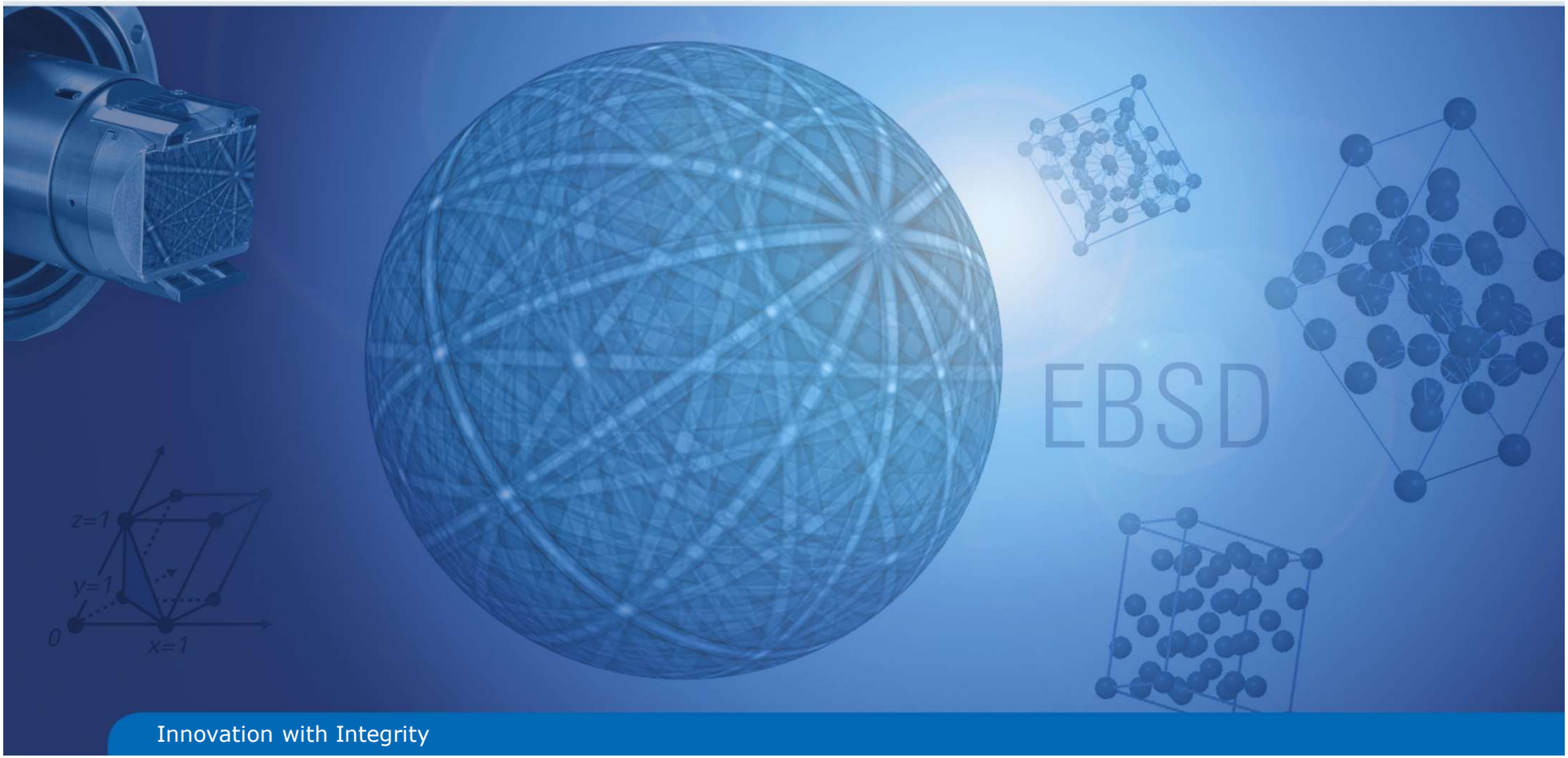
Advanced Material Characterization by Combined 3D EBSD/EDS Measurements and Post Processing with ESPRIT QUBE

