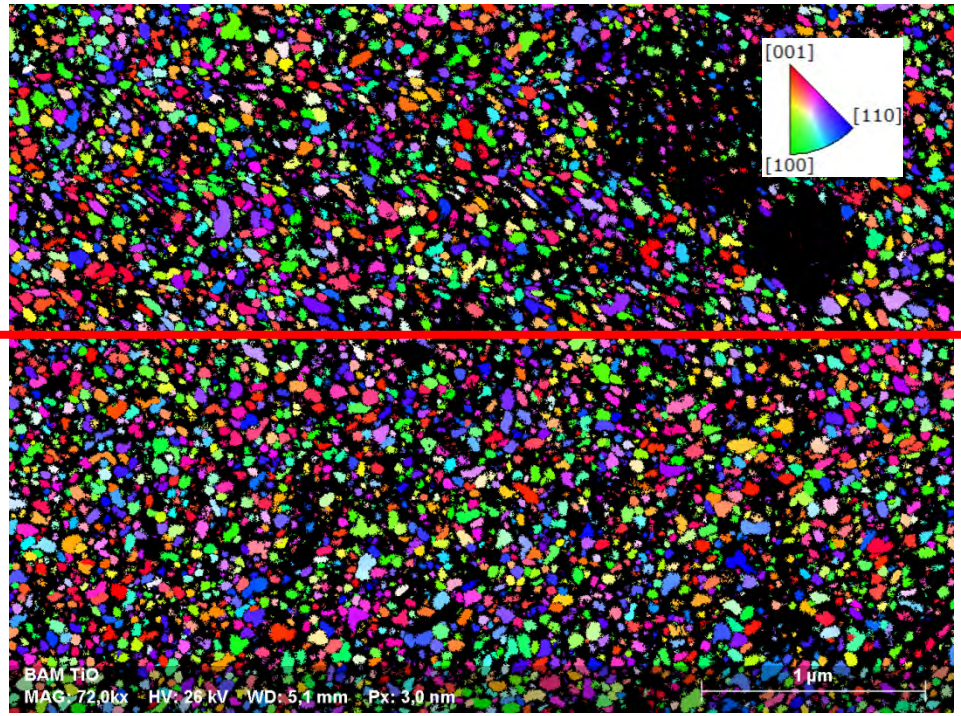
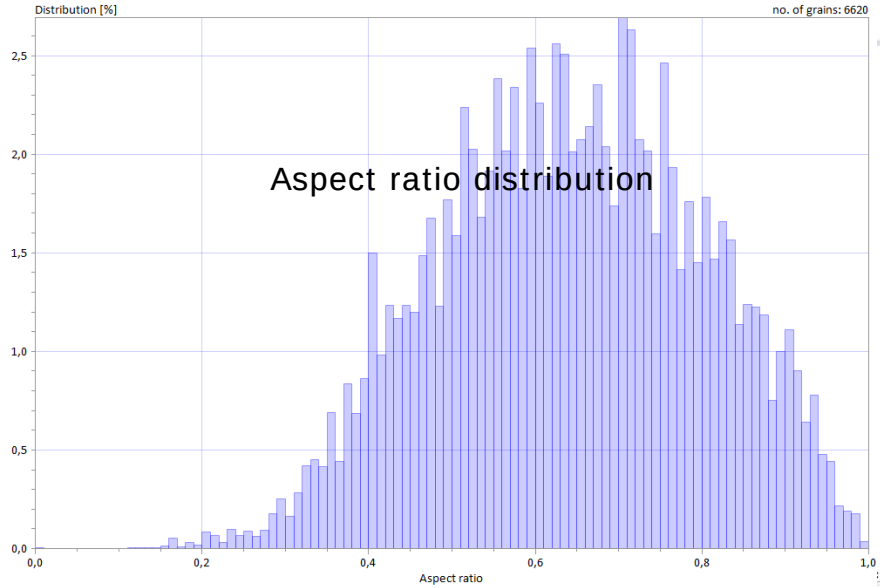
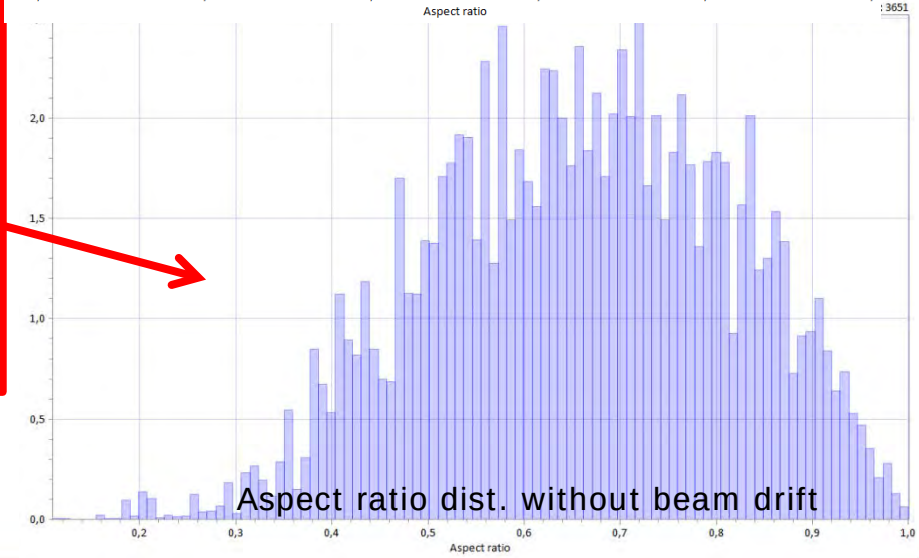






