Transmission Diffraction via a horizontally positioned detector

Fundenberger J.J. [1, 2], **Bouzy E.** [1, 2], **Goran D.** [3], **Guyon J.** [1, 2], **Morawiec A.** [4], **Yuan H.** [1, 2]

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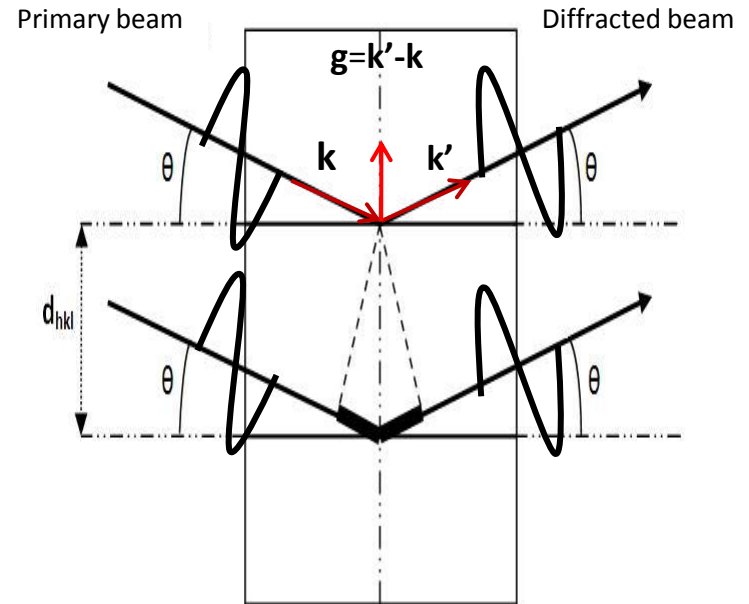
[3] *Bruker Nano GmbH, Am Studio 2D,12489 Berlin, Germany*

[4] *Institute of Metallurgy and Materials Science, Polish Academy of Sciences, Reymonta 25, 30059 Krakow, Poland*

What can we learn from diffraction ?

Diffraction $\longrightarrow$ Reciprocal lattice

- Orientation

- Elastic strains

- Dislocation density

Primary beam

Diffracted beam

$g=k'-k$

k k'

θ θ

d_{hkl}
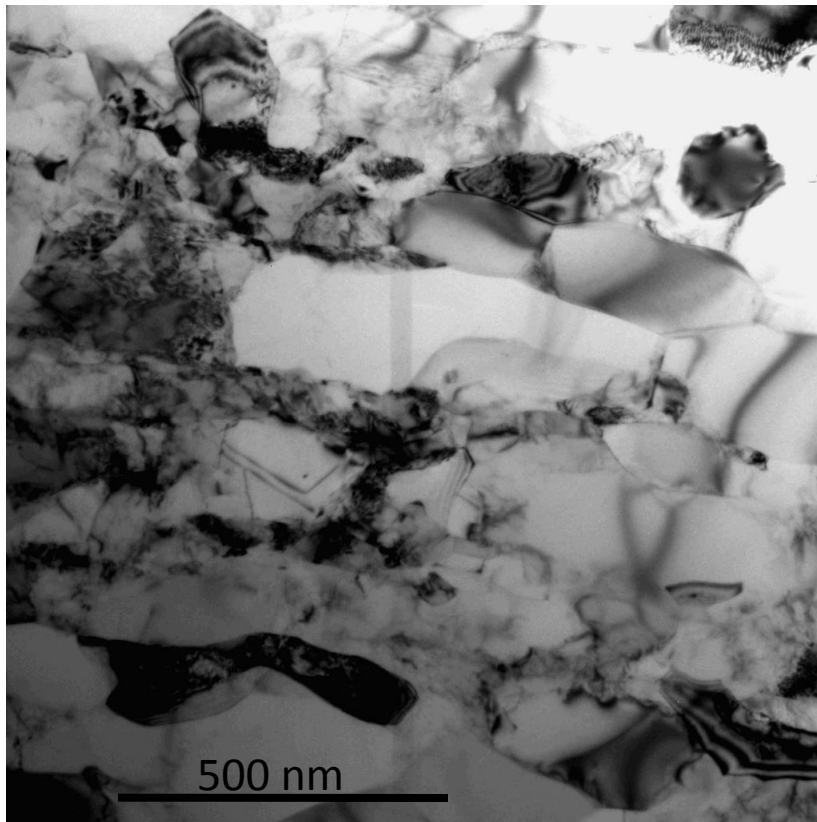
θ θ

Orientation

RX, neutrons
$\rightarrow$ ODF

electrons
$\rightarrow$ local ODF, Grain size,
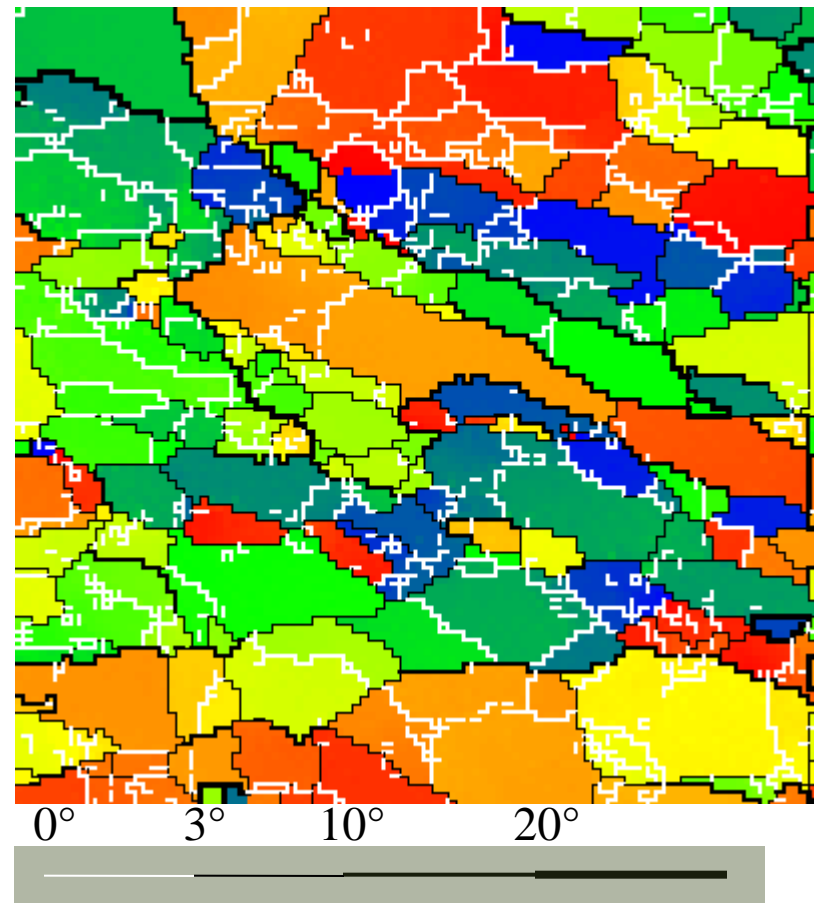GBCD, GND…

Orientation maps

Diffraction contrast is not orientation contrast
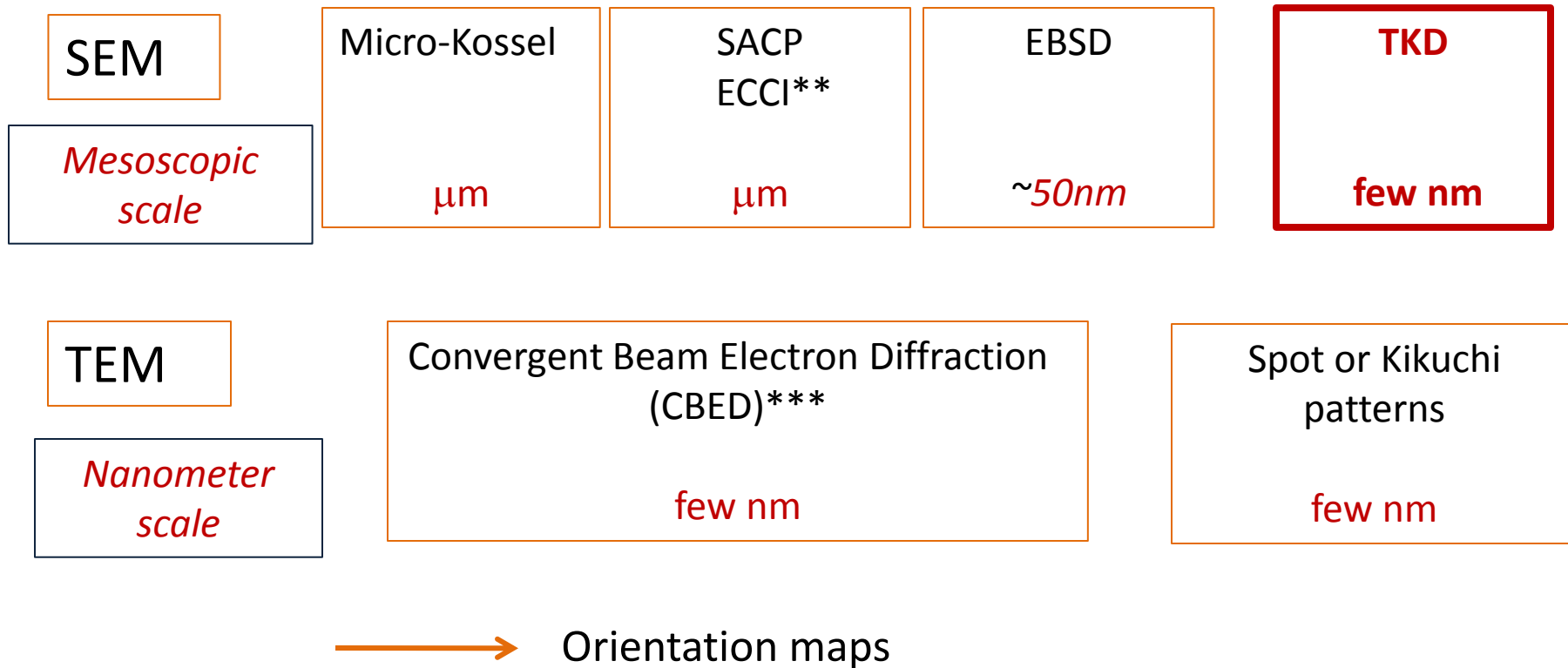
TEM Bright Field Image