Free online webinar – December 7th 2017

Bruker Nano Analytics, Germany



Innovation with Integrity

Materials investigations by 3D EBSD
- Applications of the analysis software QUBE -

S. Zaefferer, P. Konijnenberg

D. An, G. Nayyeri, A. Khorashadizadeh

Contents

- Introduction: why 3D materials investigations and how to do 3D EBSD?
- Determination of geometrically necessary dislocation densities (GNDs) from 3D orientation fields
- 3D EBSD and modelling of recrystallization
- Grain boundary character and properties
- A new feature in QUBE: non-ridgid slice alignment
- Possibilities & Limitations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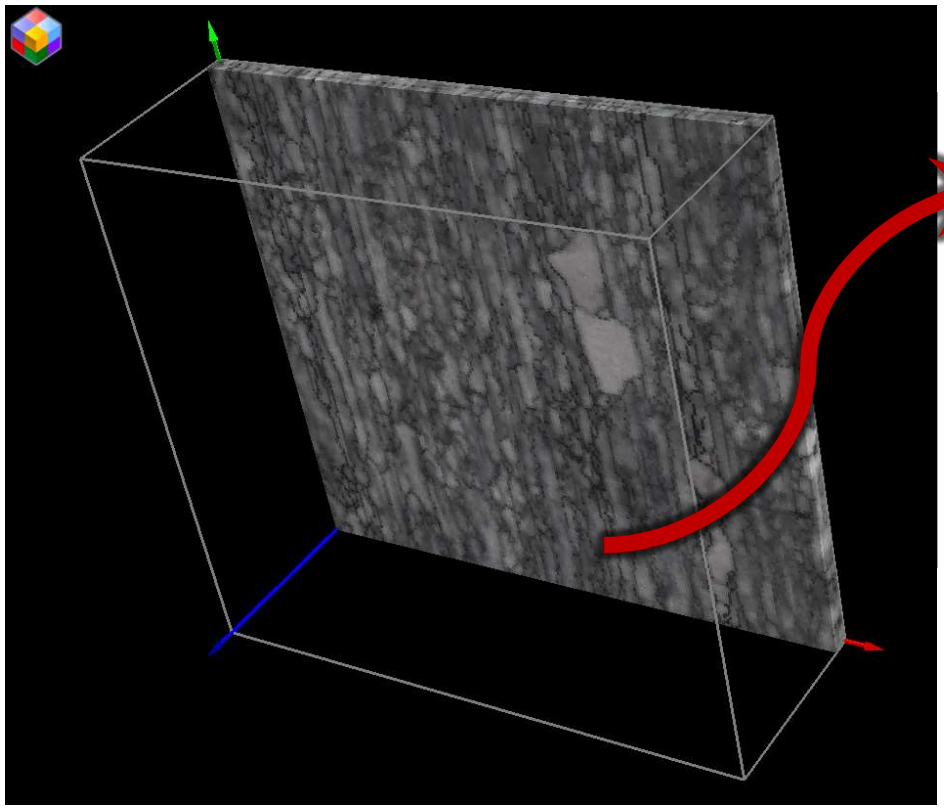
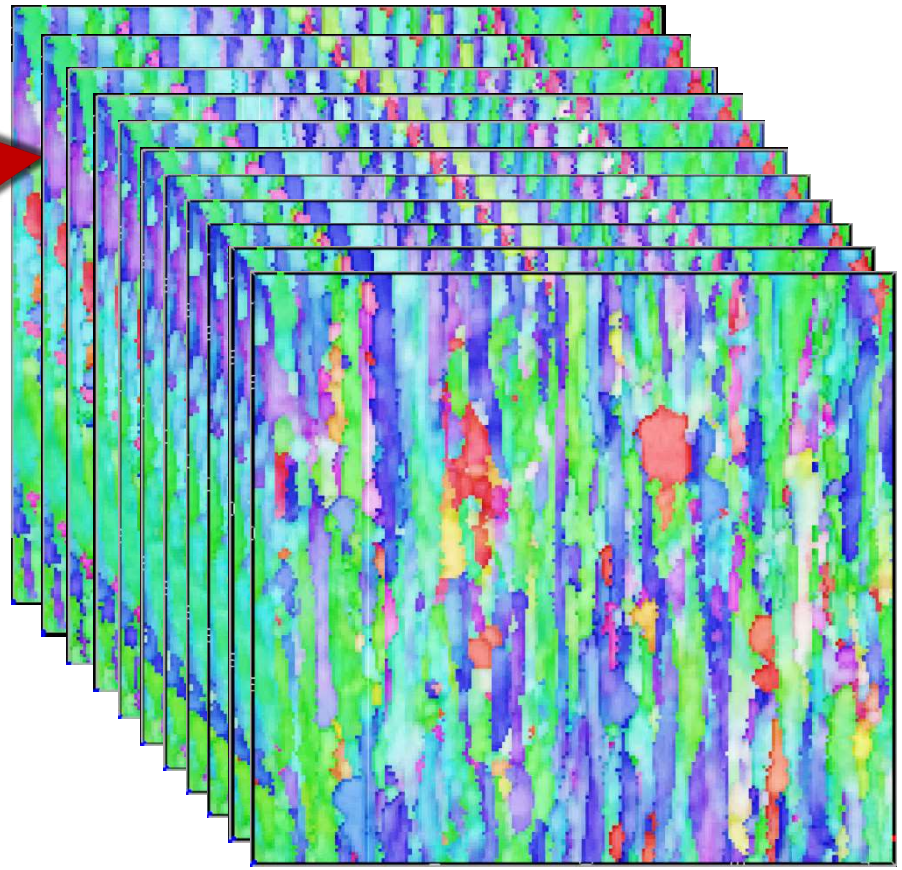
What is 3D orientation microscopy or „3D EBSD“?



