Developments and applications in Transmission Kikuchi Diffraction (TKD)

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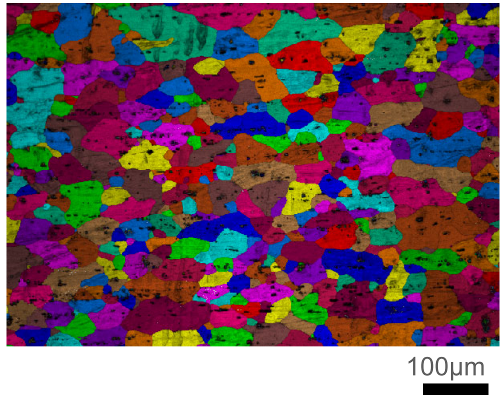
Motivation

1. Current devolopementsin the sub-micro and nano-scale enginerring require new or improved analytical techniques with better spatial resolution.
2. In our case, an improved reslution was needed for grain size and texture mapping of newly developed light alloys (Al and Mg based).

Hot forged 1.5kg towbar example made from sub-micron grain size aluminium alloy

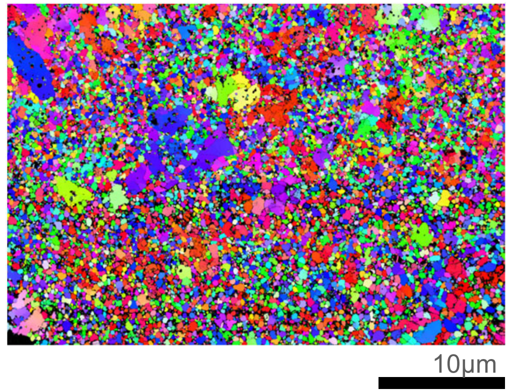
Industrial
Al alloy

Average grain size
50 µm



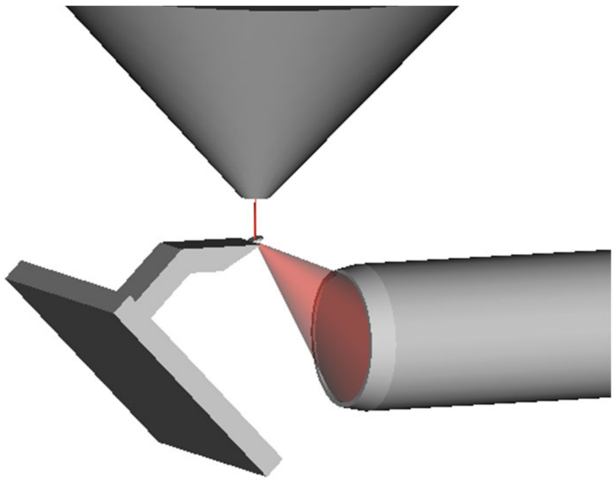
Newly deveolped
Al alloy

Average grain size
700 nm



Introduction

1. The TKD is already mature measurement technique employing the same post processing software as EBSD.
2. Any crystalline object with correct thickness (int the range of 50nm to ~500nm) can be used. Examples includes: metals and alloys, ceramic and geological samples, nano-particles, samples for APT
3. Requirements:
   - FEG-SEM
   - Thin-smaple
   - Holder



The goal of the presentation is to show you that TKD can be used as **effectively** as a standard/reflection EBSD.

Topics concerning acqusistion geometry, signal intensity, holder design and spatial resolution will be addresed in the talk.

t-EBSD - Keller,Geiss 2012; TKD - Trimby 2012

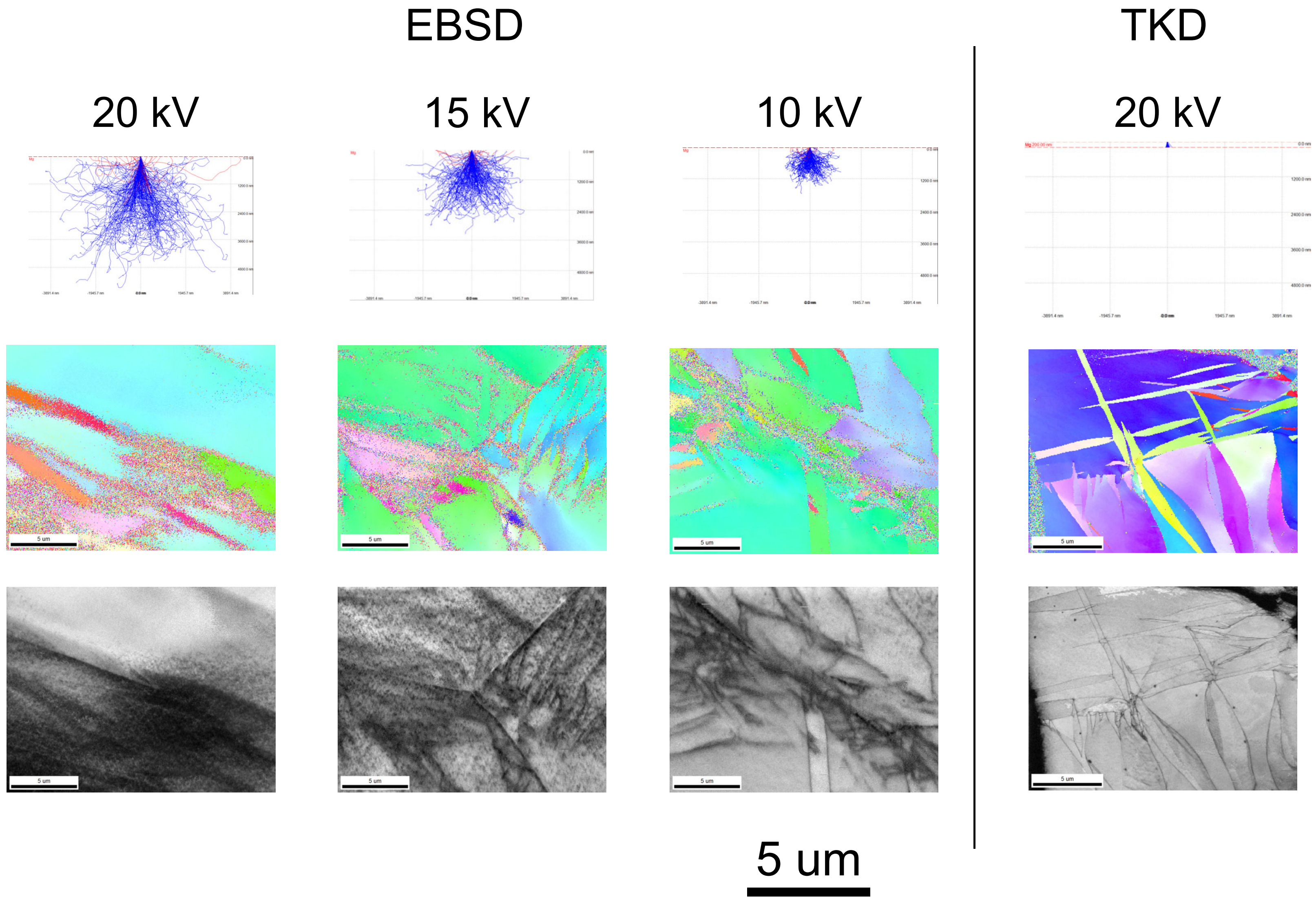
EBSD and TKD spatial resolution example

Lighter (lower Z) samples larger electron-matter interaction volume

Common solution is to use lower kV.

An example is showing defromed Mg sample where high number of twins with sub-micron thickness.

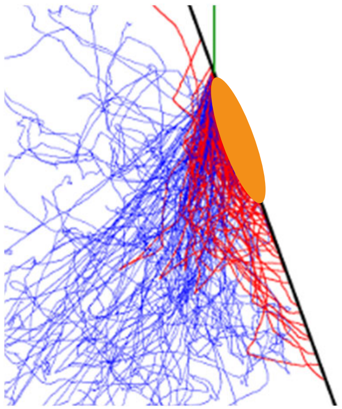
Map step size: 50 nm



EBSD signal source

Broad field of view: many reflectors are mapped simultaneously on the detector screen.

FEG-SEM instruments are capable of focusing electron beam down to 1nm in diameter, thus the only limiting factor is the matter interaction volume.

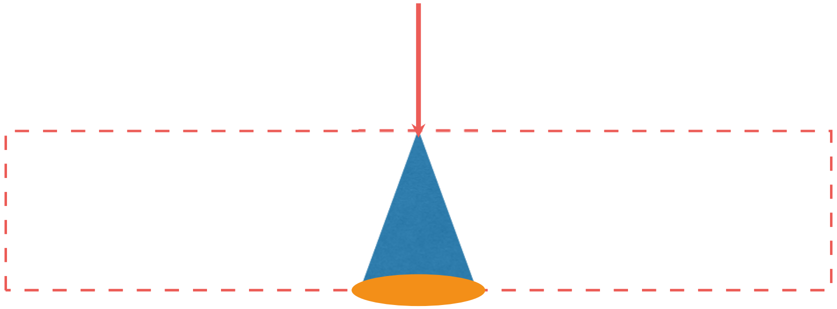


Absorbed electrons
Back-scattered electrons

Area of the usefull diffraction signal is much smaller than interaction volume
Spatial resolution is anisotropic due to the sample inclination

EBSD resolution limits:
Heavy elements ~ 50 nm
Light elements ~100 nm

TKD signal source



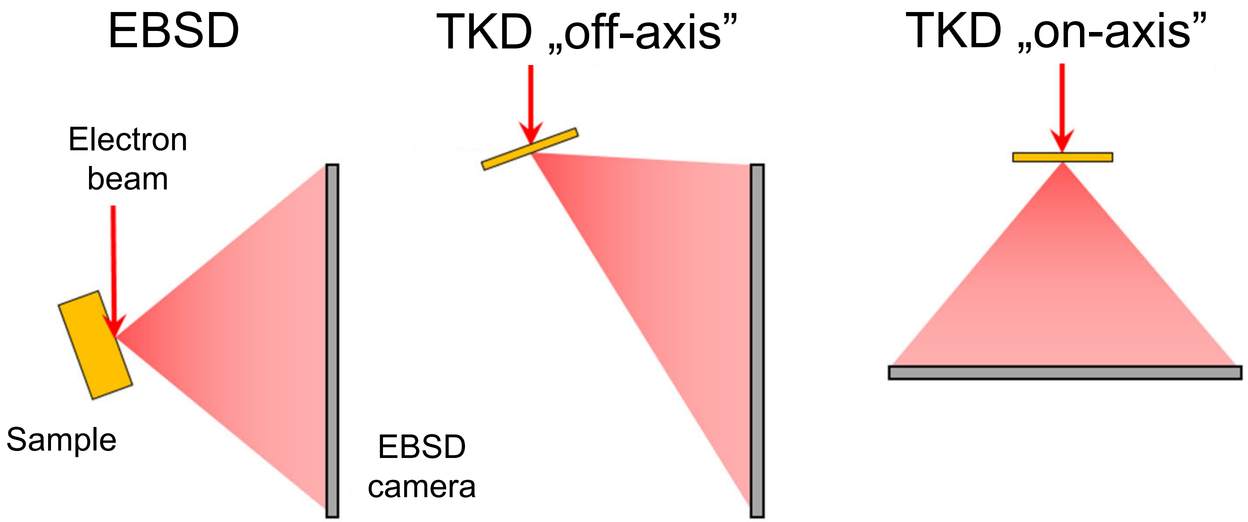
Electron interaction volume is reduced leading to the improved spatial resolution

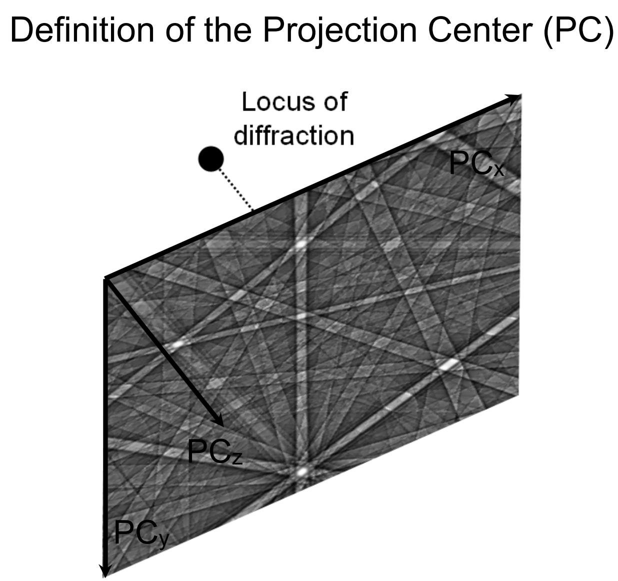
The source of diffraction is located at the bottom surface of the thin foil.
Spatial resolution anisoptropy is present in the case of “off-axis” TKD.