Orientation map from Kikuchi patterns (EP)

Methods of local orientation determination *

SEM	Micro-Kossel	SACP ECCI**	EBSD	**TKD**
Mesoscopic scale	μm	μm	*~50nm*	**few nm**

TEM	Convergent Beam Electron Diffraction (CBED)***	Spot or Kikuchi patterns
Nanometer scale	few nm	few nm

→ Orientation maps

* *"Orientation precision of TEM-based orientation mapping techniques" A.Morawiec, E.Bouzy, H.Paul, J.J.Fundenberger* **Ultramicroscopy** *136 (2014) pp. 107–118*

** *"Accurate electron channeling contrast analysis of a low angle sub-grain boundary" Mansour, H.; Crimp, M. A.; Gey, N.; Maloufi N. Scripta Met. 109 (2015) pp.76-79*

*** *"Determination of lattice parameters from multiple CBED patterns: A statistical approach" Brunetti, G.; Bouzy, E.; Fundenberger, J. J.; Morawiec A.; Tidu A. Ultramicroscopy 110 (2010) pp.269-277*
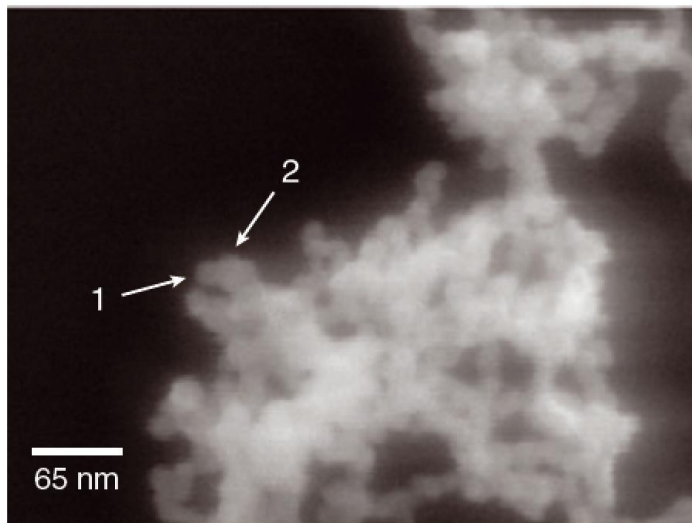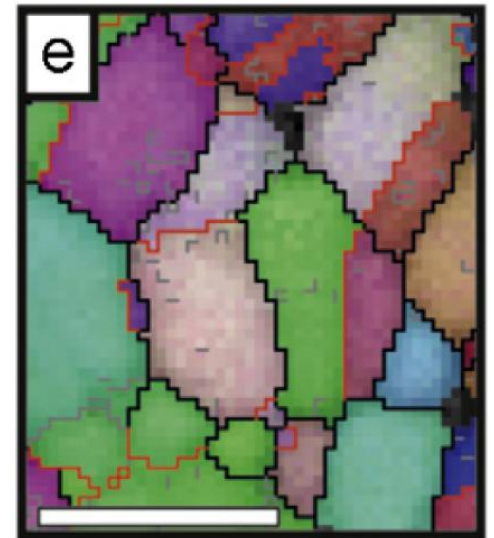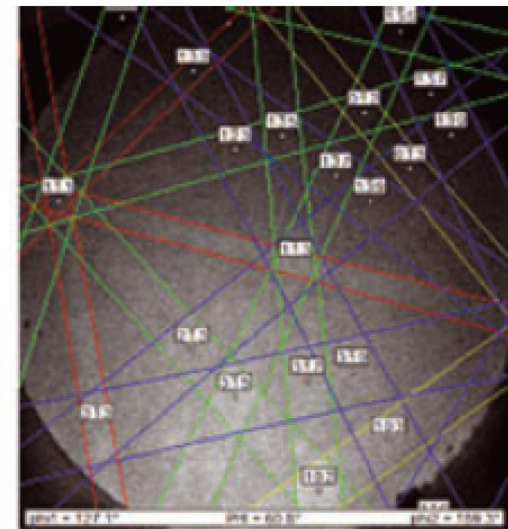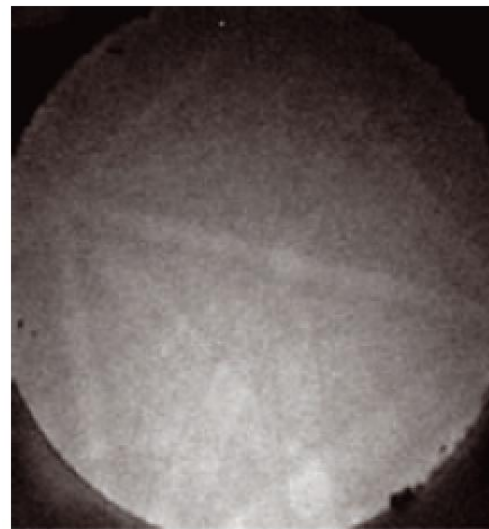
✓ High spatial resolution

Keller R.R. & Geiss R.H., *J. Microsc.*. 245, 245 (2012).

50nm
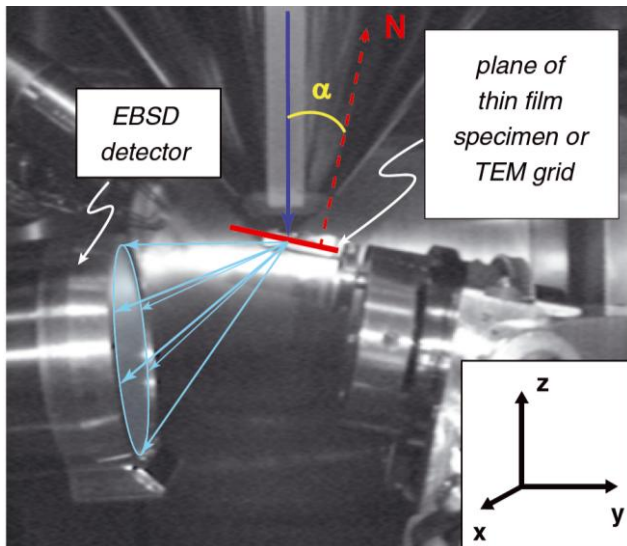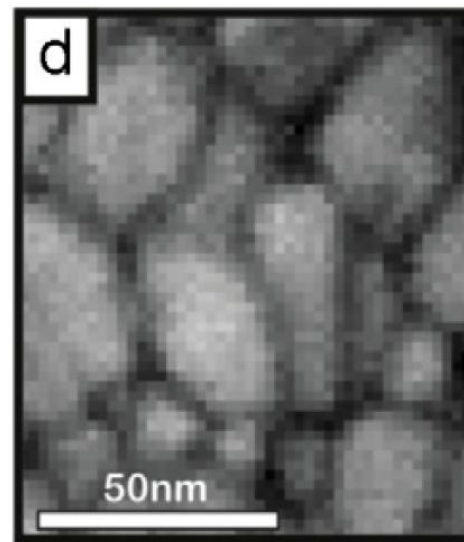
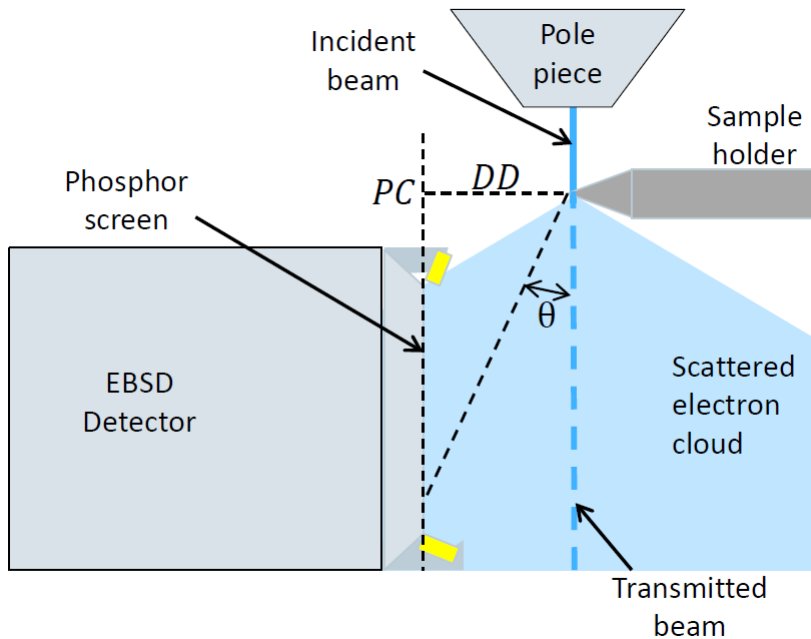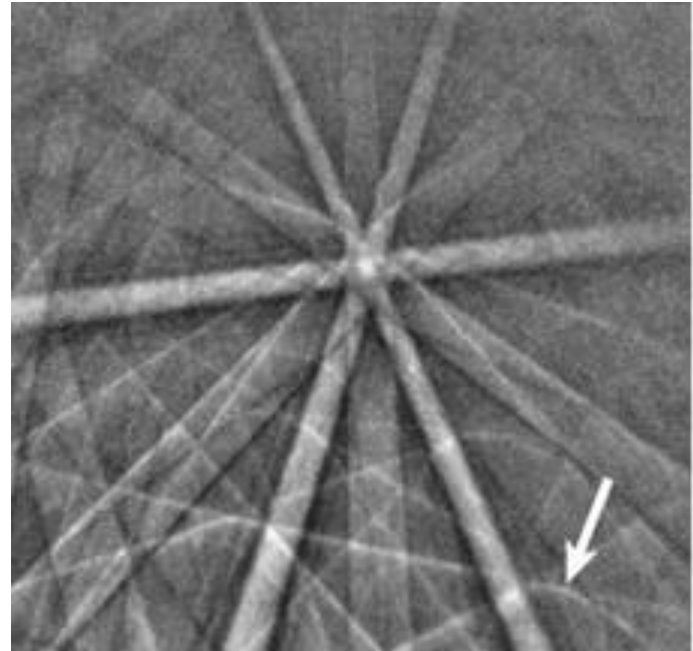**Al alloy, step 2 nm**   Trimby P.W., *Ultramicroscopy.* **120**, 16 (2012).