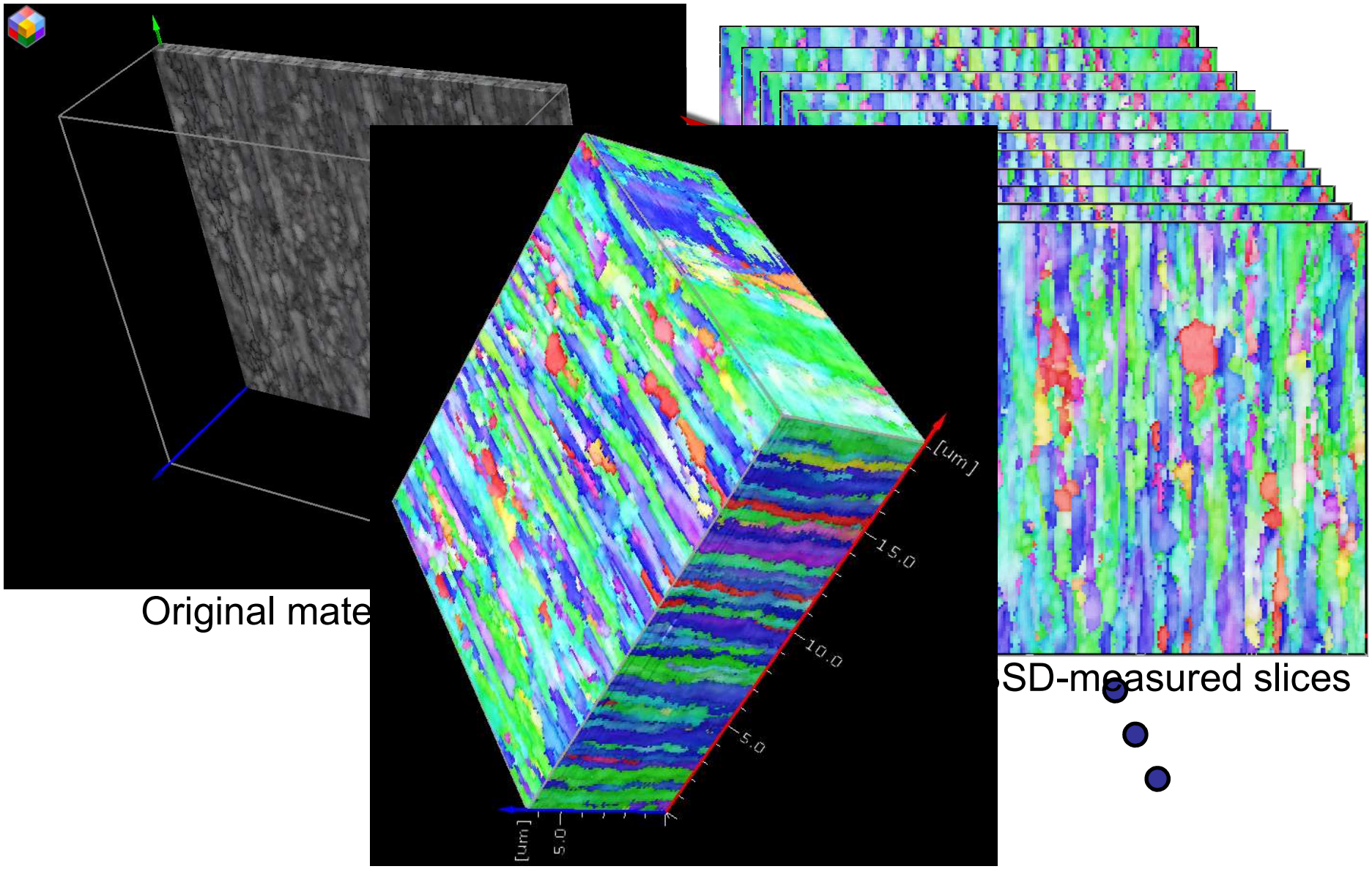
Original material



EBSD-measured slices

Reconstructed microstructure

What is 3D orientation microscopy or „3D EBSD“?