High speed TKD in SEM

characterisation of porous TiO_2 layer (DSSC)

	AFM	TEM	TSEM	TKD
Major axis (nm)	73.5	53	50	51
Minor axis (nm)	48.5	34	32	30
Aspect ratio	0.66	0.64	0.64	0.65

On-axis TKD :

- Fast: >1Mio. points in 1hour
- Reliable: providing analysis of statistical significance with > 5000 NPs measured in one map (FIB lamella)
- Practical: low contamination, sample can be further investigated

Materials Characterization Volume 131, September 2017, Pages 39–48
"*Characterization of the inner structure of porous TiO_2 nanoparticle films in dye sensitive solar cells (DSSC) by focused ion beam (FIB) tomography and transmission Kikuchi diffraction (TKD) in the scanning electron microscope (SEM)"* Wollschläger *et al., 2017*

High speed TKD in SEM

Summary

- Stronger signal

  - Faster measurements - up to 660fps

  - Use of low probe current for improved spatial resolution - < 2nm

- Minimum gnomonic distortions

  - Improved band detection and indexing quality

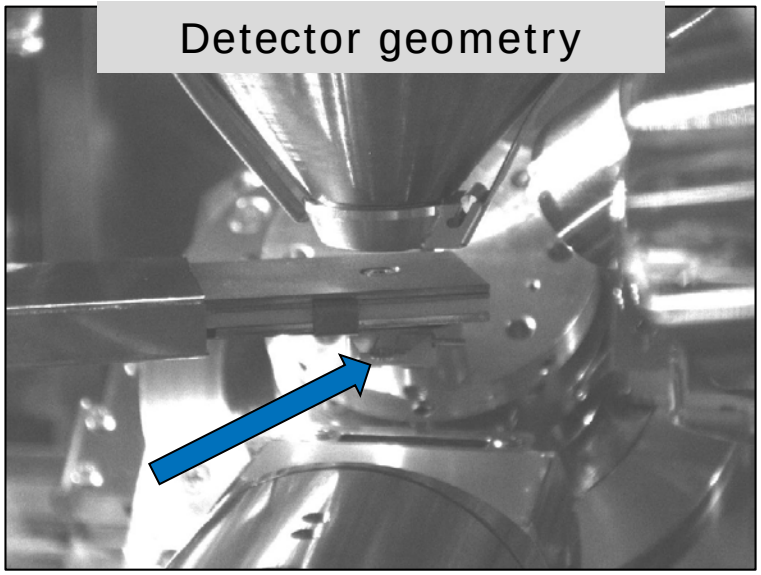
- Direct electron detection imaging (Si diodes) for Dark & Bright field images – ~1nm resolution