65 nm

Fe-Co nanoparticles

Keller R.R. & Geiss R.H., *J. Microsc.*. **245**, 245 (2012).

Trimby P.W. *Ultramicroscopy* 120, 16 (2012)

- Important distortion of Kikuchi bands

- Change of intensity from top to bottom

Orientation imaging from Kikuchi patterns using TEM

E.P. (Euclid's phantasies)

DigitalMicrograph®

TEM control

Camera control

Corrected patterns

EP

In-house indexing software

CM 200

Kikuchi / CBED   **Correction**   **Detection of lines**   **Indexation Orientation**
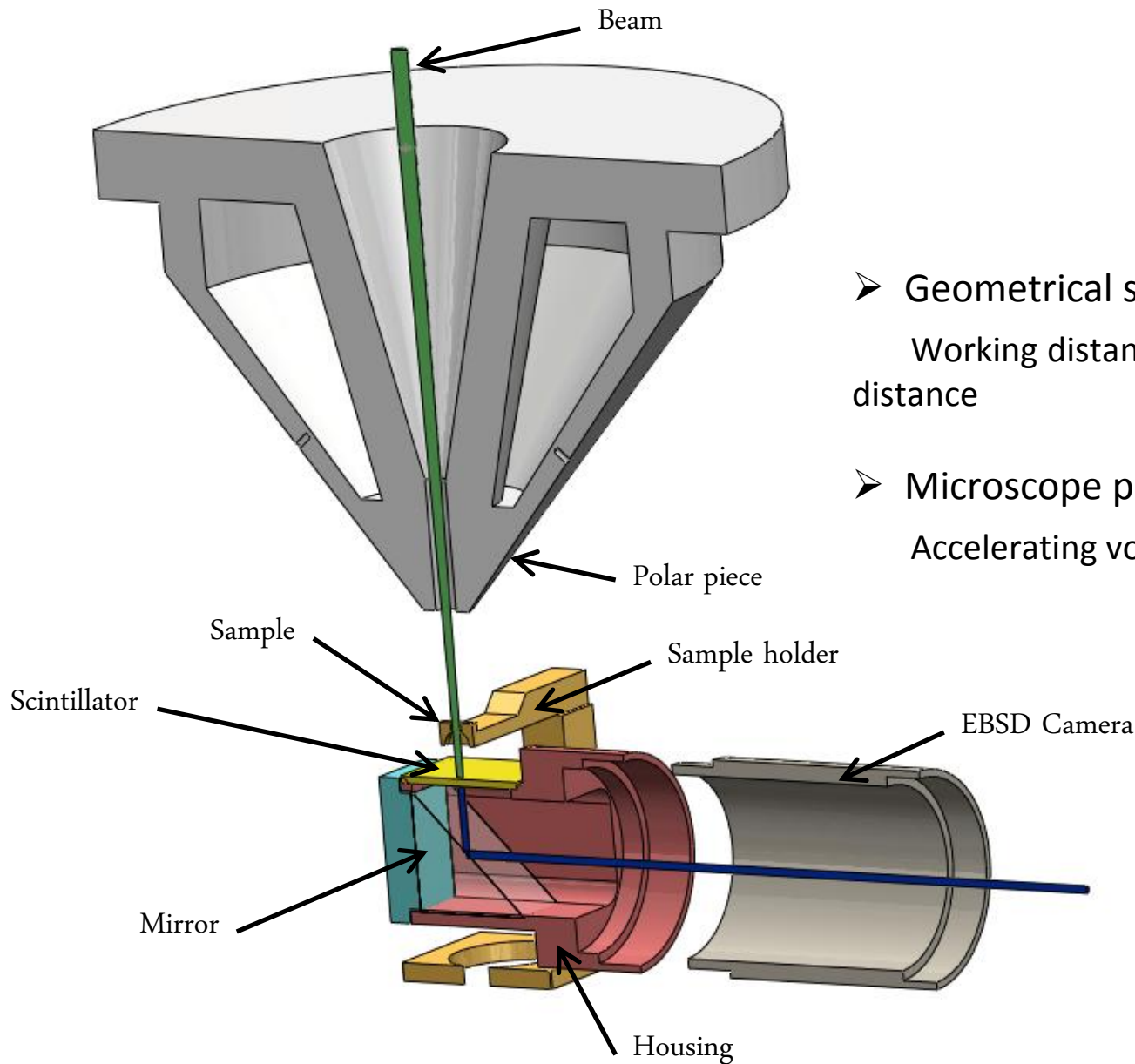
Spatial resolution about 10nm
Angular resolution about 0.2°

e⁻
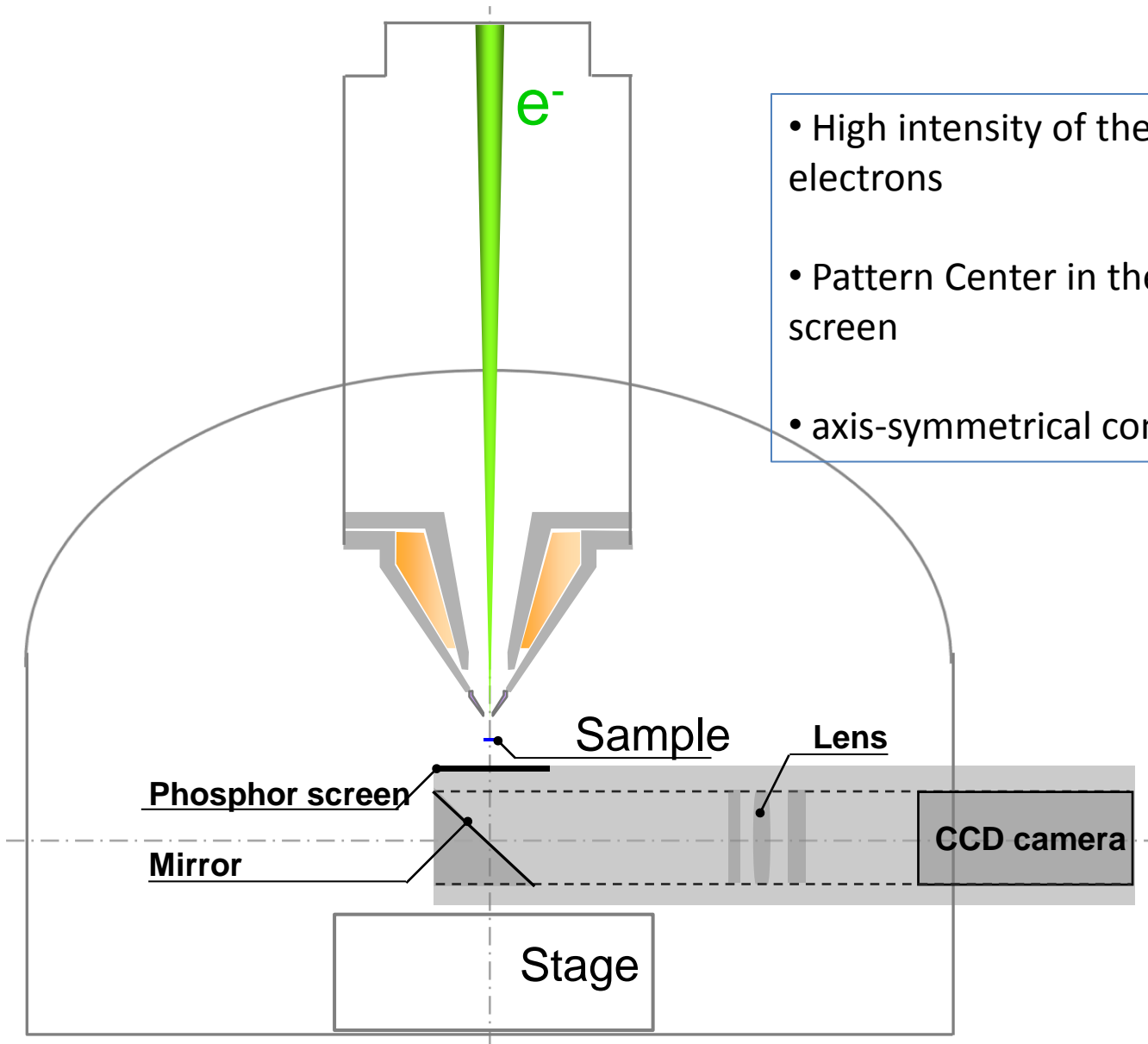
- High intensity of the forward scattered electrons

- Pattern Center in the center of the phosphor screen

- axis-symmetrical configuration

Sample

Lens

Phosphor screen

Mirror

CCD camera

Stage

We propose to name this new set-up:
On Axis Transmission Diffraction in SEM
(On axis TD-SEM)

8

Beam

Polar piece

Sample

Sample holder

Scintillator

EBSD Camera

Mirror

Housing

➢ Geometrical set-up:

Working distance (WD), sample-scintillator distance

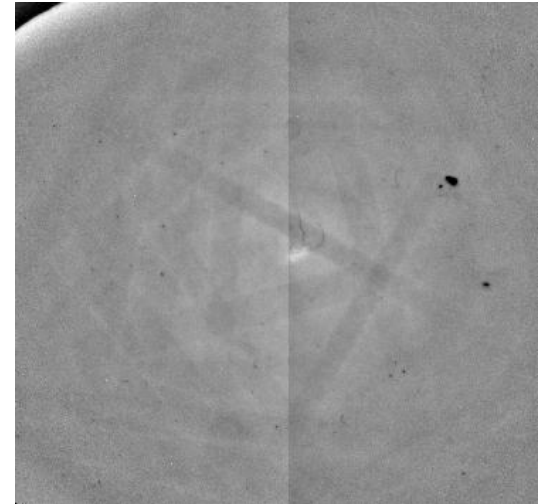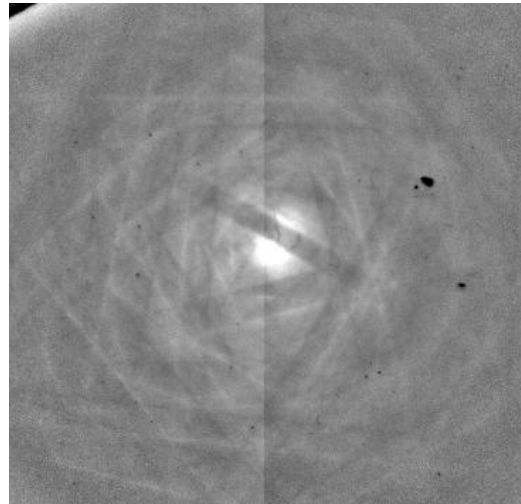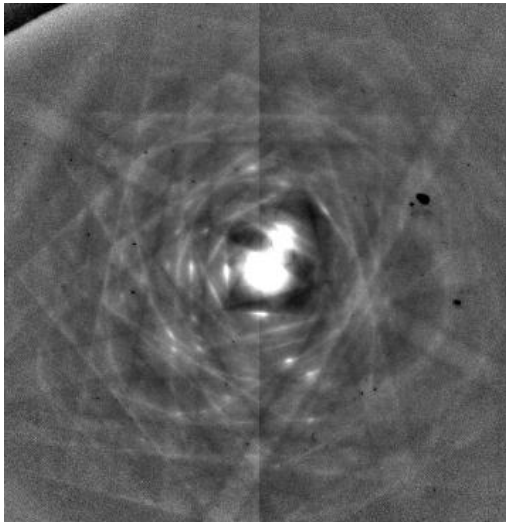➢ Microscope parameters:

Accelerating voltage, beam current

Diffraction patterns
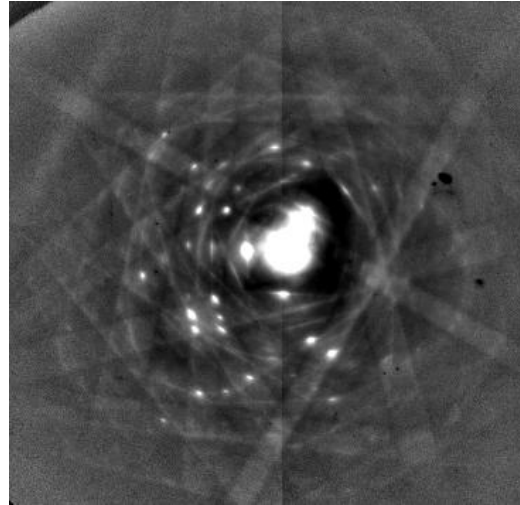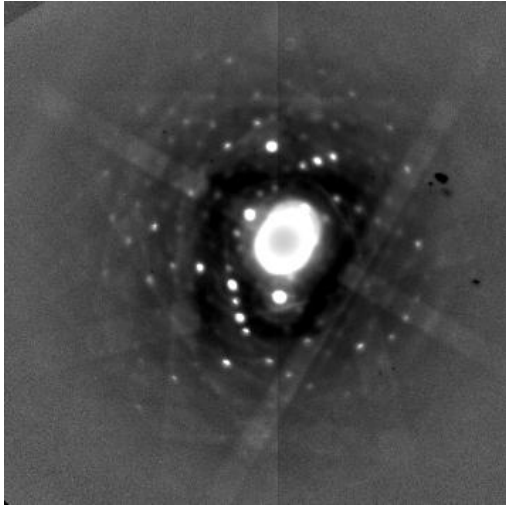
The type (spot/Kikuchi) and the contrast (line, band, …) of the diffraction patterns change depending on the thickness.
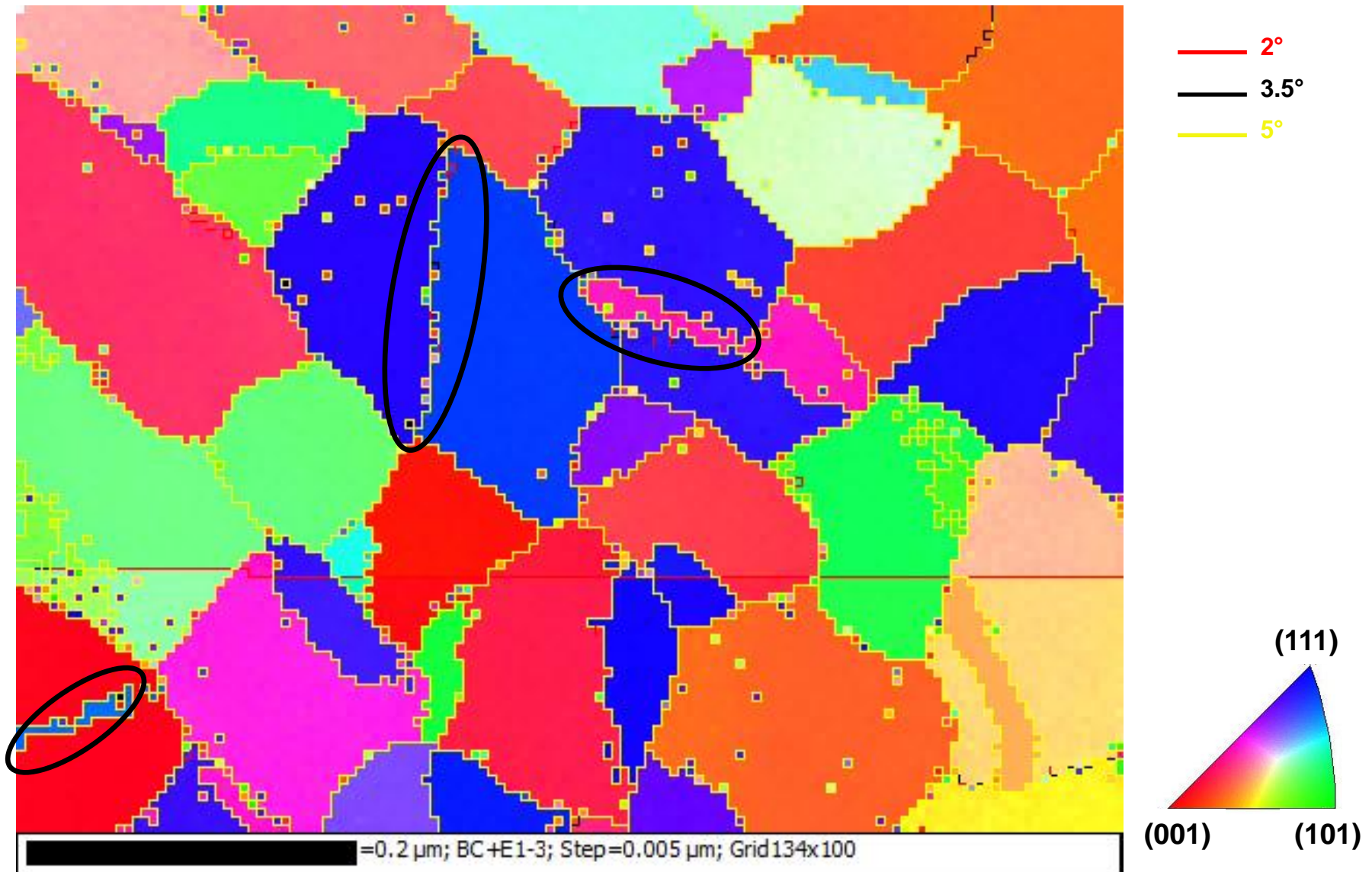
Patterns from Si
with increasing thickness
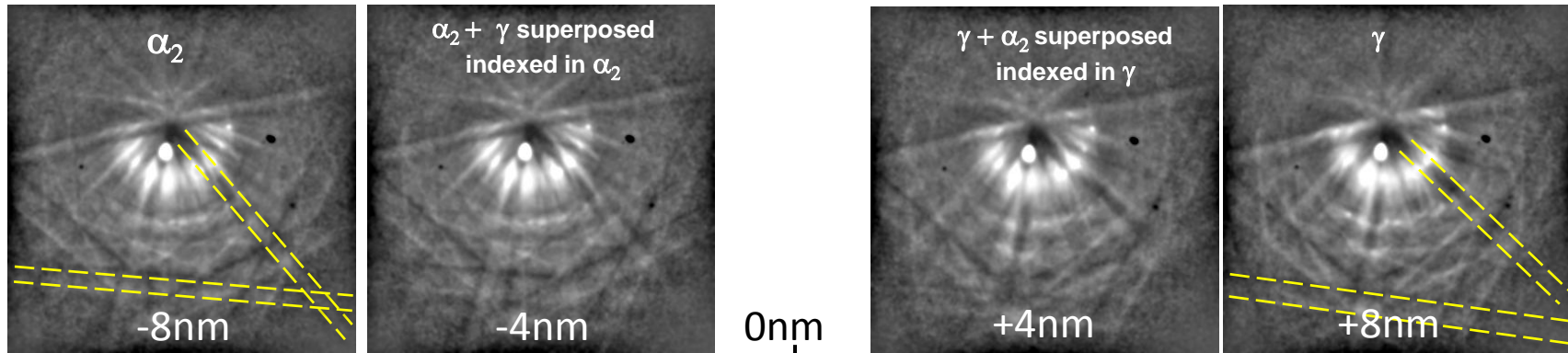(same trend for decreasing voltage)