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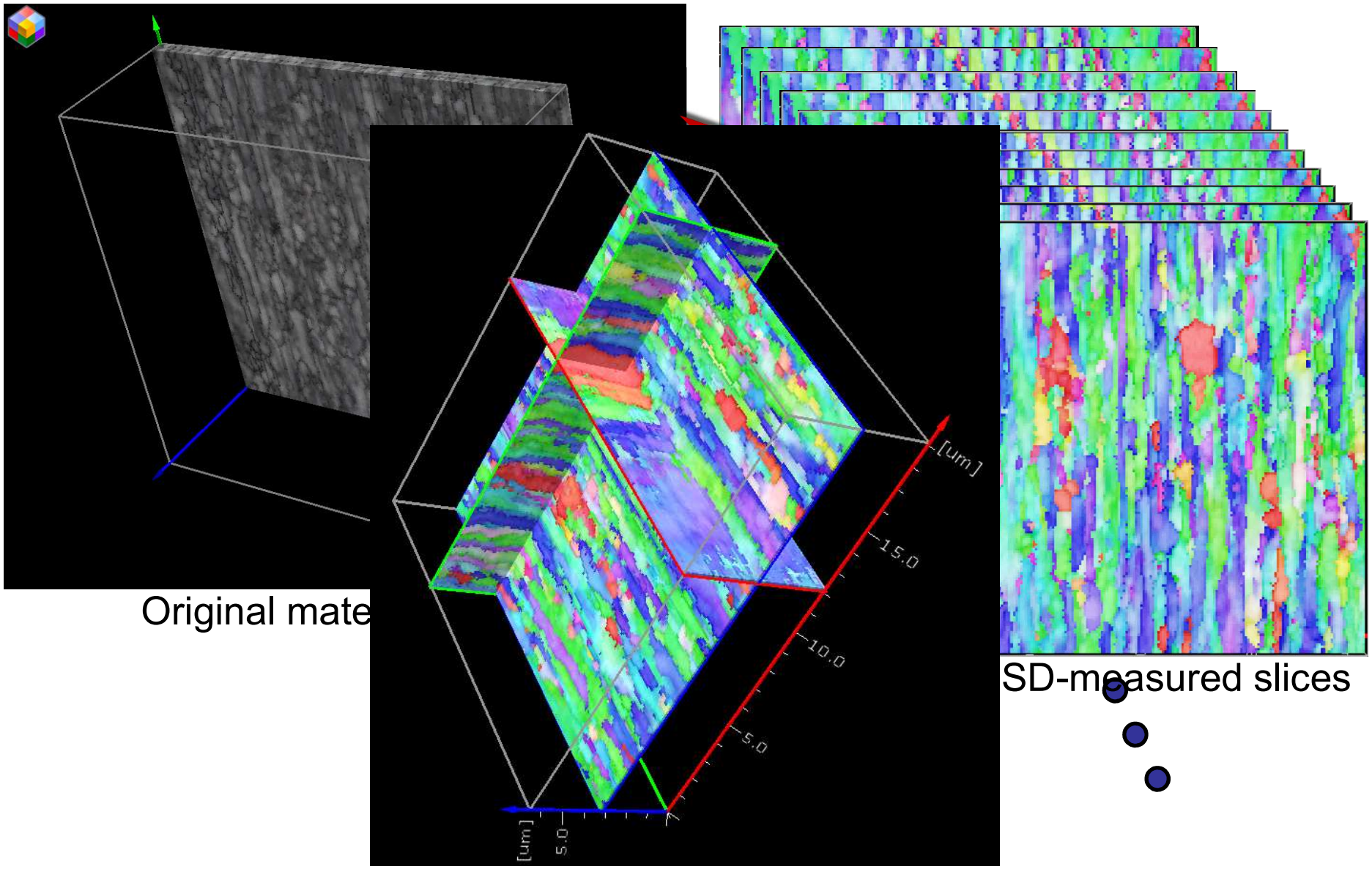