TKD resolution limits are in the range of 10 to 20 nm

Geometry

TKD configuration





EBSD projection center:
PCx usually around 0.5
PCy in the range of 0.2 to 0.7
PCz in the range of 0.5 to 0.9

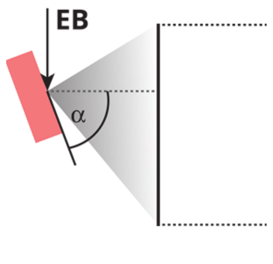
TKD projection center:
PCx usually around 0.5
PCy is negative
PCz in the range of 0.5 to 0.9

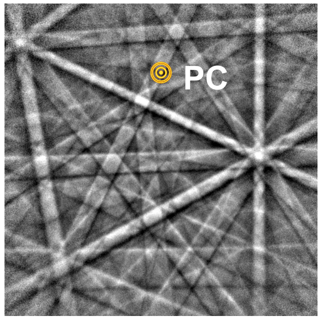
The effects of projection center on registered signal

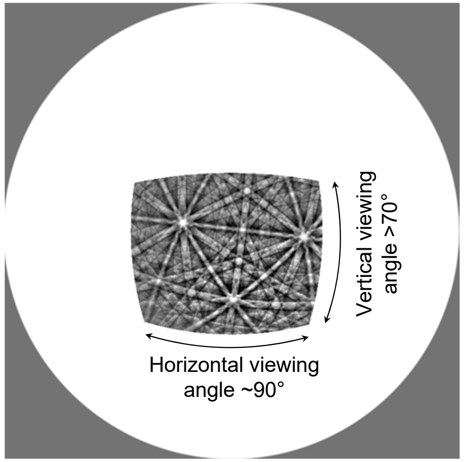
Stereographic projection

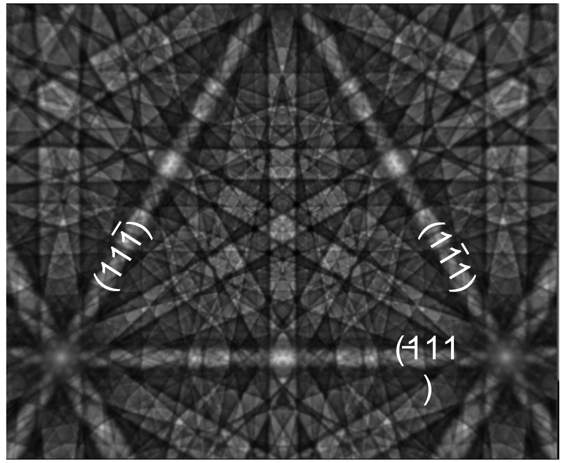
Gnomonic projection

EBSD





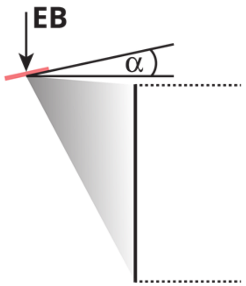


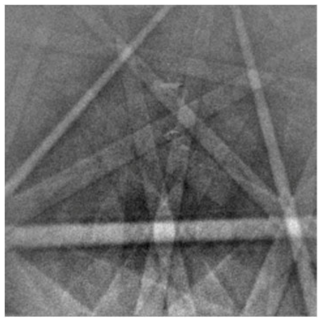


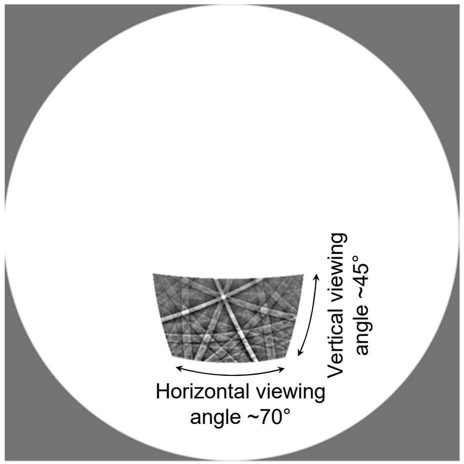
PCx=0.50; PCy=0.50; PCz=0.80

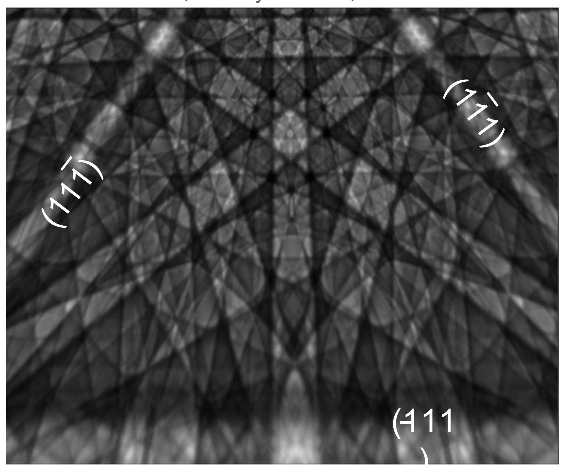
TKD

PC





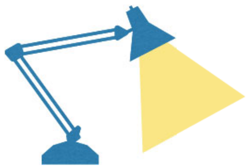




PCx=0.50; PCy=-0.40; PCz=0.80

TKD

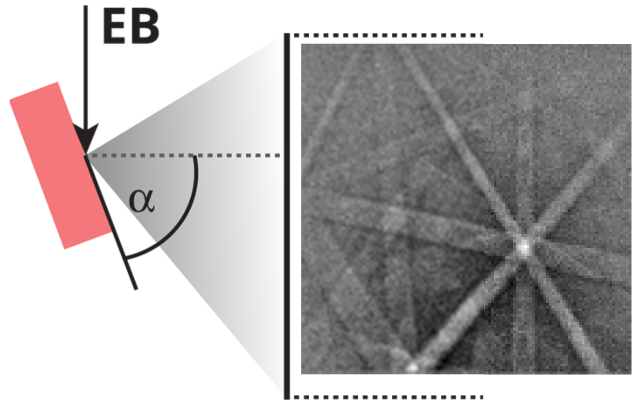
- Smaller viewing angle
- High gnomonic distortions



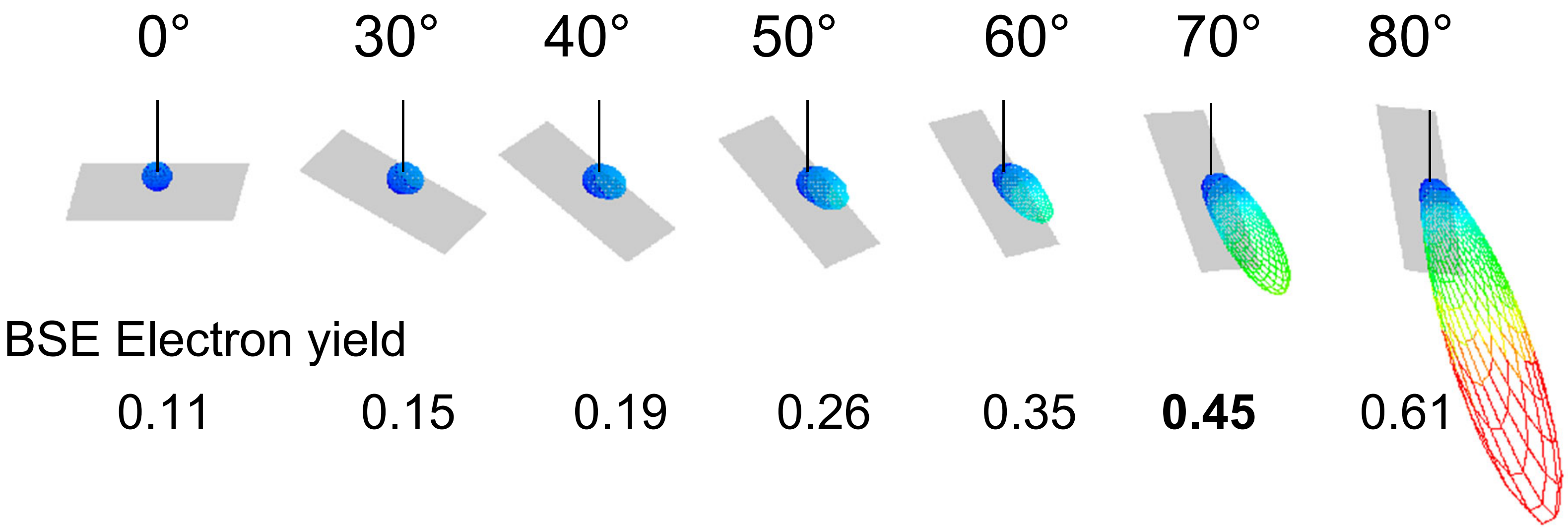
Signal intensity

Sample inclination angle - the best camera performance

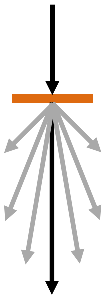
- Angular distribution of electrons changes with the sample inclination
- Optimum inclination is **60° to 70°**
- Electron yield (amount of electrons backscattered) increases with the sample inclination



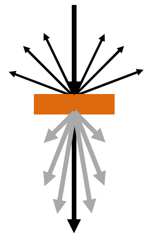
Monte Carlo simulation of back scattered electrons, Mg example.