increasing thickness

increasing thickness

=0.2 µm; BC+E1-3; Step=0.005 µm; Grid 134x100

Si, step size 5 nm, 20 kV, 2.7 nA

α₂ -8nm

α₂ + γ superposed indexed in α₂ -4nm

0nm

γ + α₂ superposed indexed in γ +4nm

γ +8nm

Physical resolution about +ou- 8 nm

Interface
α₂ / γ

α₂   γ

Effective resolution +ou- 4 nm

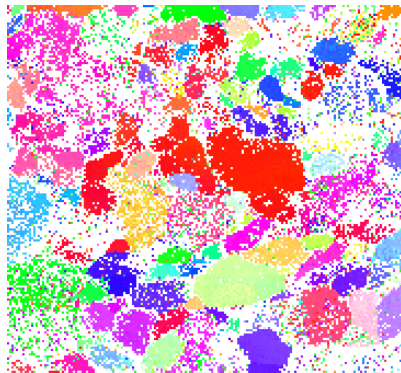(Thickness of the TiAl alloy specimen = 100 nm)

50nm

α simulation

γ simulation
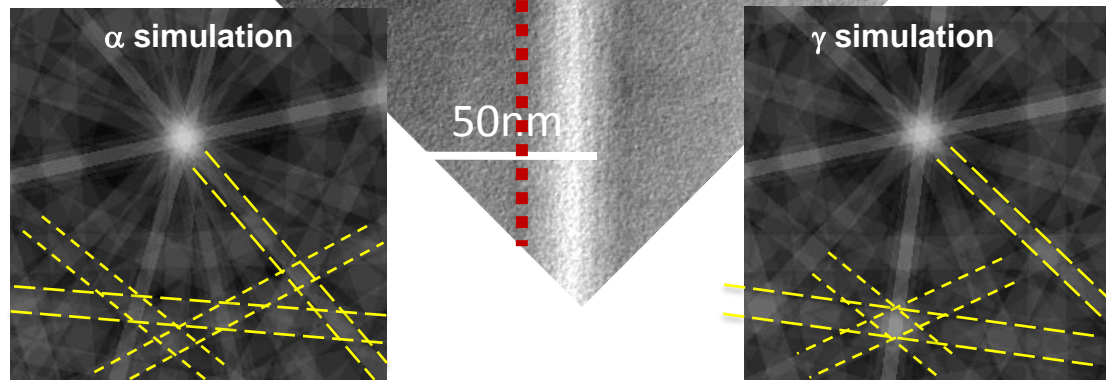
Angular resolution around 0.3°
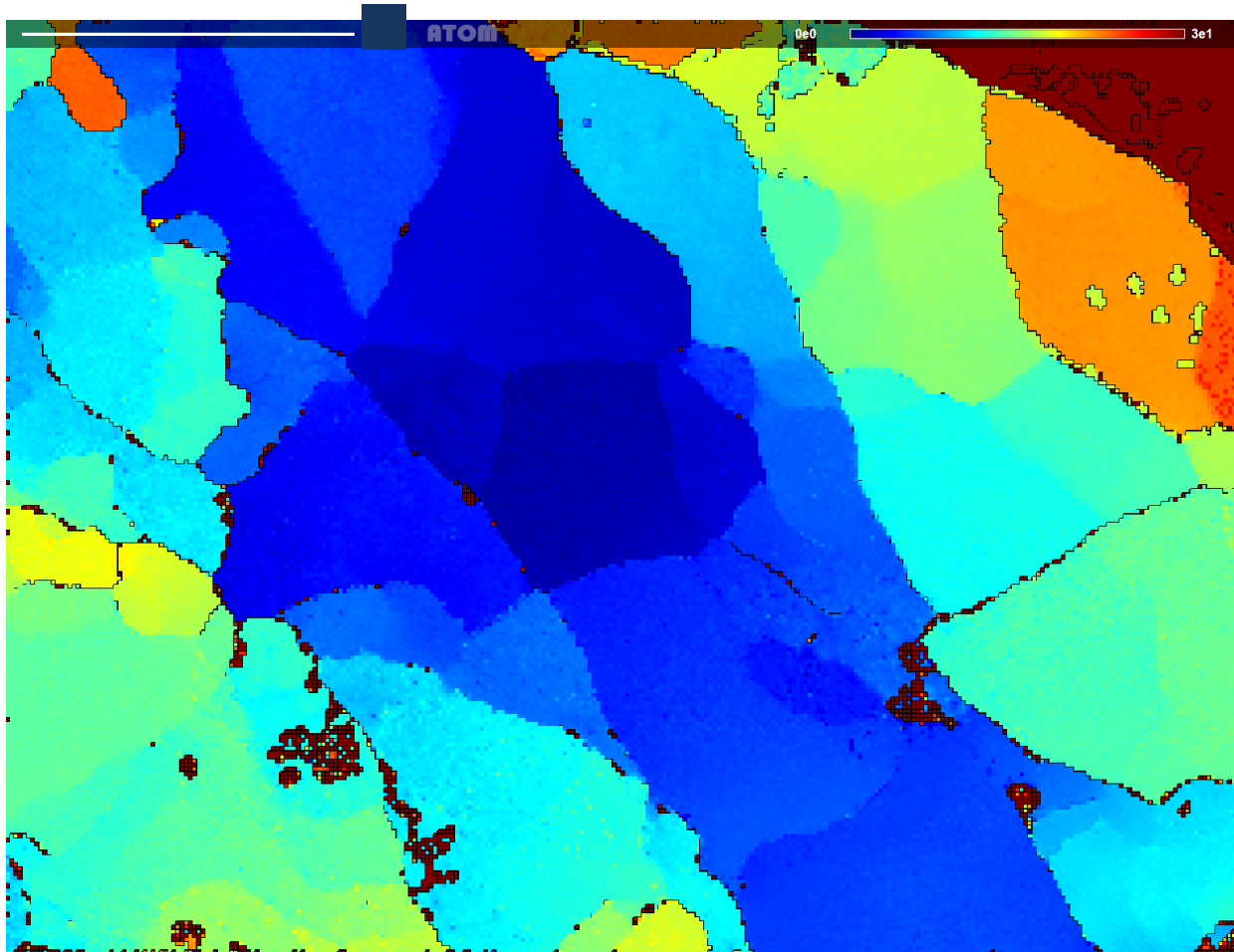
Al strongly deformed by ECAP
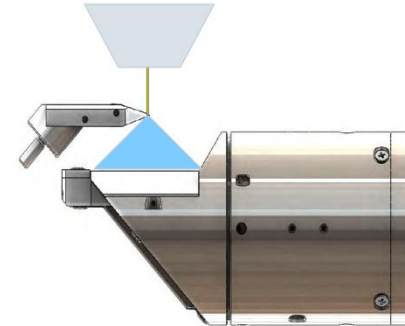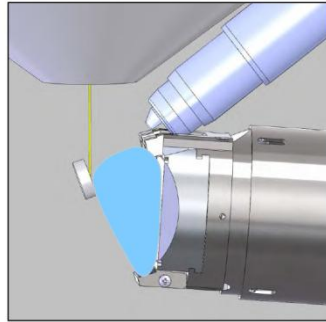
20 KV
2.14 nA
Binning: 4x4
Step size: 10nm

500nm
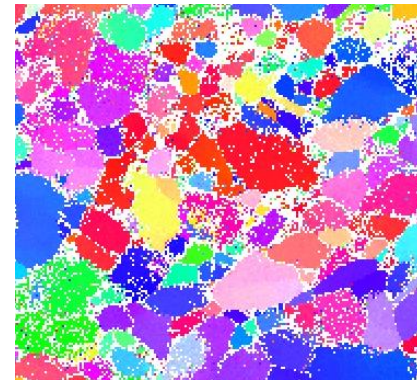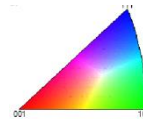
500nm

120°H.P.T. Al-Mg	Off-axis	On-axis
Raw indexation rate (%)	55.2	77.5
Mapping time (min)	53.75	3.19 *(1/17)*