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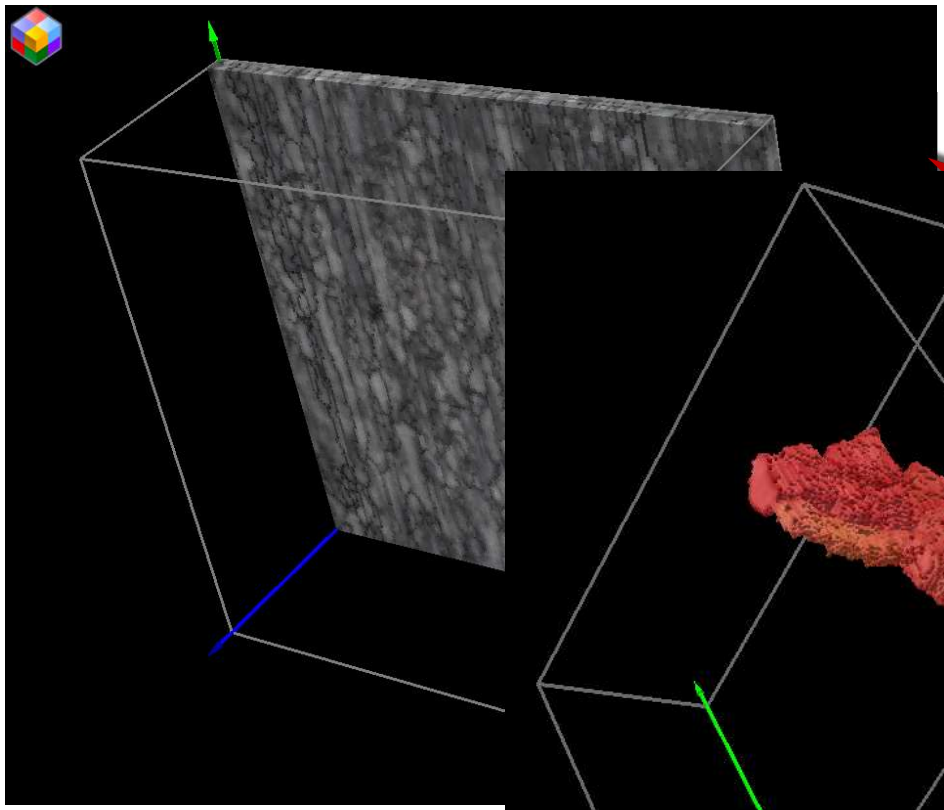