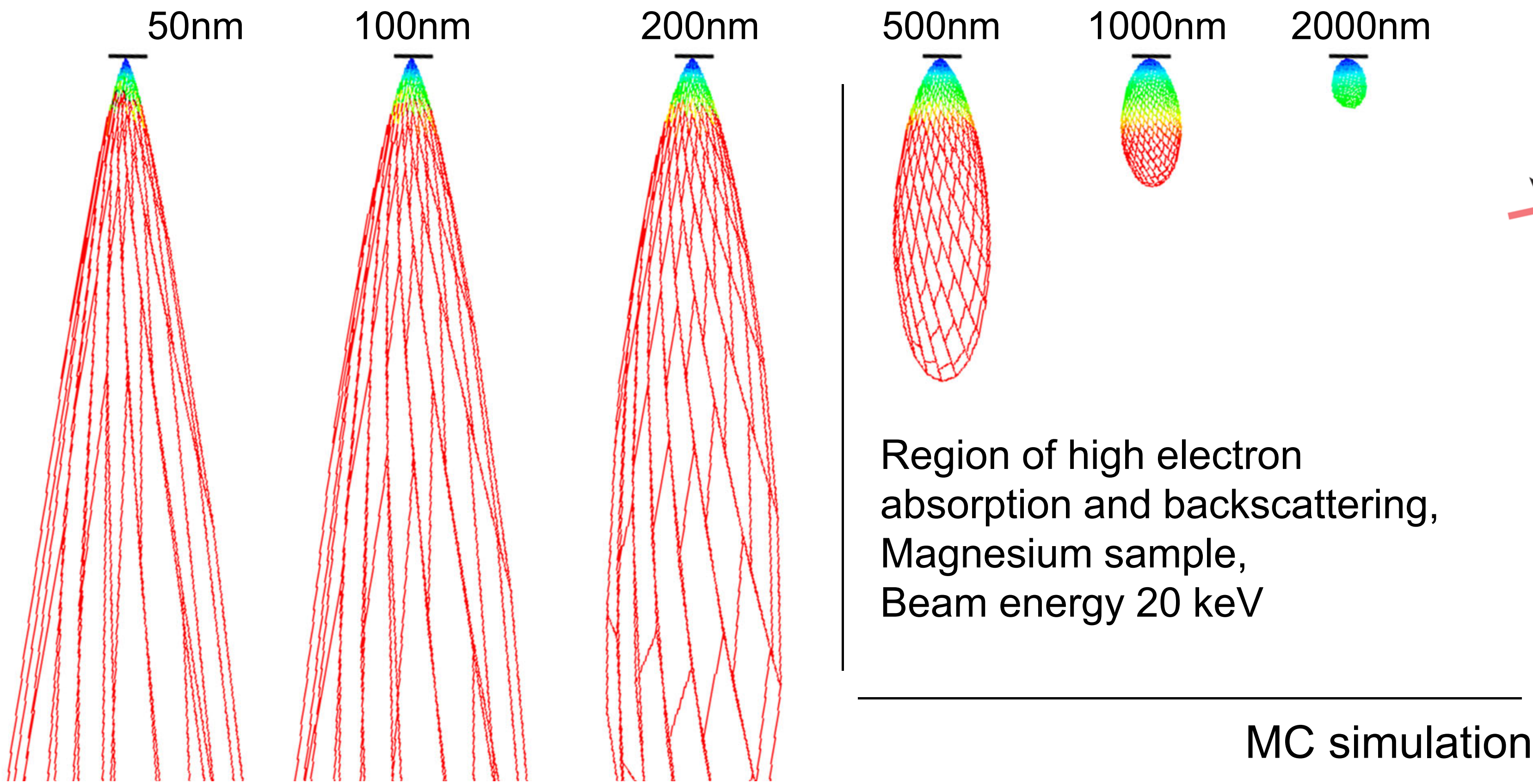


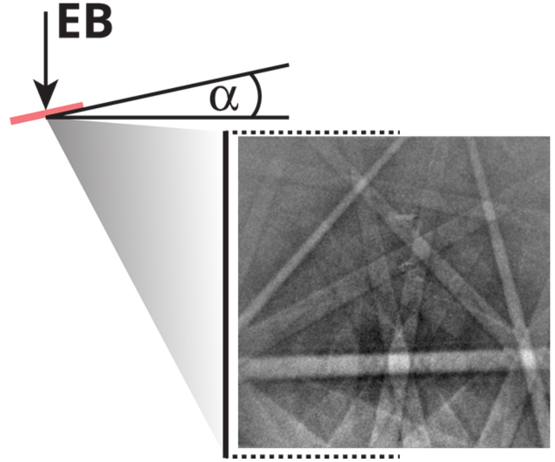
TKD Specimen thickness - electron distribution



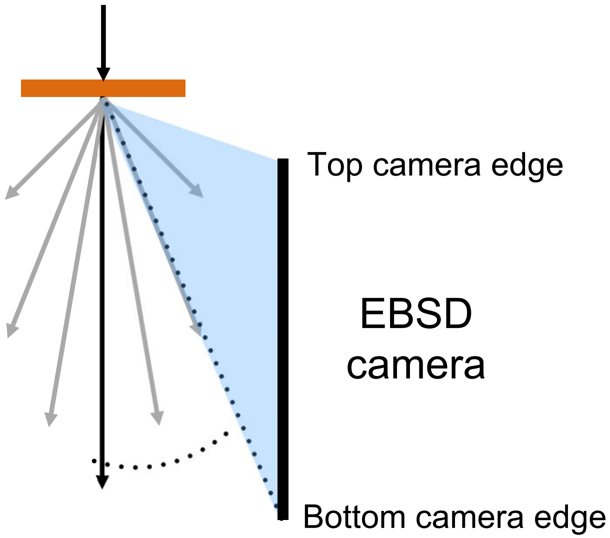
- Angular distribution of transmitted electrons electrons changes with the sample thickness
- Electron yield (amount of electrons backscattered) increases with inclination

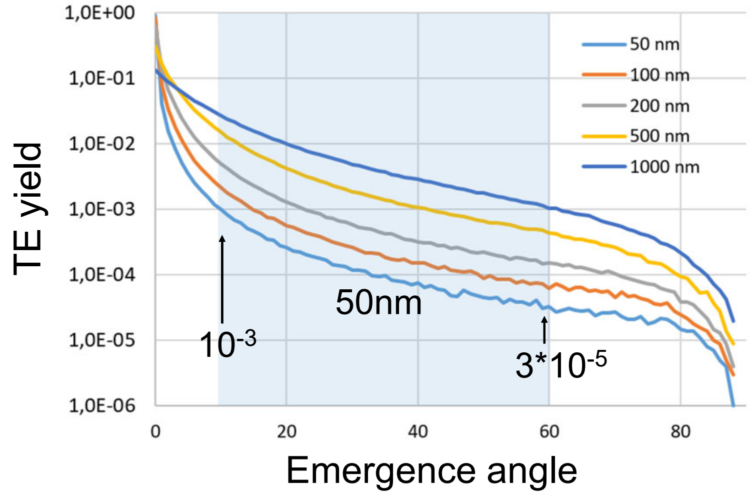


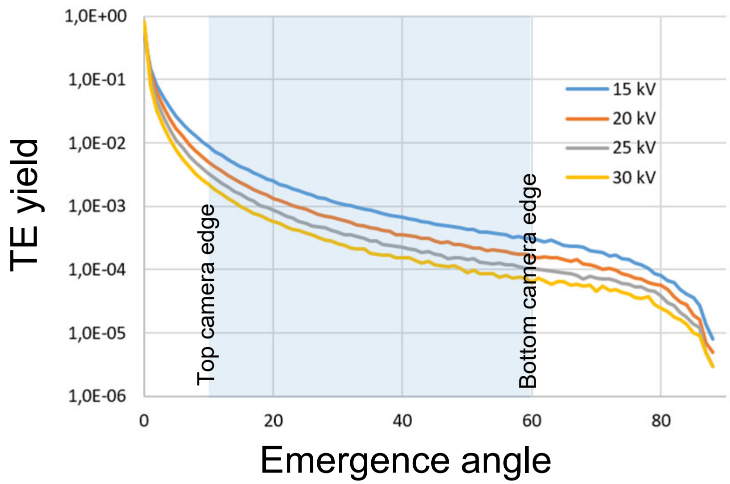




Electron yield in the EBSD camera, Mg sample



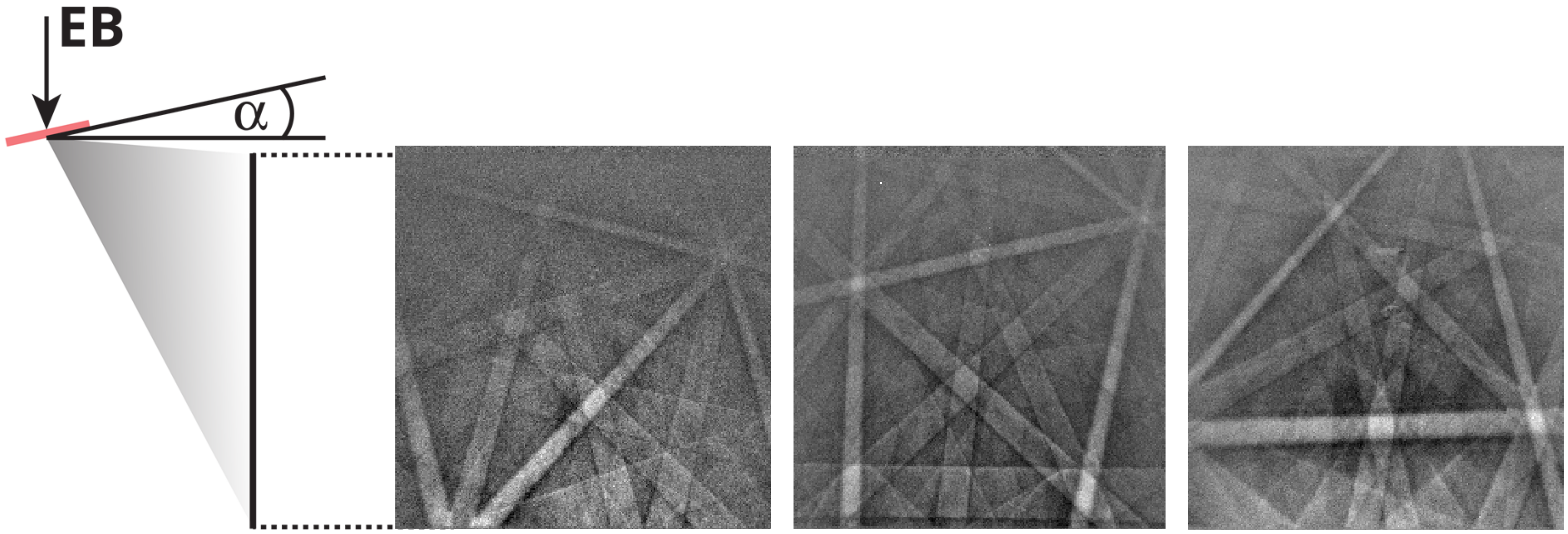




- At higher sample thickness absorption and backscattering effect are becoming significant
- Thinnest sample region offers best spatial resolution but at the same time intensity of the patterns registered in the camera is lowest

Electron yield in camera is very small. For a typical imaging conditions of **20 - 30 keV** and **50 - 300 nm** specimen thickness is in the range of **0.01 to 0.1**