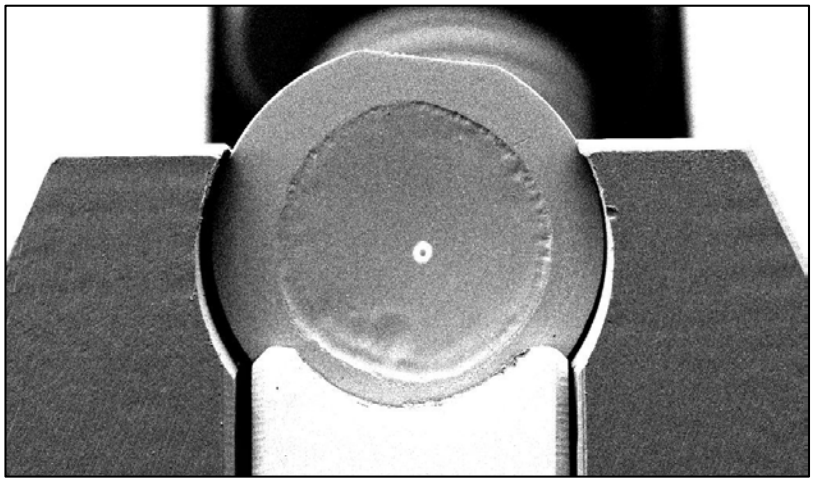
- large area orientation mapping at nanometer resolution

- **Combined EDS/TKD measurement with FlatQuad**

Fast TKD/EDS simultaneous analysis

OPTIMUS TKD & XFlash FlatQuad





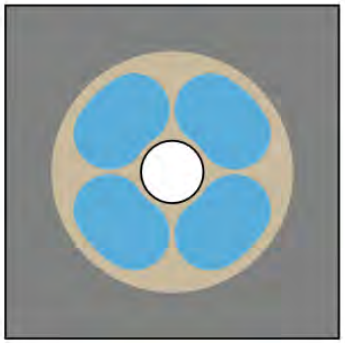
Fast simultaneous TKD/EDS measurements:

- *e*-Flash^{FS} EBSD™ detector retrofitted with OPTIMUS TKD™
- XFlash® FlatQuad
- Sample: ODS ferritic steel
- Bruker designed TKD sample holder

Fast TKD/EDS simultaneous analysis

XFlash FlatQuad

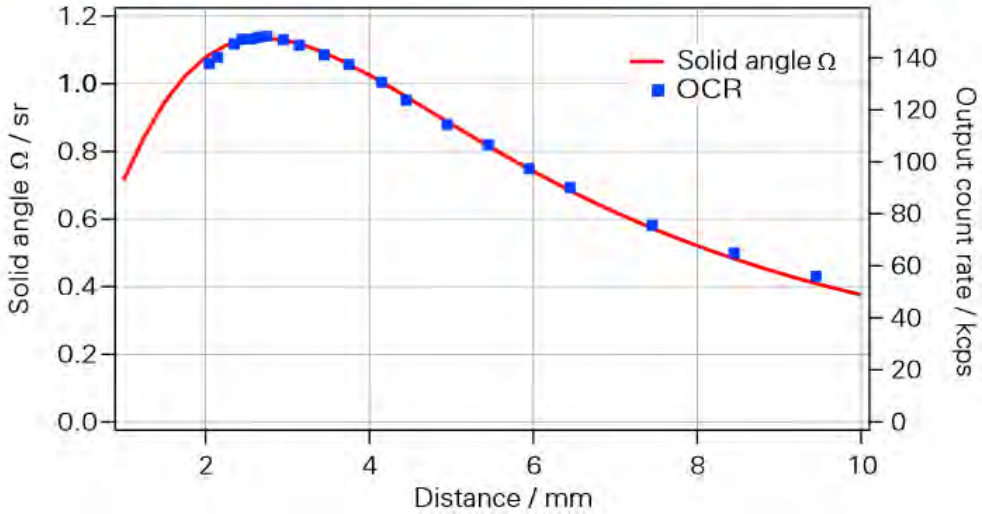
Detector layout



XFlash FlatQuad EDS detector:

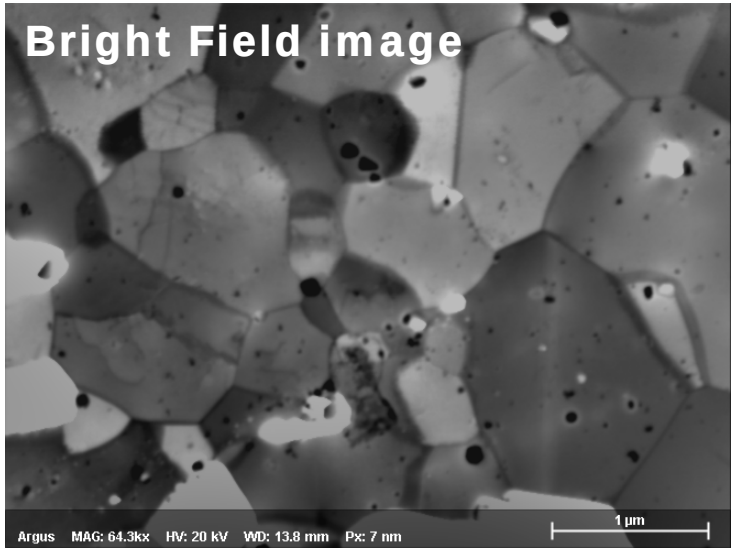
- 4x15mm^2
- Highest solid angle (up to 1 sr)
- High take-off angle > 60°

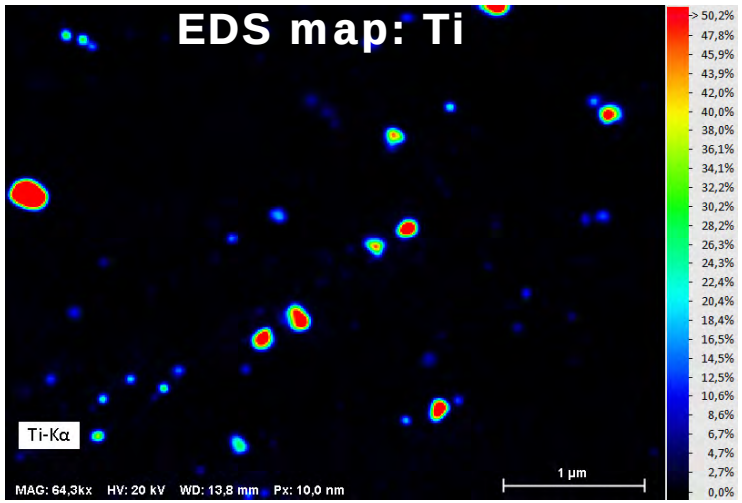
Solid angle and OCR as a function of the detector-sample distance



Fast TKD/EDS simultaneous analysis

OPTIMUS TKD & XFlash FlatQuad



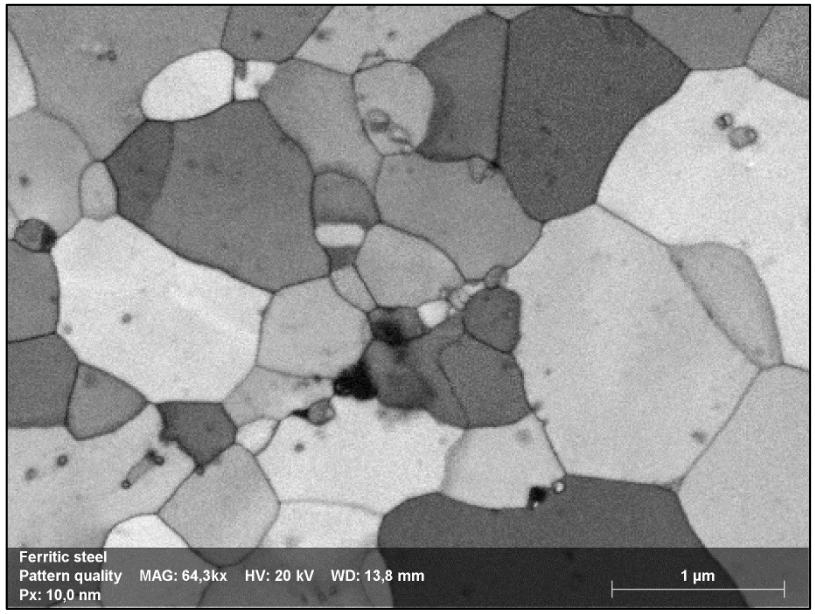


Key EDS parameters:

- Acquisition speed: 272 spectra/sec
- Probe current: 7nA
- Input count rate: ~1.5Mcps
- Dead time: 33%
- Output count rate: ~1Mcps
- Counts per spectrum: >3000

Super fast TKD/EDS measurements

OPTIMUS TKD & XFlash FlatQuad integration

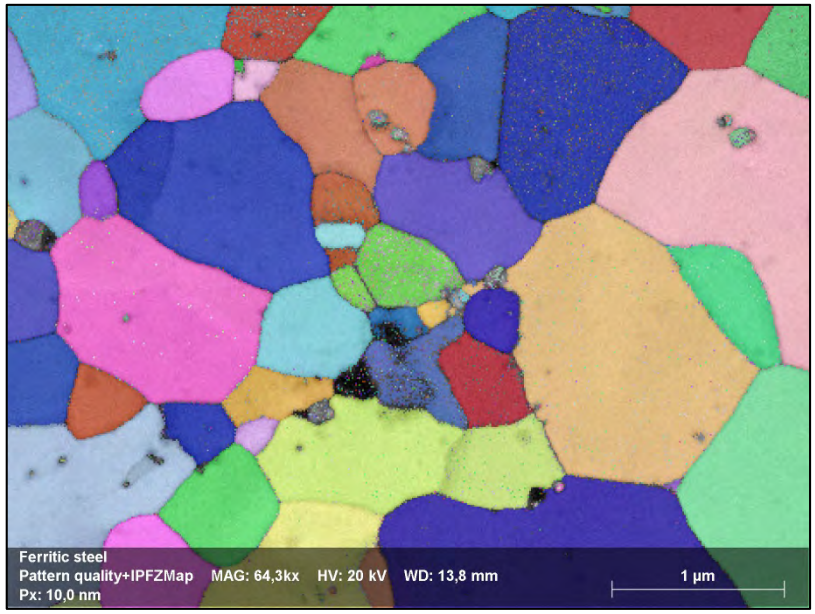


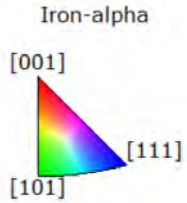
Offline phase ID and reanalysis:

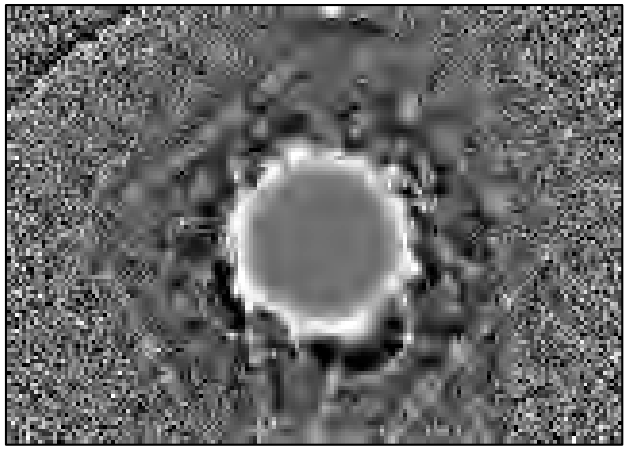
- Speed with just reindexing: 10,000pps
- Reanalysis time: 18s
- Ferrite (red) and Ti_2O_3 (green)

Super fast TKD/EDS measurements

OPTIMUS TKD & XFlash FlatQuad integration







Offline phase ID and reanalysis:

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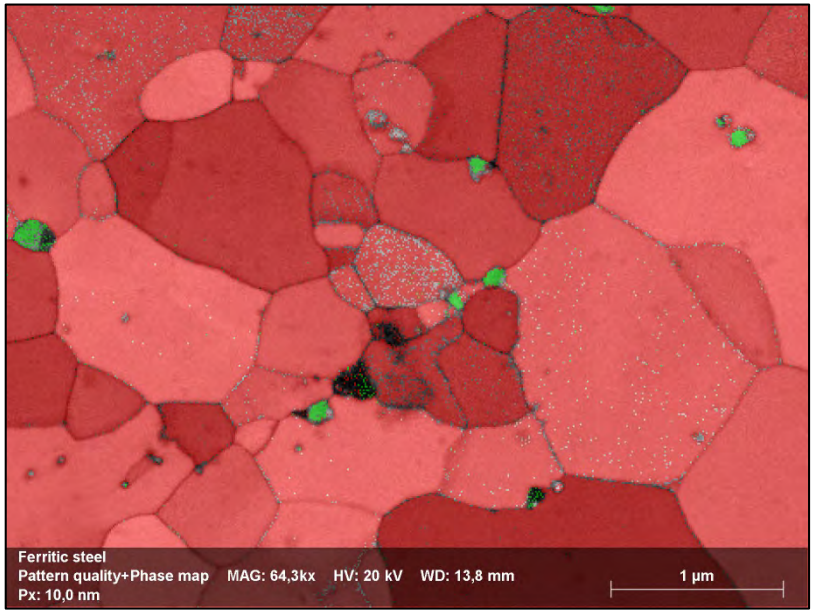
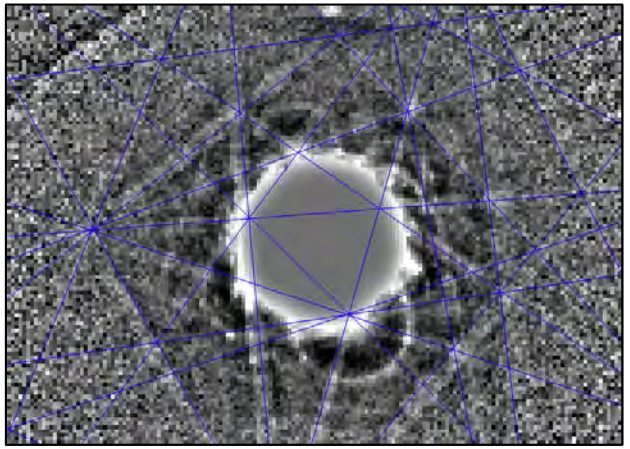
Super fast TKD/EDS measurements