120° HPT Al-Mg   180° HPT Al-Mg

=0.5 µm; BC+IPF_Z; Step=0.01 µm; Grid204x187

=0.5 µm; BC+IPF_Z; Step=0.01 µm; Grid230x212
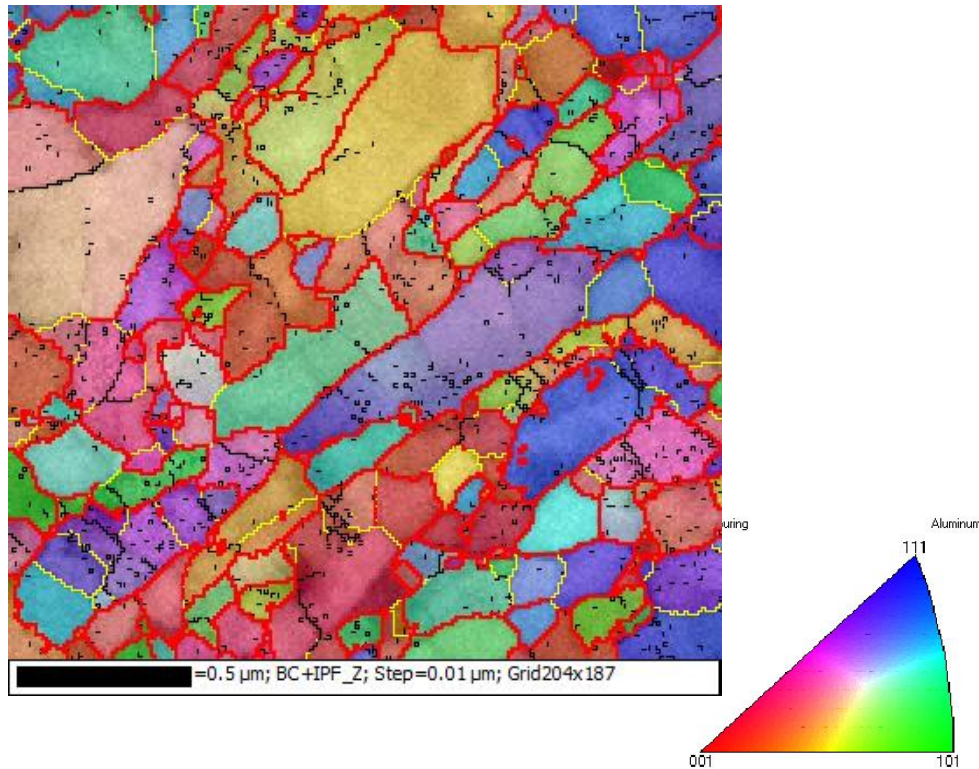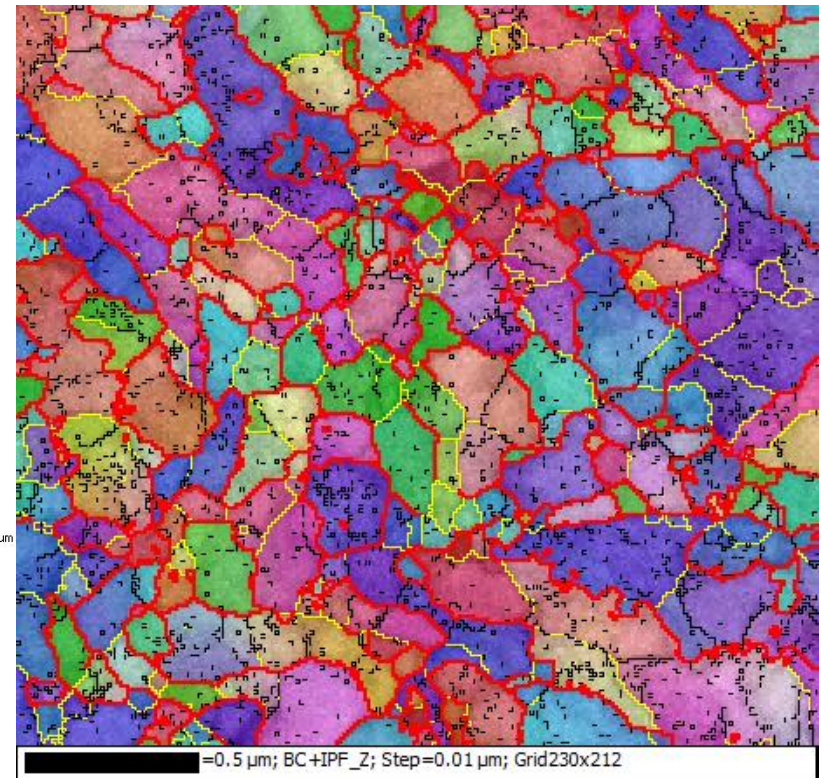
20KV, **2nA**, 10nm, binning 4x4
map time: 4mn (120°), 5mn (180°)

—— **2°**< < 5°
—— 5°< < 15°
—— 15°<

Mean grain size:

120° HPT Al-Mg: 134nm +ou- 3nm

180° HPT Al-Mg: 121nm +ou- 3nm

Standart deviation of the mean grain size calculated from 6 orientation maps.

Conclusions & Perspectives

- A new experimental configuration for Transmission Diffraction in the SEM is suggested. It consists in Transmission Diffraction with an on-axis phosphor screen as a TEM working at low voltage. (named on axis TD-SEM)
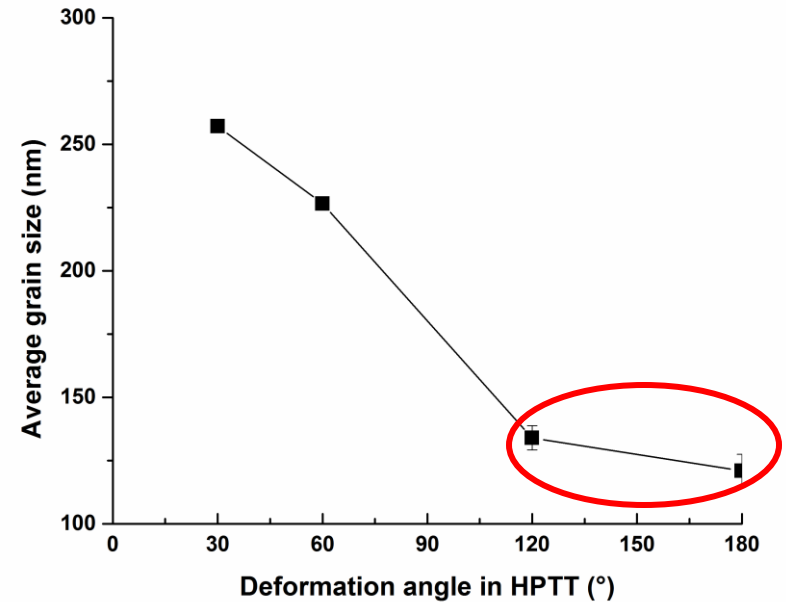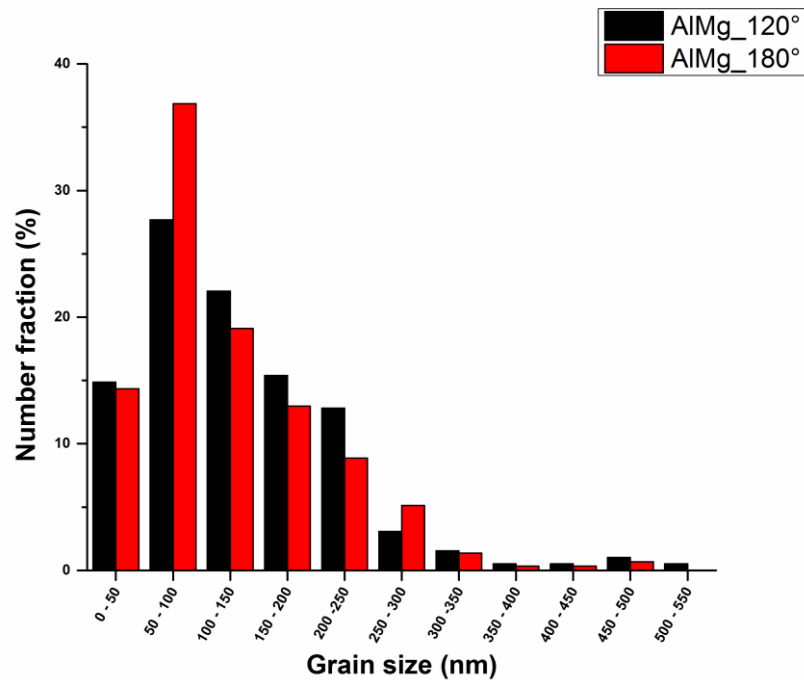
- Its feasibility is demonstrated.

- More complex electron diffraction patterns are obtained than in EBSD. This requires different strategies for spots/lines/bands detection software.

- A good lateral resolution is achieved : better than +-4nm

- The high intensity of forward scattered electrons allows:
> - either to decrease the intensity of the electron probe current in order to improve the lateral spatial resolution
> - or to increase the frequency of the pattern acquisition in order to reduce the acquisition time for an orientation map. (problems of electron beam stability on current SEMs).

- It it is shown that the on-axis Transmission Diffraction on SEM technique is a reliable technique to estimate the grain size distribution and the local misorientation distribution for SPD microstructures.

Bruker's OPTIMUS™ Detector Head for TKD in SEM

Thank you for your attention

« Orientation mapping by transmission-SEM with an on-axis detector»
J.-J. Fundenberger, E.Bouzy, D.Goran, J.Guyon, H.Yuan, A.Morawiec *Ultramicroscopy*, **In Press, Accepted Manuscript**, *Available online 10 November 2015*

« Transmission Kikuchi Diffraction (TKD)via a horizontally positioned detector »
J.-J. Fundenberger, E. Bouzy, D. Goran, J. Guyon, A. Morawiec and H. Yuan
Microscopy and Microanalysis / Volume 21 / Supplement S3 / August 2015, pp 1101-1102