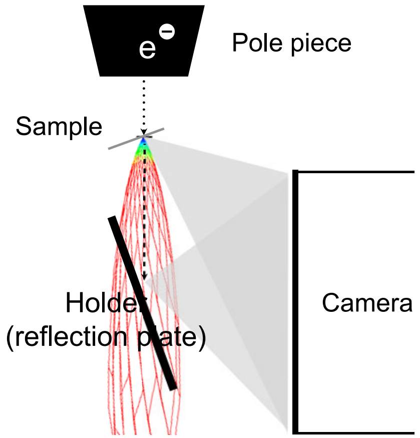
Effect of thin foil inclination

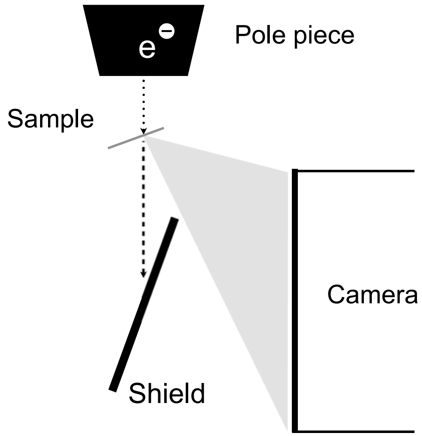


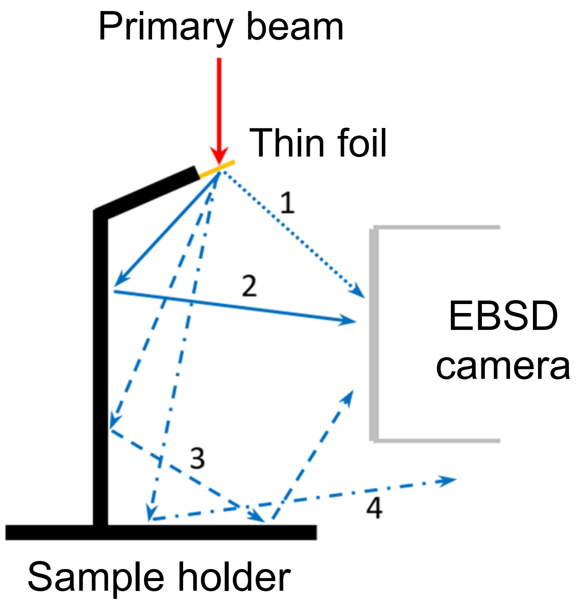
- Depending on microscope setup, inclination may be required to prevent shadowing effect
- Foil is inclined at negative angles with respect to standard EBSD
- Foil inclination slightly increase electron yield in the camera

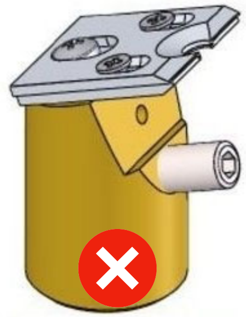
Holder with a shield

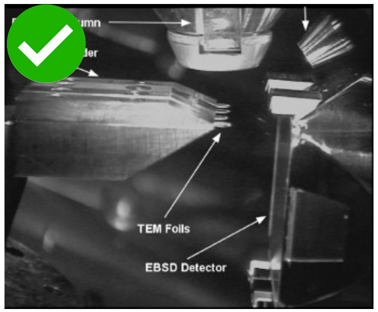
The idea of shielded holder - do we need shielding?

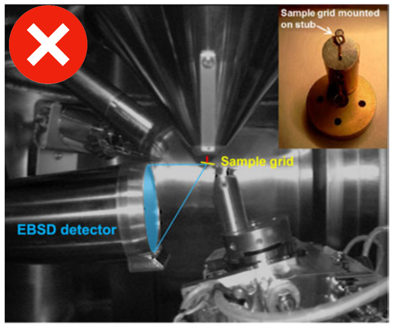


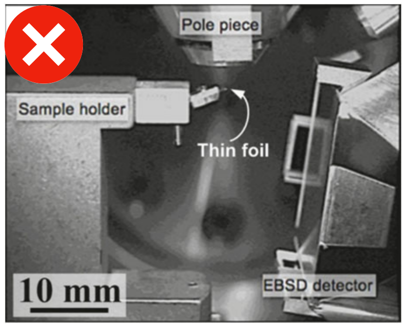


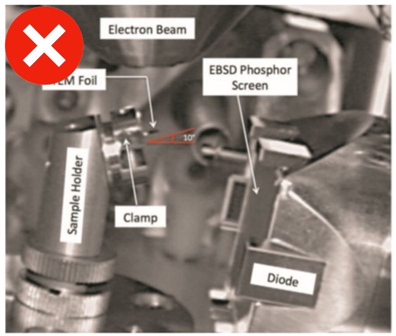


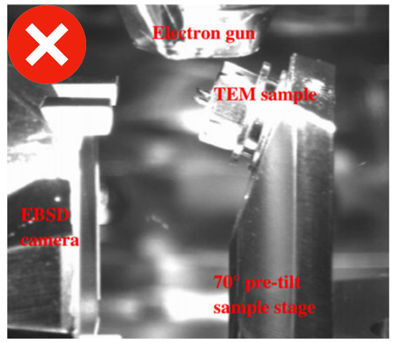






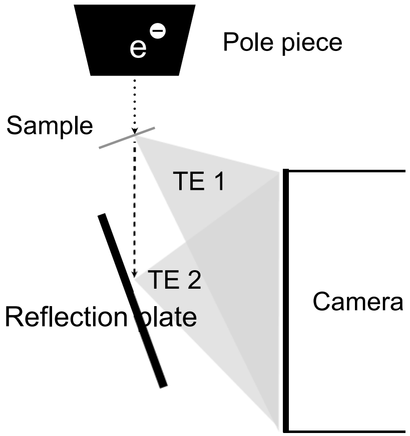


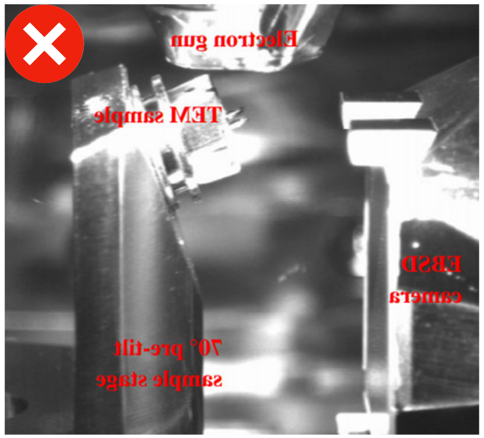




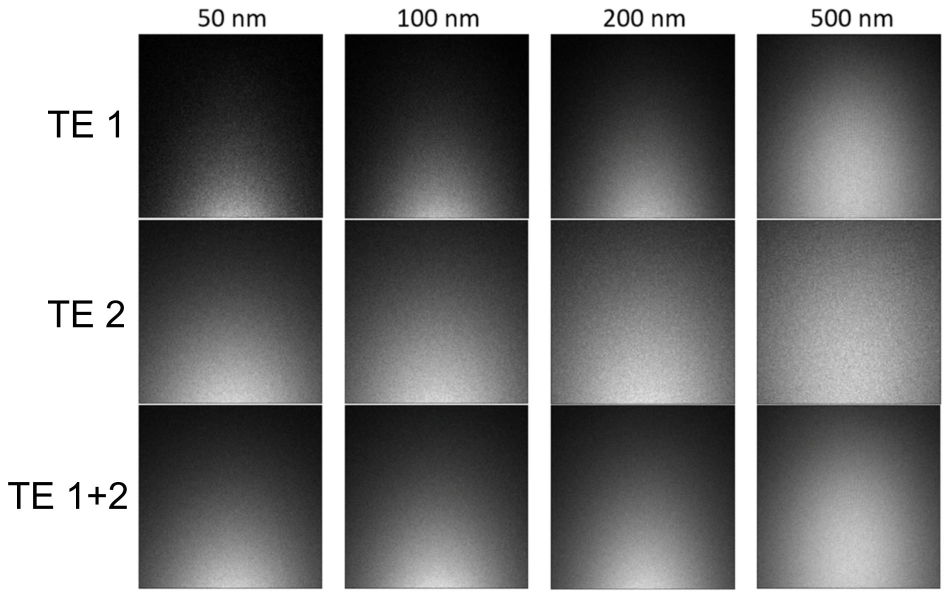
Examples from literature

Parasitic electrons intensity





Monte Carlo simulations, Mg thin foil

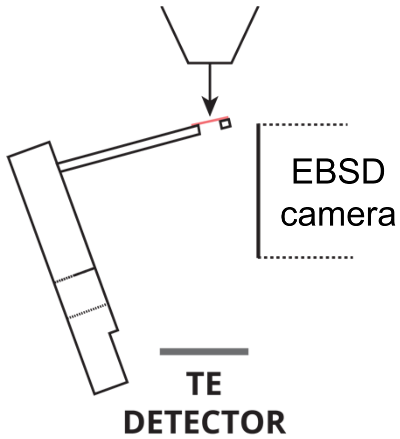


Worst case scenario!!!

Foil thickness, nm	50	100	200	500
	Fraction of electrons in the camera			
TE 1	0,011	0,022	0,049	0,110
TE 2	0,058	0,058	0,058	0,045
Parasitic interference, %	84,1	72,5	54,2	29,0