OPTIMUS TKD & XFlash FlatQuad integration



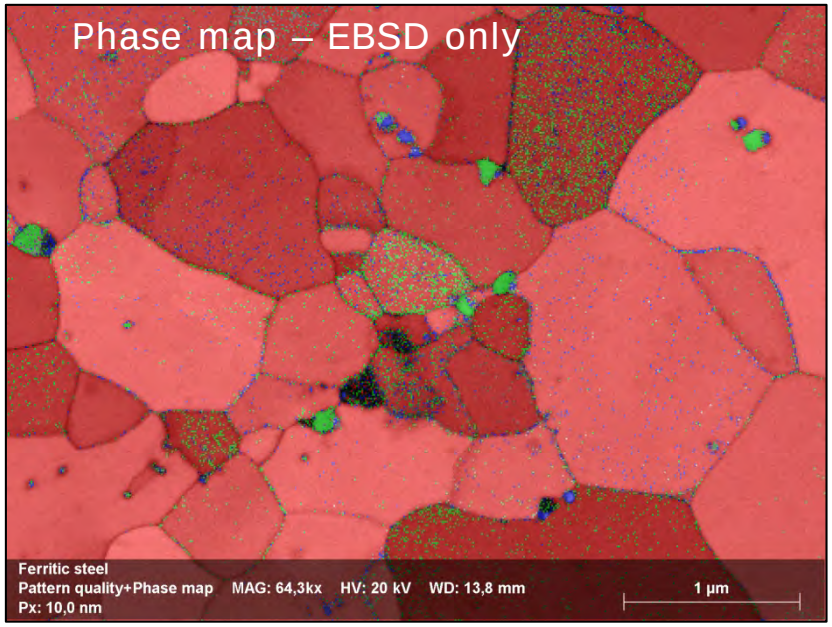


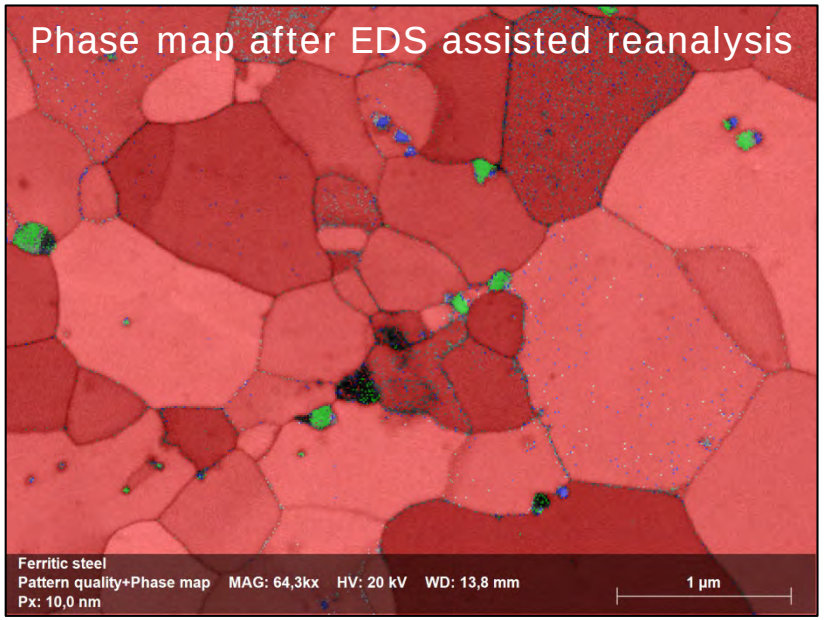
Offline phase ID and reanalysis:

- Speed with just reindexing: 10,000pps
- Reanalysis time: 18s
- Ferrite (red) and Ti_2O_3 (green)

Super fast TKD/EDS measurements

OPTIMUS TKD & XFlash FlatQuad integration





Offline phase ID and reanalysis :

- Speed of EDS assisted reanalysis: 500pps
- Reanalysis time:5:28min
- Ferrite (red), Ti_2O_3 (green), $Y_2(Ti0.993Cr0.007)_2O_7$ (blue)

Fast TKD/EDS simultaneous analysis

OPTIMUS TKD & XFlash FlatQuad

Summary:

- Fast simultaneous TKD/EDS measurements
- Completion of map:
  - Offline phase ID
  - Offline super fast reindexing (reanalysis)
  - Offline fast EDS assisted reanalysis
- High efficiency and high quality data

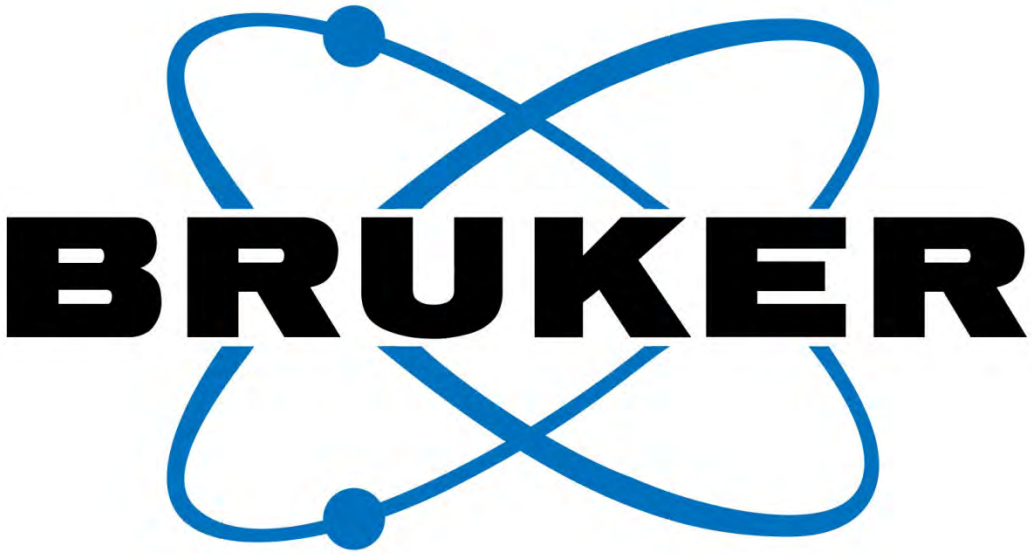
Integration of two unique, high performance detectors!

Q&A

Session is now open for questions

Please use the Q&A box to send us your questions.

(press: *send)*



Innovation with Integrity