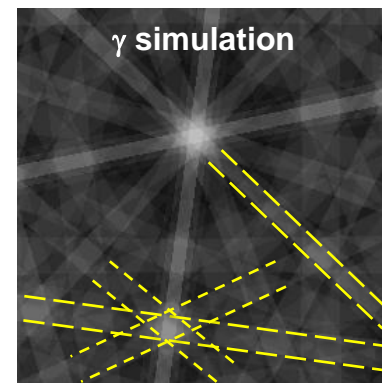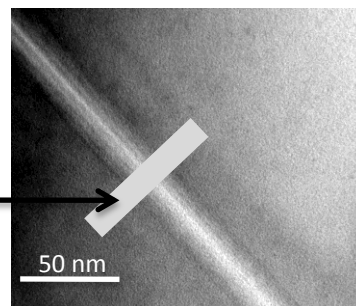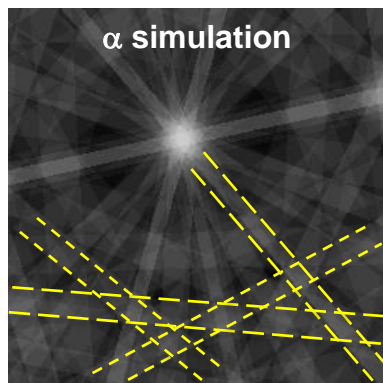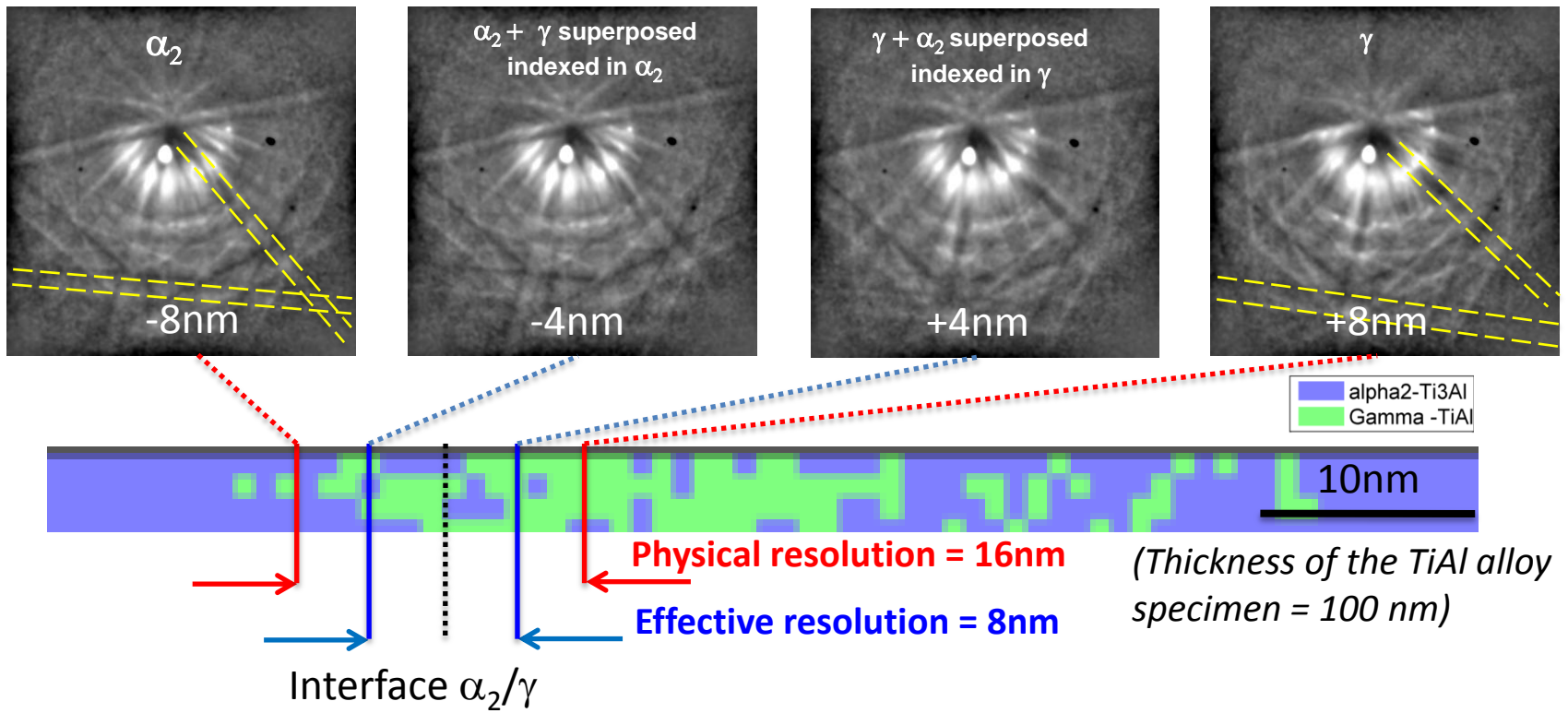Mont Rainier

Thank you for your attention

Conclusions & Perspectives
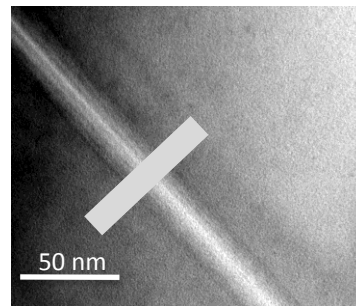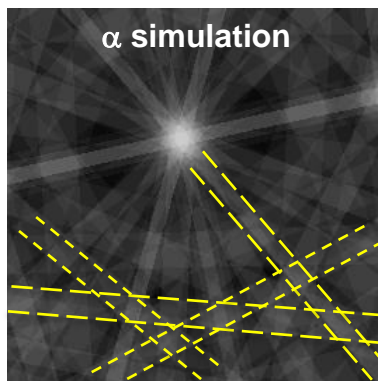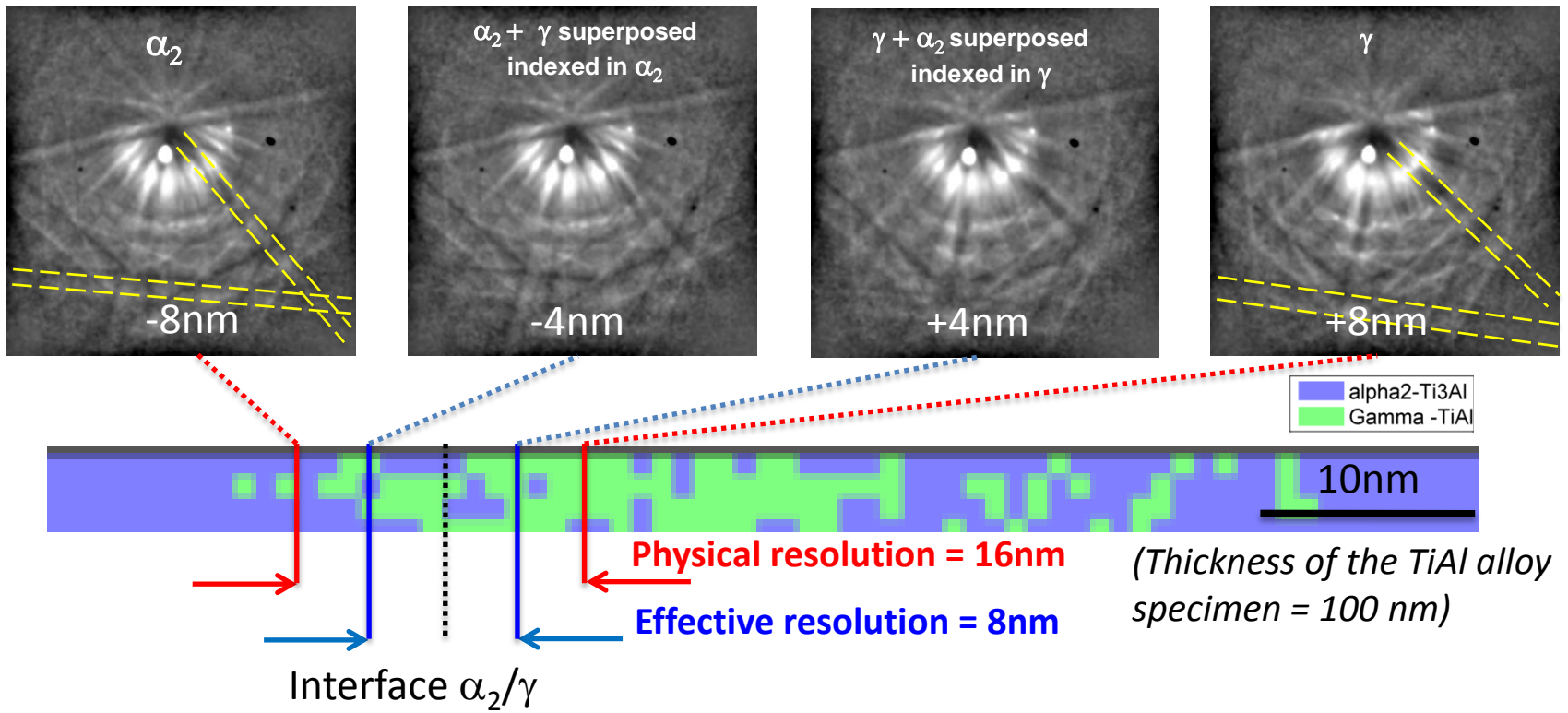
1. Feasibility of horizontally positioned detector for TKD

2. More <u>complicated</u> diffraction patterns than in EBSD.

3. Good lateral spatial resolution : < 5 nm

4. High intensity of forward scattered electrons
   → low electron probe current
   → high frequency of acquisition

5. Problems of electron beam stability for orientation mapping

α_2

$\alpha_2 + \gamma$ superposed indexed in α_2

$\gamma + \alpha_2$ superposed indexed in γ

γ

-8nm

-4nm

+4nm

+8nm

alpha2-Ti3Al
Gamma -TiAl

10nm

Physical resolution = 16nm

(Thickness of the TiAl alloy specimen = 100 nm)

Effective resolution = 8nm

Interface α_2/γ

α simulation

γ simulation

50 nm

α₂ -8nm

α₂ + γ superposed indexed in α₂ -4nm

γ + α₂ superposed indexed in γ +4nm

γ +8nm

alpha2-Ti3Al
Gamma -TiAl

10nm

Physical resolution = 16nm

Effective resolution = 8nm

(Thickness of the TiAl alloy specimen = 100 nm)