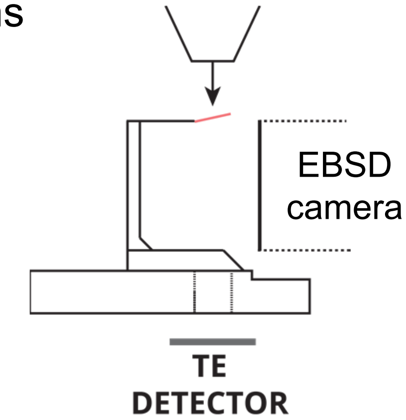
TKD holder example

Tilted stage

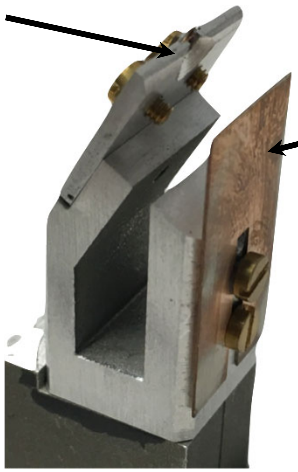


All possible configurations
with pretilted holder

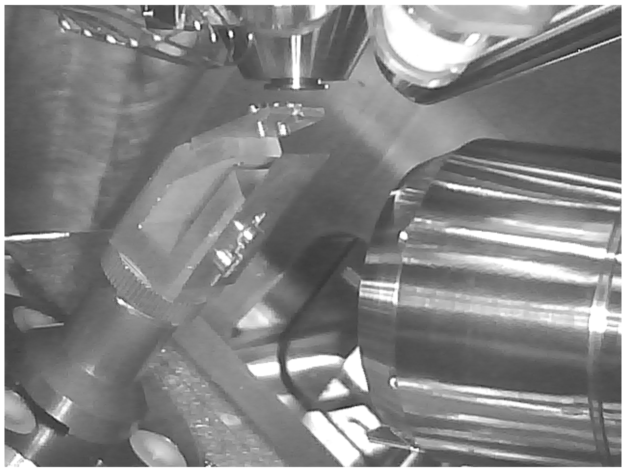
Horizontal stage



Thin foil



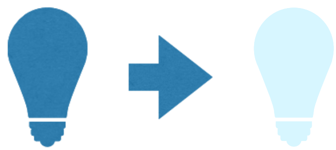
Shield



Holder is pretilted to 60°

Stage inclination 40°

Total sample inclination 20°



Low intensity imaging

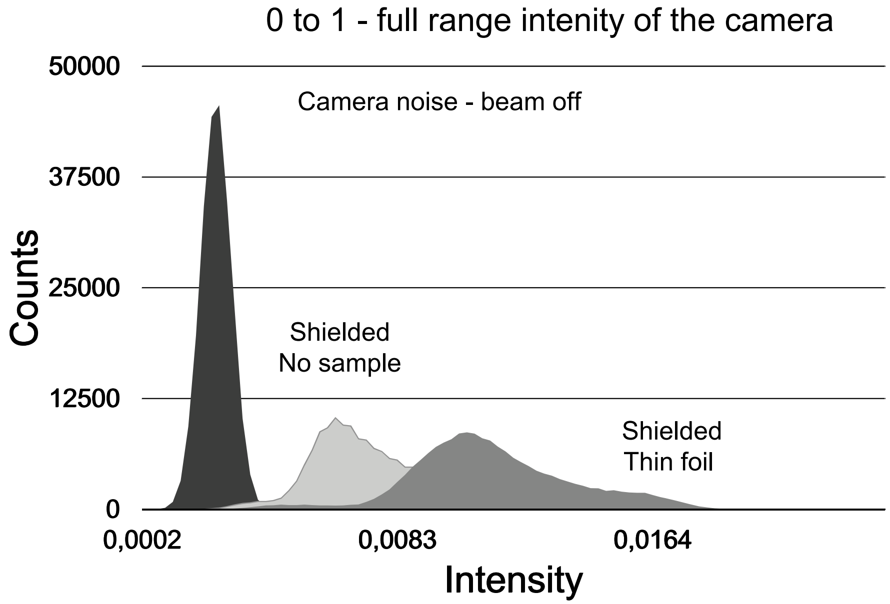
Low intensity TKD imaging with shield

Acquisition parameters:

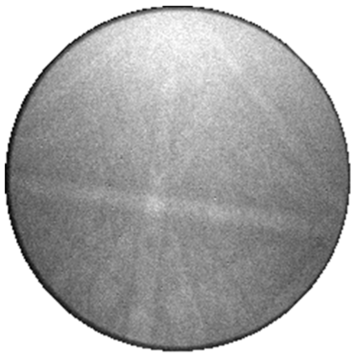
- 480x480 - no binning
- Gain, 0 (zero)
- Integration time, 100 ms
- Primary beam energy, 30 keV
- Beam current, 16 nA
- EDAX, Hikari camera

Fraction of the full dynamic range of the camera:

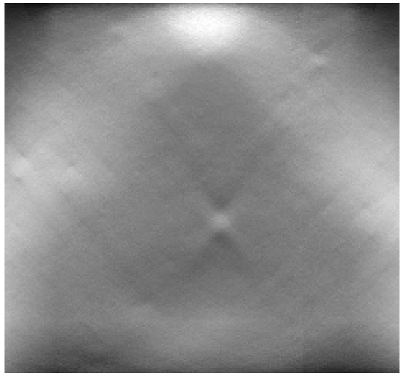
- Camera noise: 0.2%
- Hole/shielded signal: ~1%
- Low intensity pattern: ~2%
- Usually we are runing above 10%

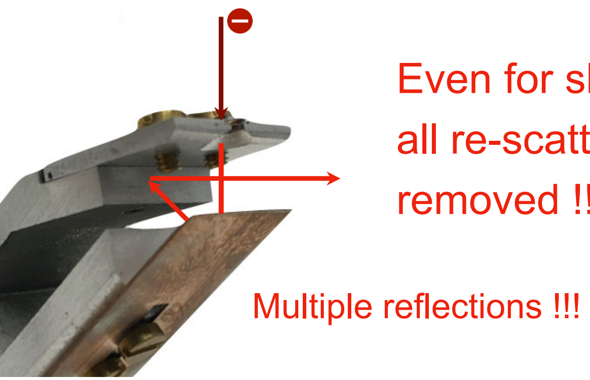


Pattern
(histogram stretched)



Hough transformation





Even for shielded configuration not all re-scattered electrons are removed !!!

How fast can we go?

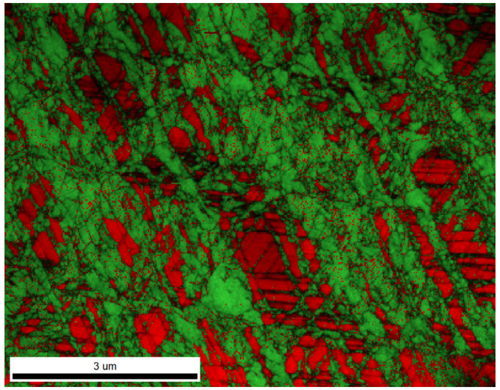
Highly deformed (0.6 true strain) stainless steel sample AISI 304

- Electron energy: 30 keV
- Beam current: 16 nA
- Step size: 20 nm
- Saturation of the camera: 20%
- Raw data (no map cleaning)

Camera parameters

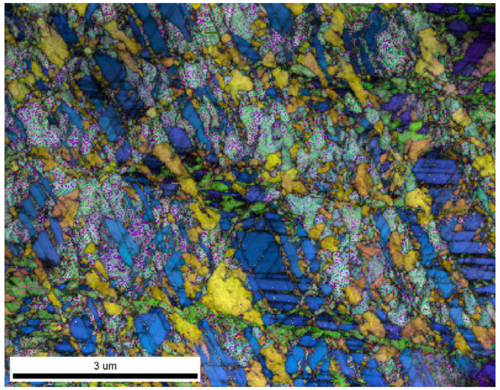
Camera binning (pattern resolution)	Camera speed, PPS	Integration time, ms	Average CI
4x4 (120x120)	100	10	0.21
4x4 (120x120)	200 (max)	5	0.13
6x6 (80x80)	300 (max)	3.3	0.12
8x8 (60x60)	360 (max)	2.5	0.12
10x10 (48x48)	450 (max)	2	0.13

Phase map
Austenite
Martensite

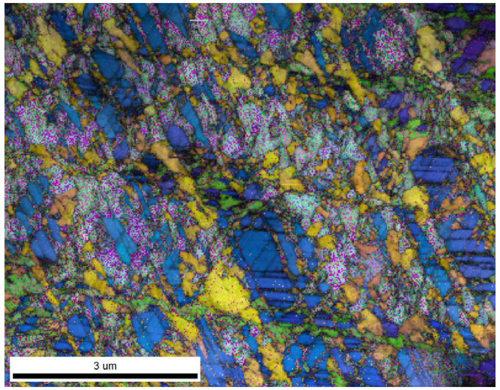


Same sample area, different speed and binning, IPF map

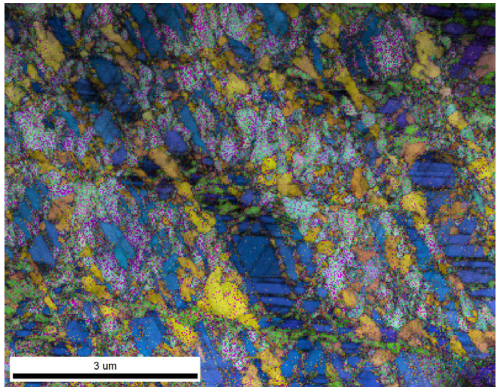
100 pps (4x4)



300 pps (6x6)

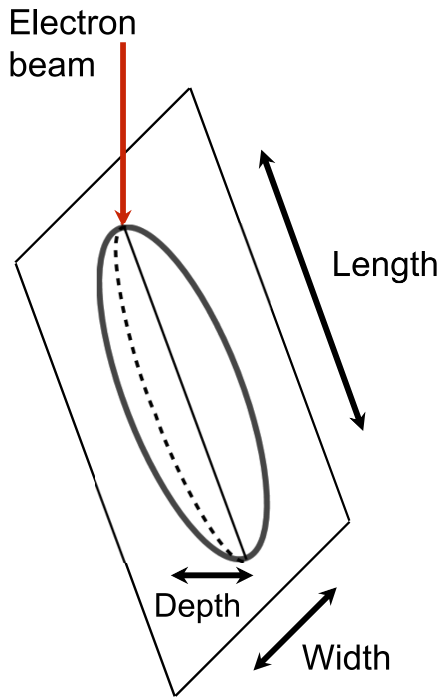


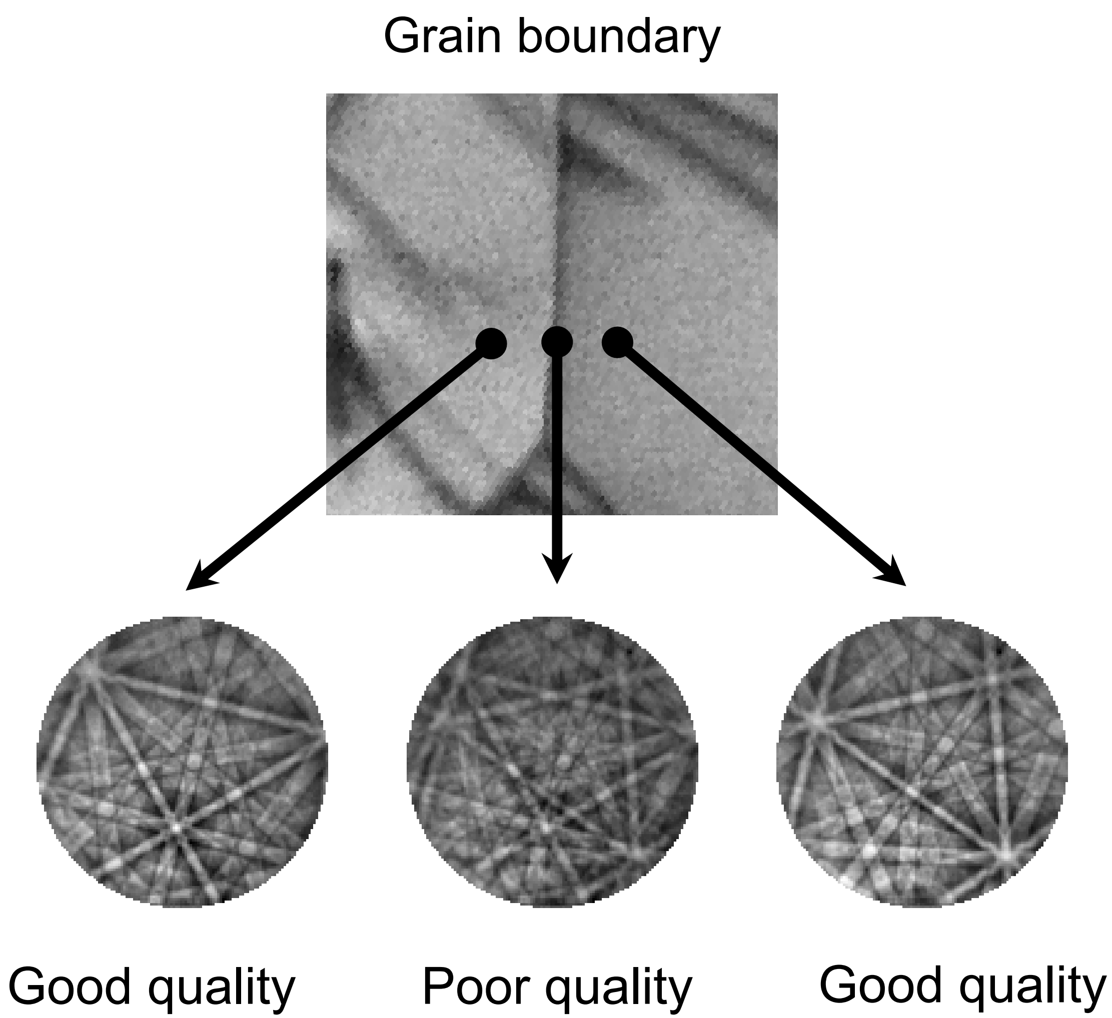
450 pps (48x48)