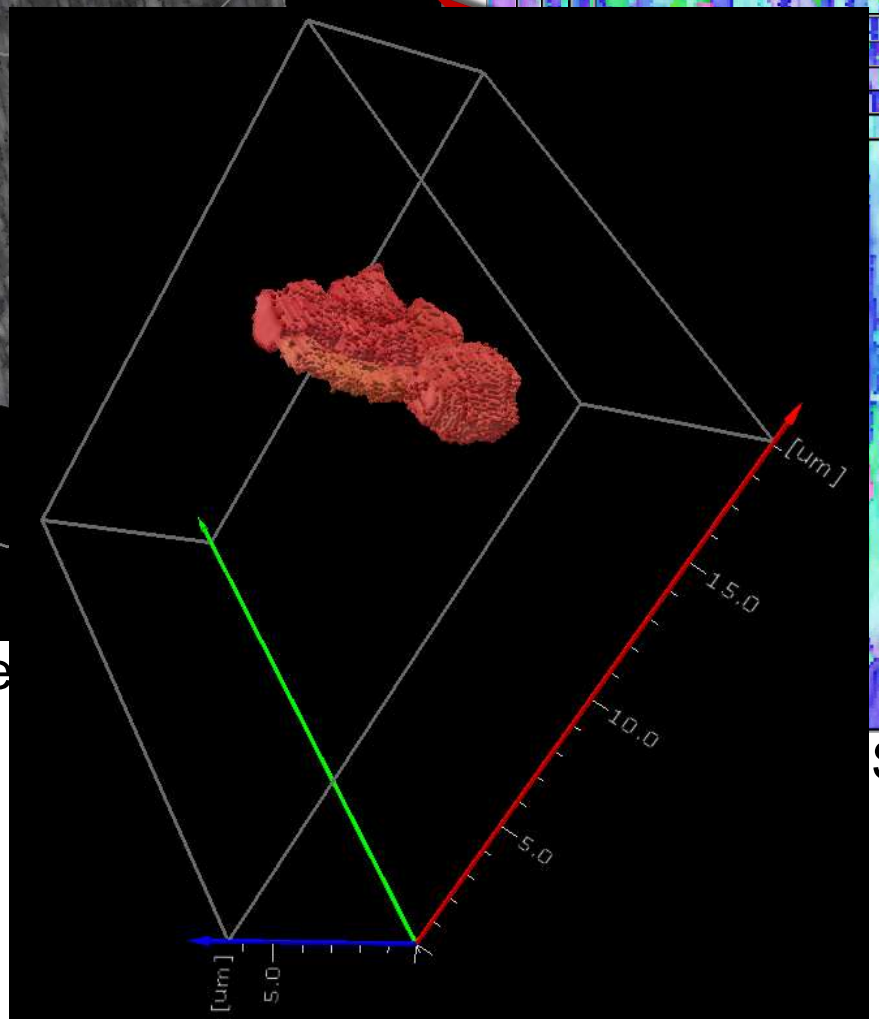
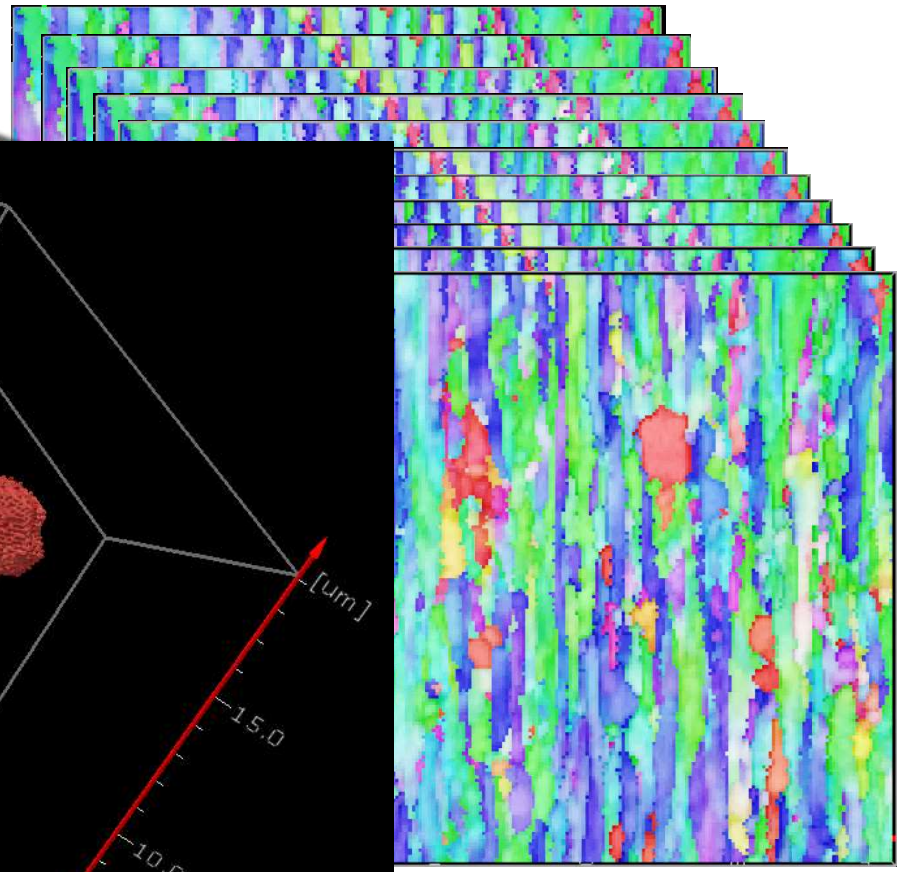