Interface α₂/γ
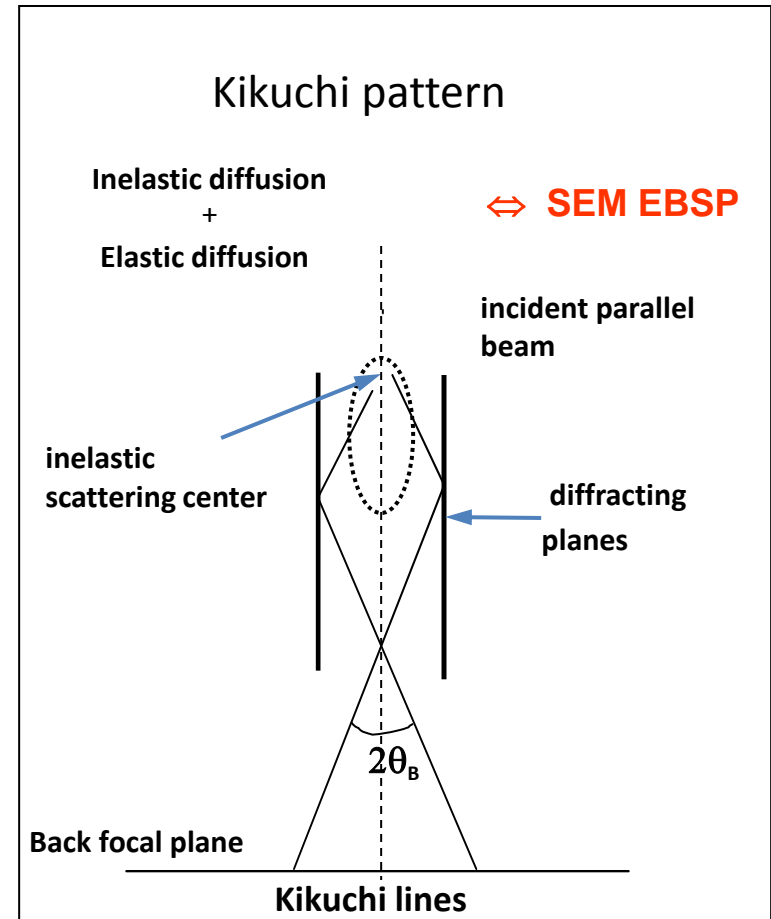
α simulation

50 nm

γ simulation

Spatial resolution:

- *TKD on-axis configuration vs. conventional TKD*:
Thermal diffuse scattering (TDS). Lower mean free path for TDS at low angles.

➡ High intensity Kikuchi patterns at low angles

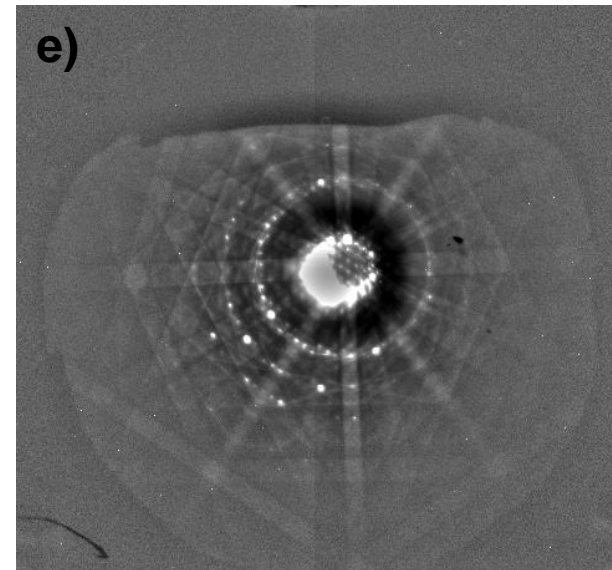➡ Smaller electron probe intensity can be used
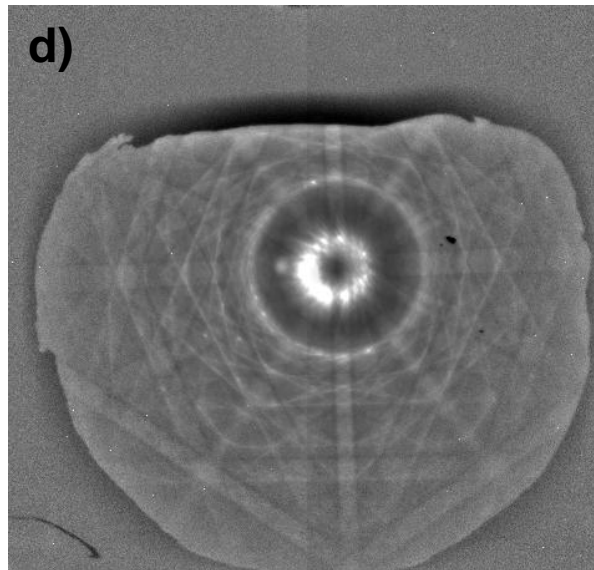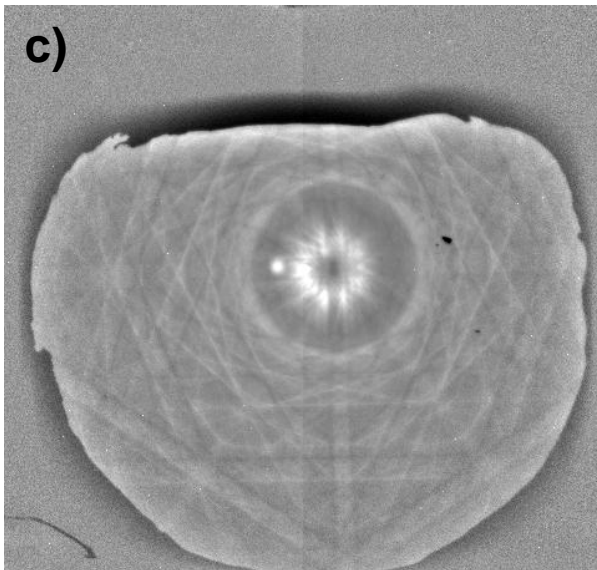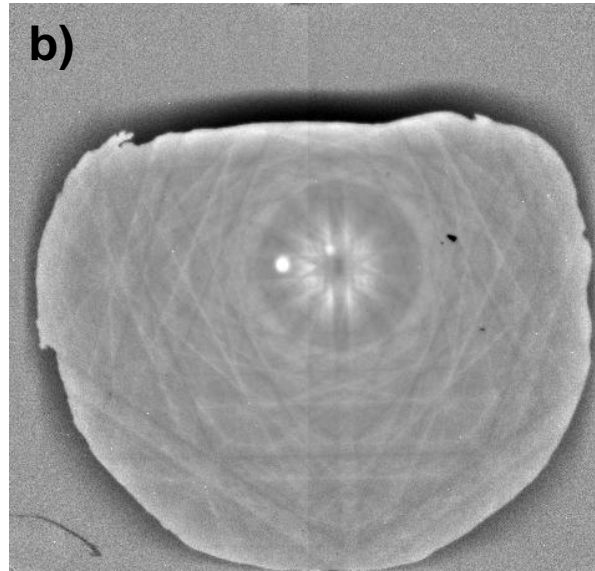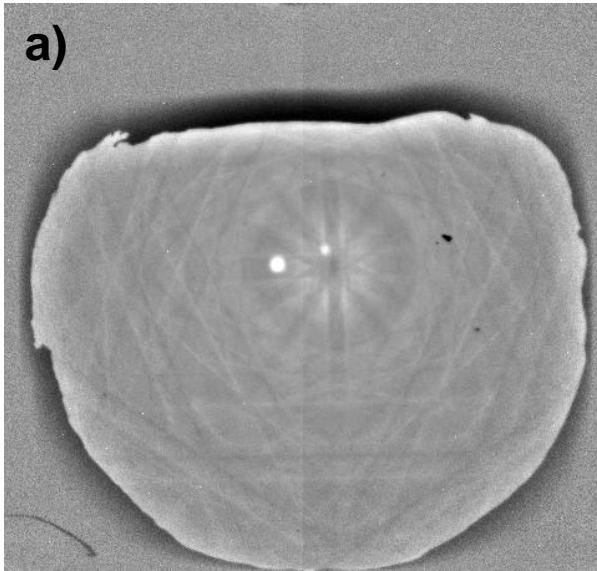
➡ Smaller electron beam size

-TKD on-axis configuration vs. orientation map by TEM: Lower mean free path for TDS at low electron energy.

➡ Thinner specimen can be used

➡ Smaller electron beam broadening in the specimen

Kikuchi pattern
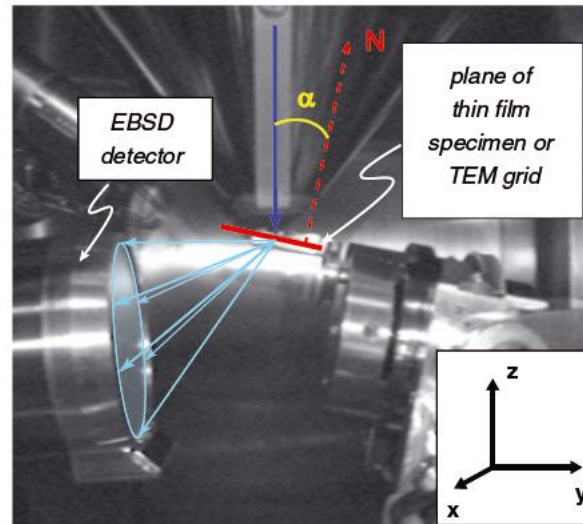
Inelastic diffusion
+
Elastic diffusion

⇔ **SEM EBSP**

incident parallel beam

inelastic scattering center

diffracting planes

$2\theta_B$

Back focal plane

Kikuchi lines

✓ **Contrast = f (thickness of sample)**

Si: 20 kV, **2.7 nA**

plane of thin film specimen or TEM grid

EBSD detector

Low kV EBSD mode Orientation map

Si thin film: Pattern Quality+IPFZMap
Px: 30 nmMapSize: 337 x 253
2 µm

TKD mode Orientation map

Si thin film: Pattern Quality+IPFZMap
Px: 11 nm MapSize: 600 x 450
1 µm

Pas : 30nm

Pas : 11nm