Spatial resolution

Reslolution limits



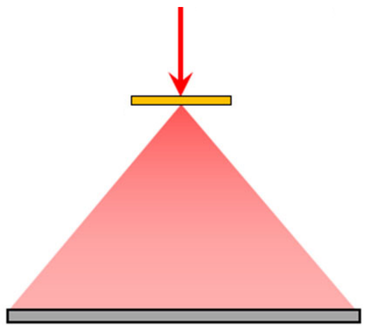


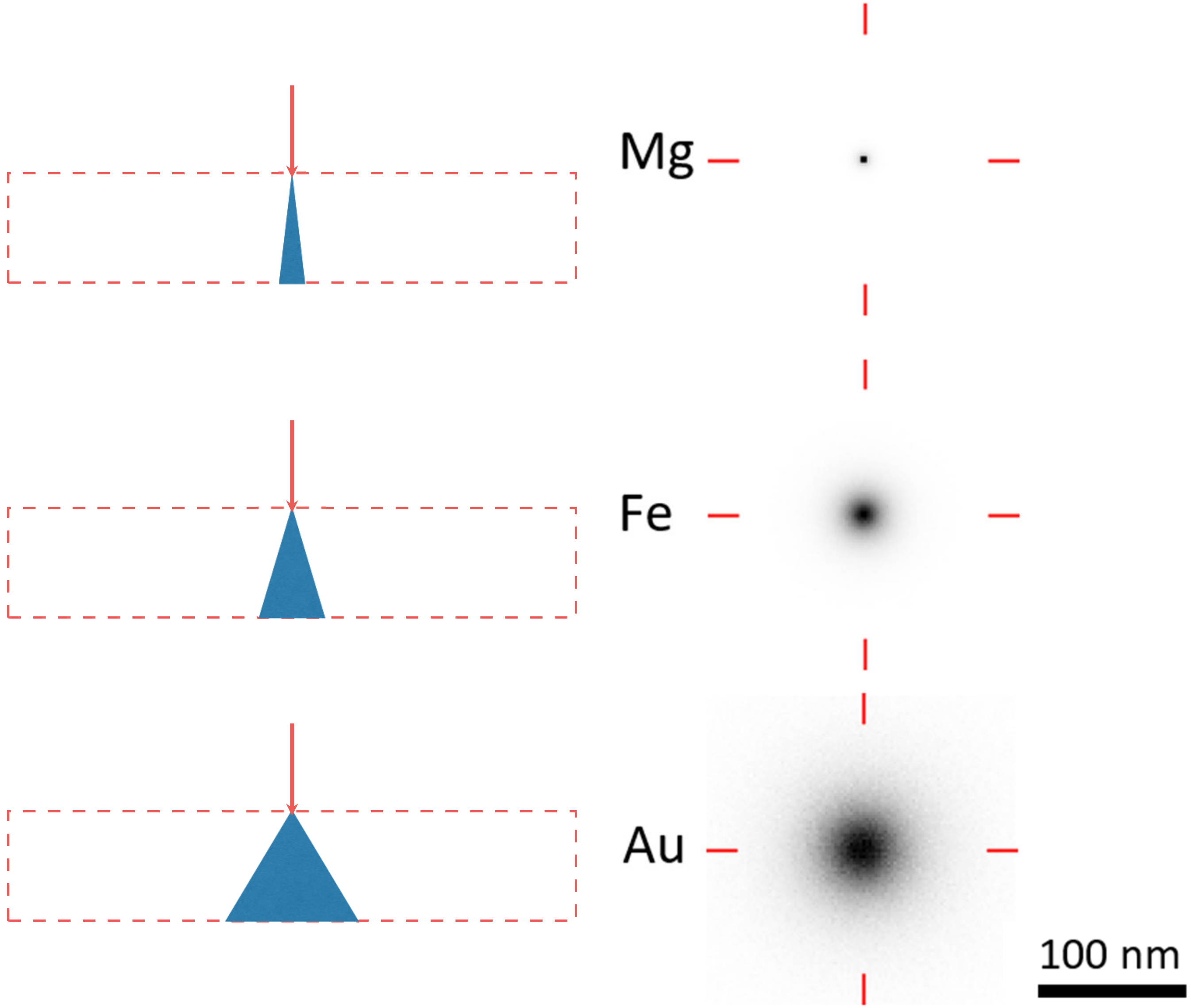
Diffraction quality is substantially deteriorated in the presence of microstructure defects

Distribution of all transmitted electrons on the exit surface of thin foil

- Diffraction signal originates from the bottom of the foil surface
- Electron sprad depend on the kV and the average sample atomic mass
- If the camera is positioned directly under thin foil symmetry of the measurement setup is not broken and there is only one parameter defining spatial reolution

TKD „on-axis"

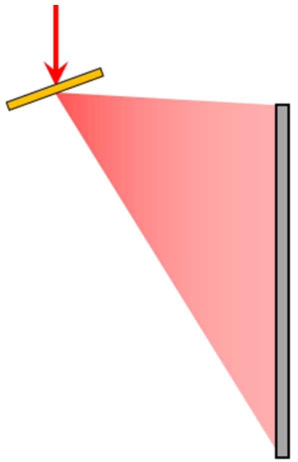


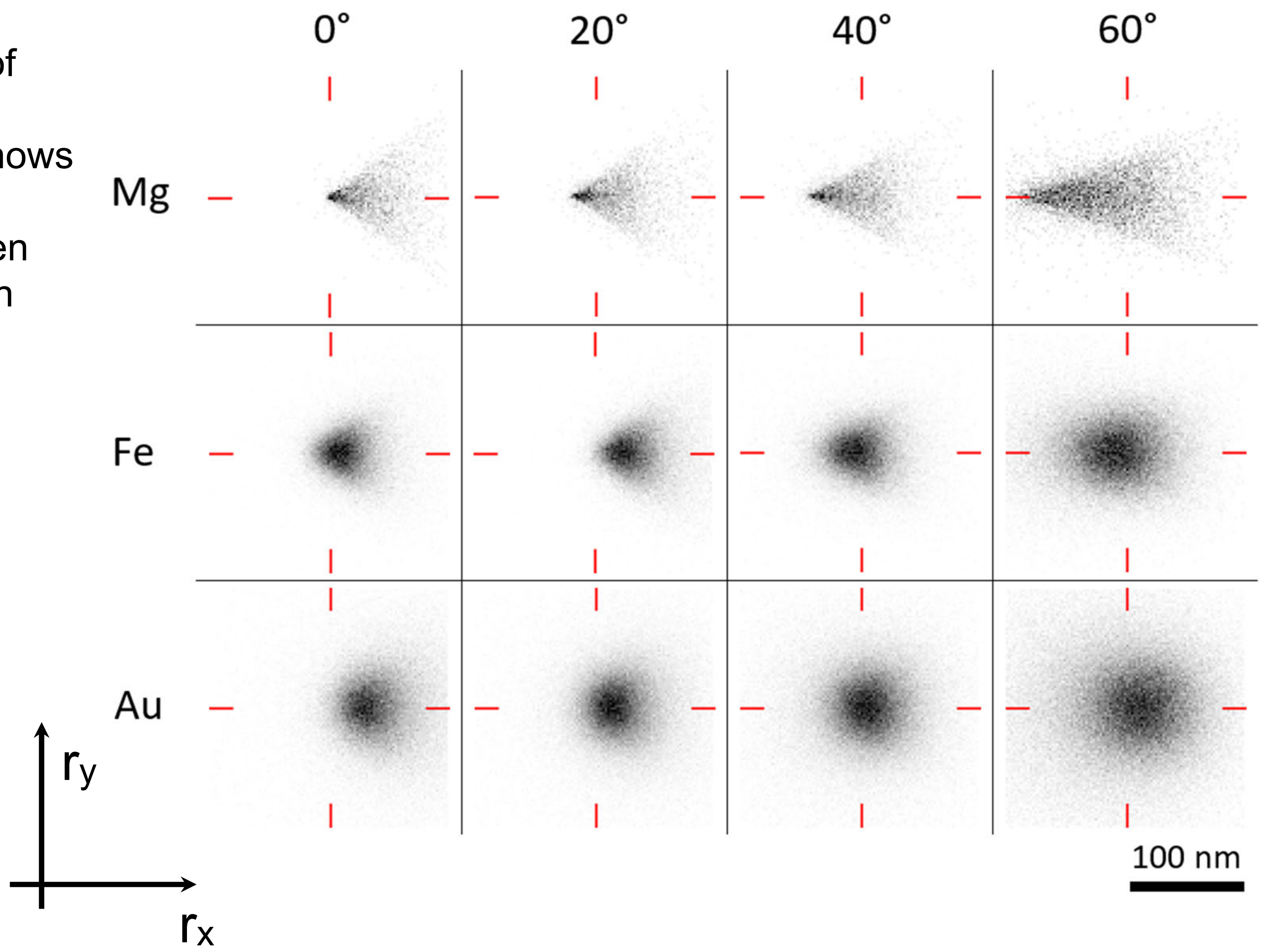


Distribution of detected electrons on the exit surface of thin foil

- Camera is positioned on the side of the thin foil
- Distribution of detected electron shows characteristc “comet tail”
- Sample-camera symmetry is broken leading to the two spatial resolution parameters

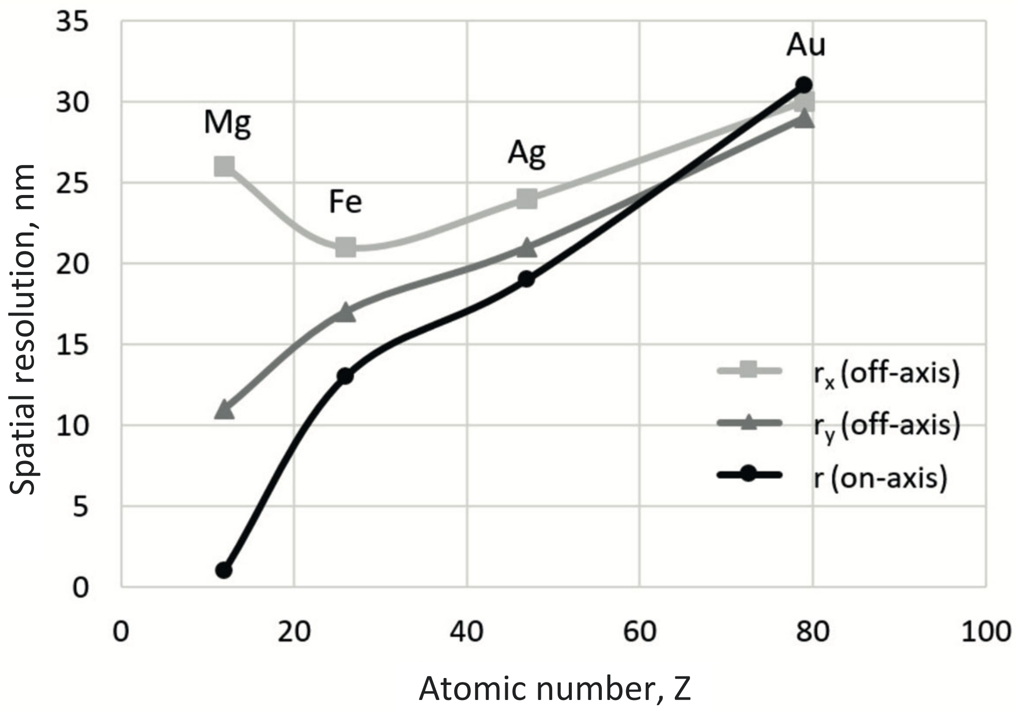
TKD „off-axis”

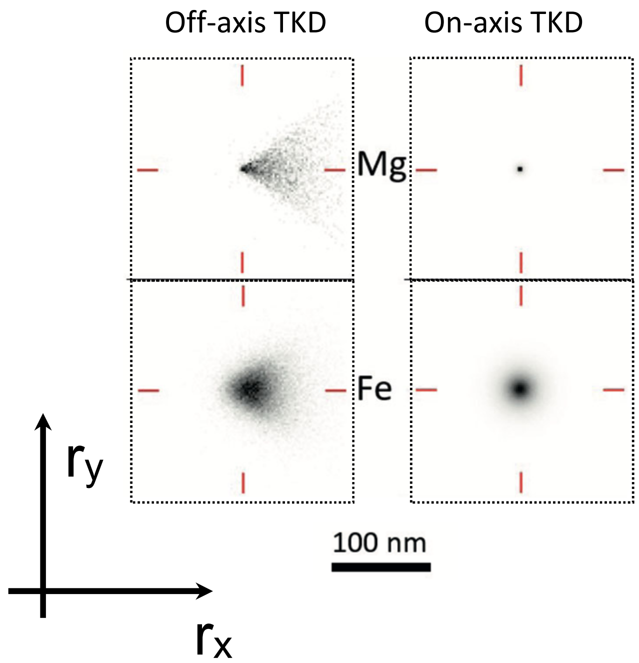




Spatial resolution summary

Calculated resolution parameters are based on the electron distribution on the exit foil surface (MonteCarlo simulation)





- For the on-axis setup only one resolution parameter is defined
- For the off-axis setup two resolution parameters are defined

Possible problems with TKD

Foil bending and carbon contamination

Thin foil bending due to stress relaxation - creation of two free surfaces

Carbon contamination due to insufficient speed and/or too small step size

Formation of **FCC**, **BCC**, **HCP** structures

austenite **γ**, martensite **ε**, martensite **α**

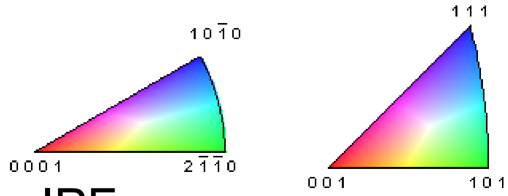
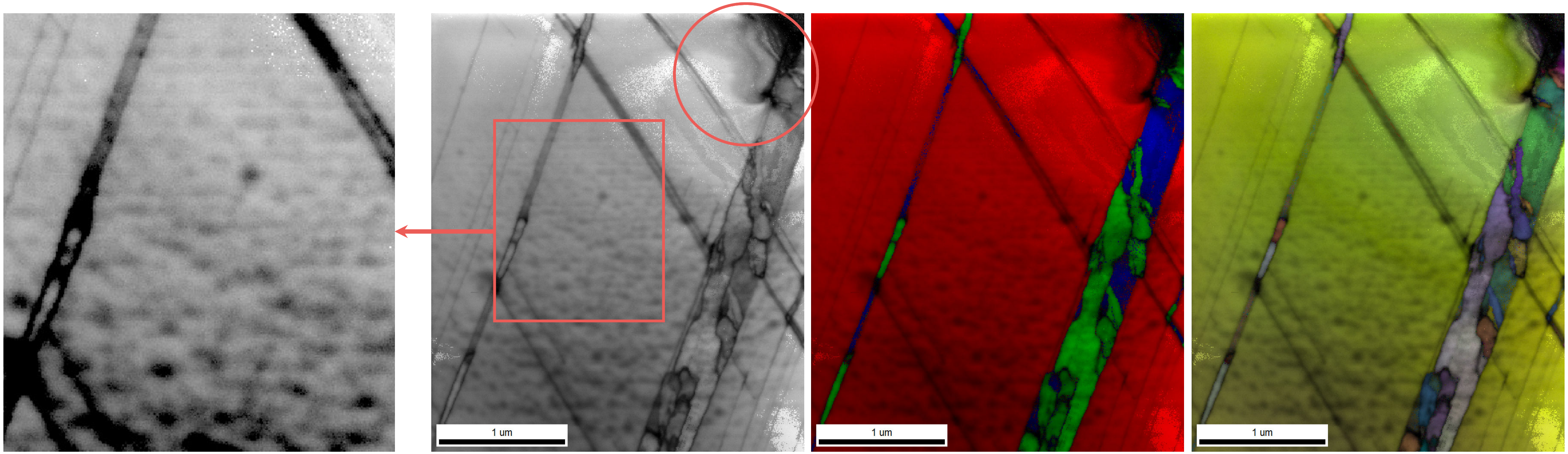


Image Quality map

Phase map

IPF map

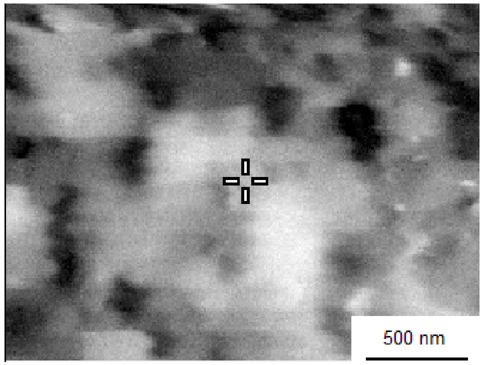


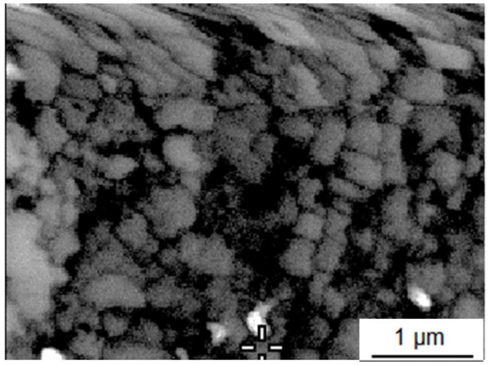
Deformed stainless-steel sample. Conditions: Beam 30kV, 16nA; Step 5nm; 200pps

1 um

Other possible issues

- Mechanical stability of the microscope stage is poor. The SEM stage is designed to handle heavy samples, so high drift is expected. A possible solution is to set the stage in a horizontal position with the proper holder, wait patiently for stage stabilization, or use drift correction if available.
- Electropolishing of the thin foil gives the best results (if it is possible to conduct), however, it creates a wedge sample. In such a case care should be taken to acquire the correct background for compensation.
- FIB sample preparation often creates a significant layer of deformed material on both surfaces. Final polishing on low-kV should be used to remove damaged layers. Alternatively, argon ion polishing can be used as the last thin foil preparation step.
- Thin foil preparation is creating two free surfaces. One should remember that it changes the thermodynamic state of the system and in extreme cases, it can alter material microstructure.





Recommendations

- Prepare good-quality samples. Good sample is 90% of success.
- While using high spatial resolution and collecting large maps use the highest possible mapping speed. Carbon contamination and stage mechanical instability will be reduced.
- If the sample allows use a high current condition of 10nA or more.
- Use a shielded holder to reduce the number of rescattered electrons.
- Set camera intensity to a low level. Raw images in the camera will be dark, thus background correction or histogram equalization is necessary to see Kikuchi bands.
- In the case of the microscope stage having significant backlash, use a horizontal holder with a pre-tilted sample grip section.

If the system is setup correctly you can expect the following:

- Spatial resolution in the range of 10 to 20 nm.
- Acquisition speed within the range of full camera speed for heavy elements.
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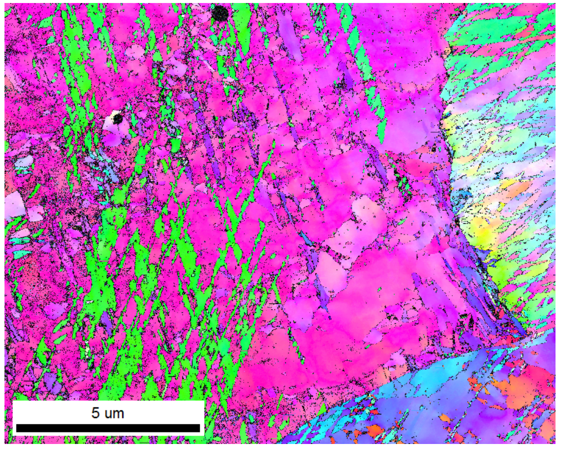

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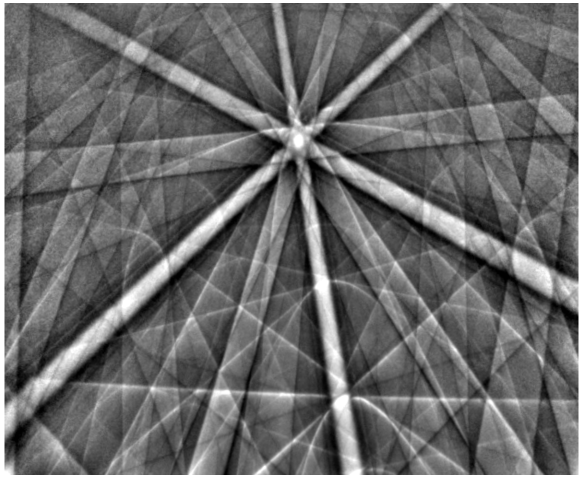

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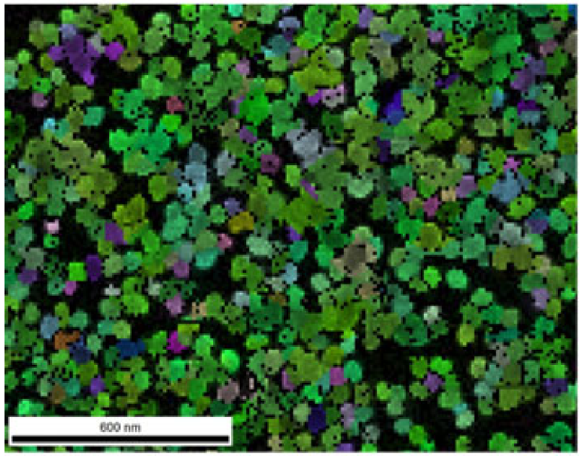

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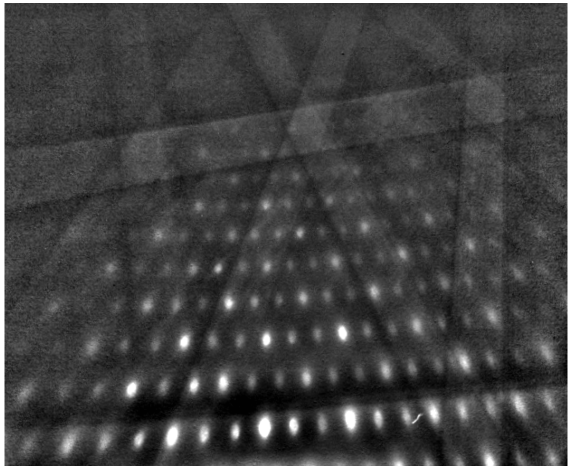

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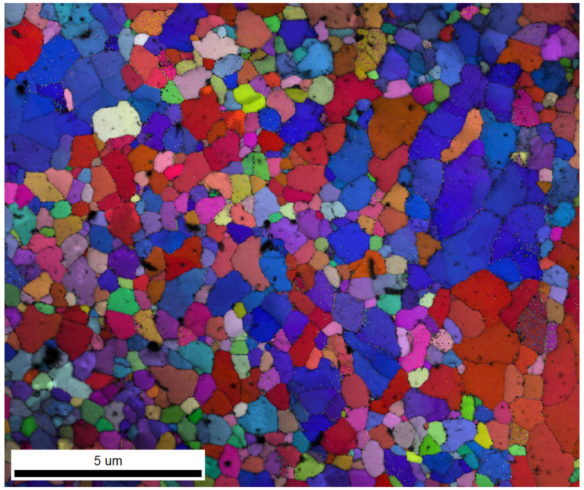

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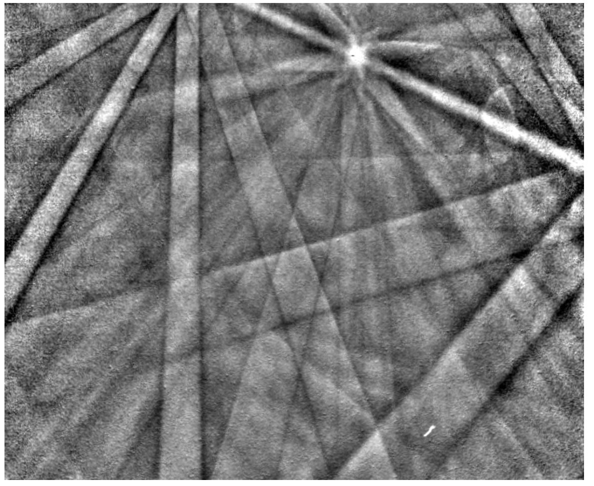

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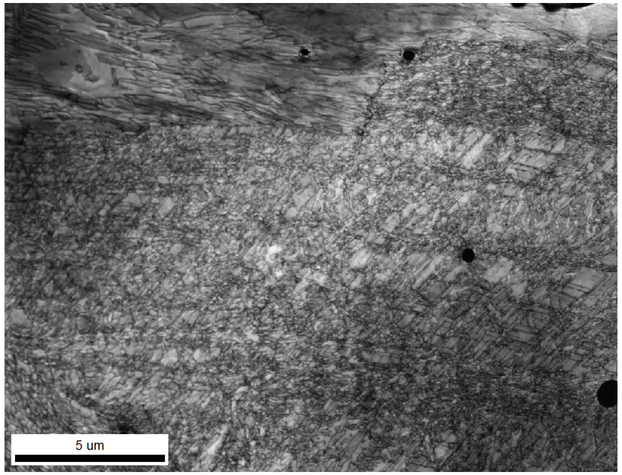

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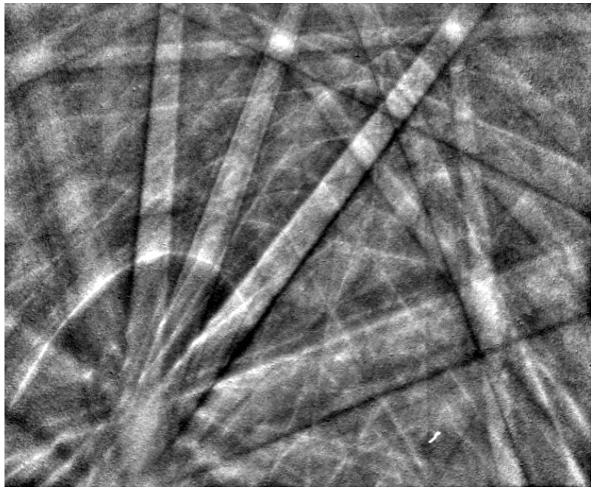

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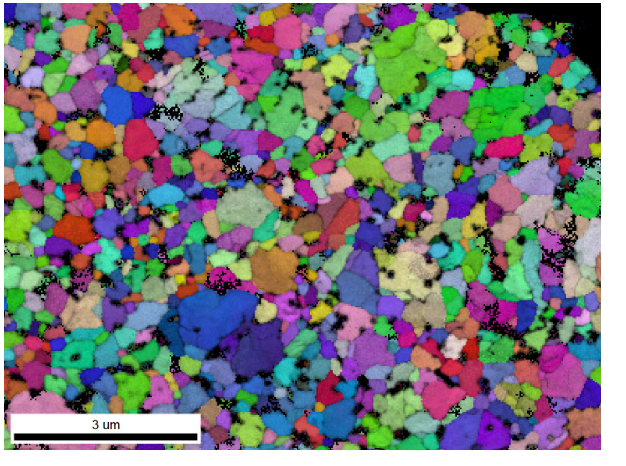

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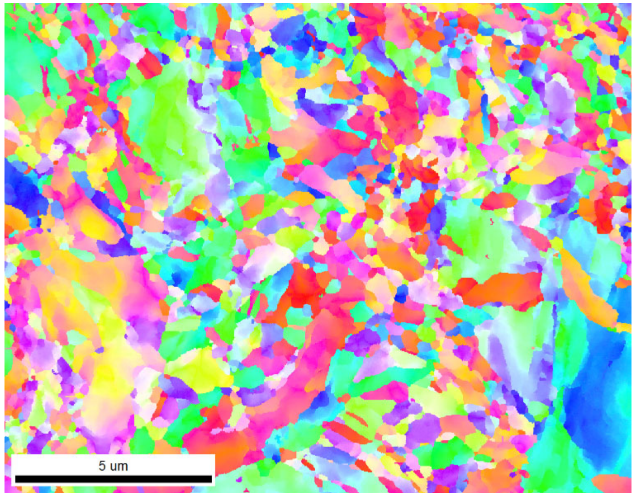

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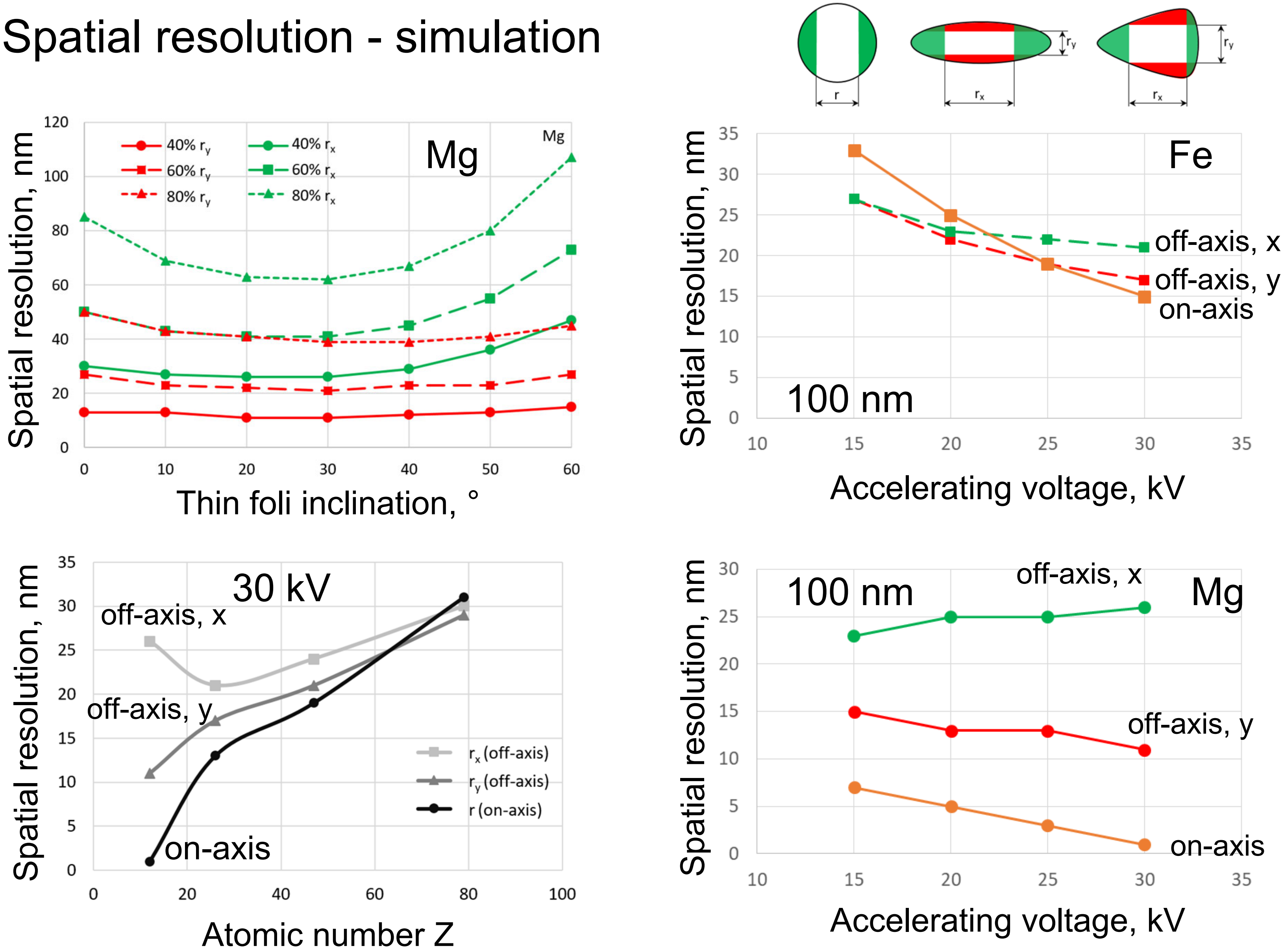
Spatial resolution - simulation



Electron yield in the EBSD camera, Fe sample