Original mate

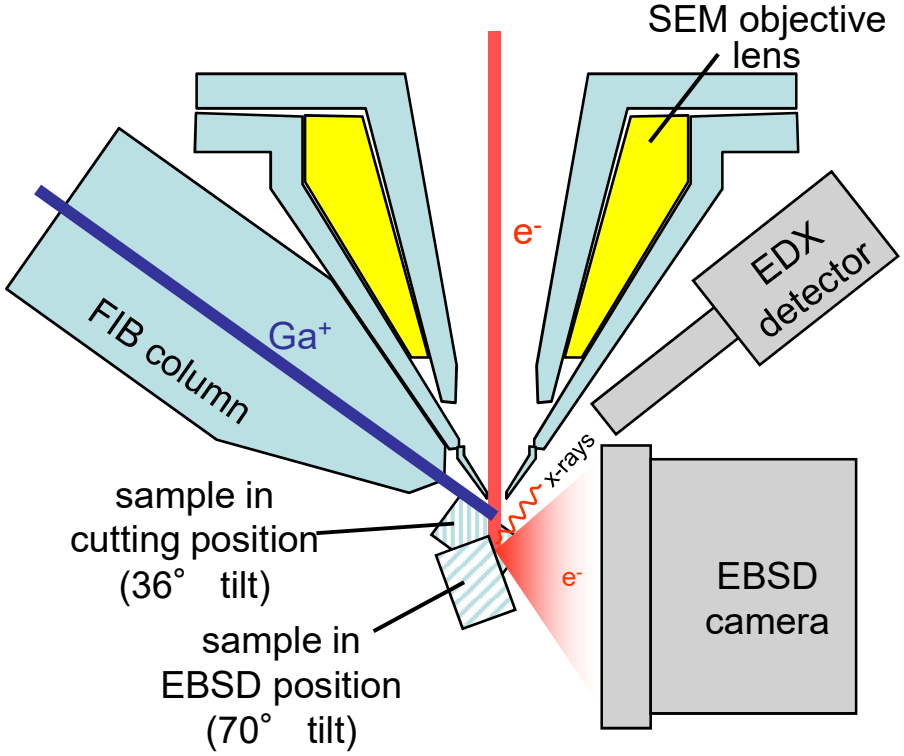


Reconstructed microstructure

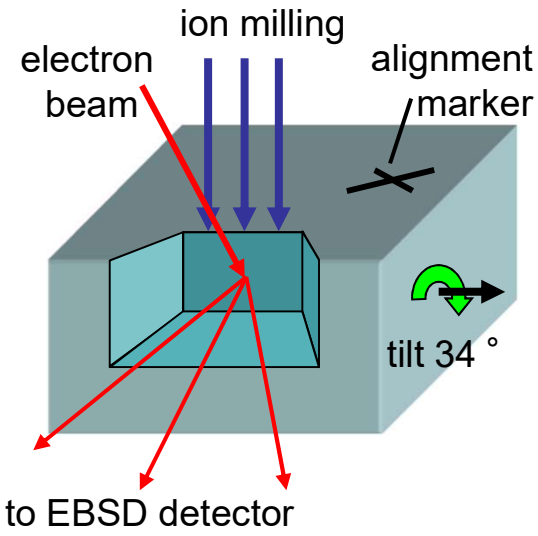


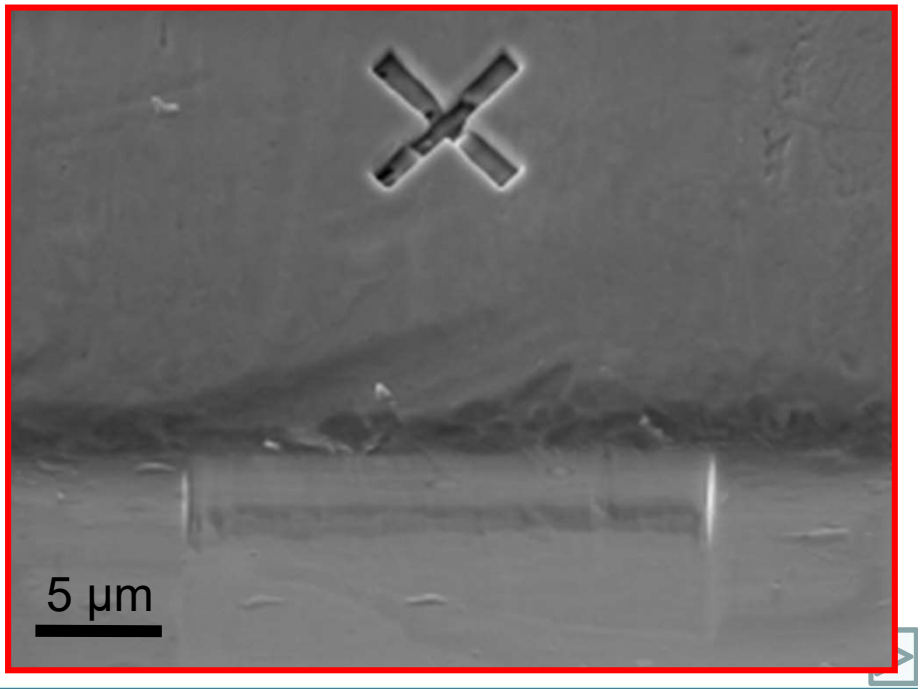
SD-measured slices

How to do 3D EBSD?



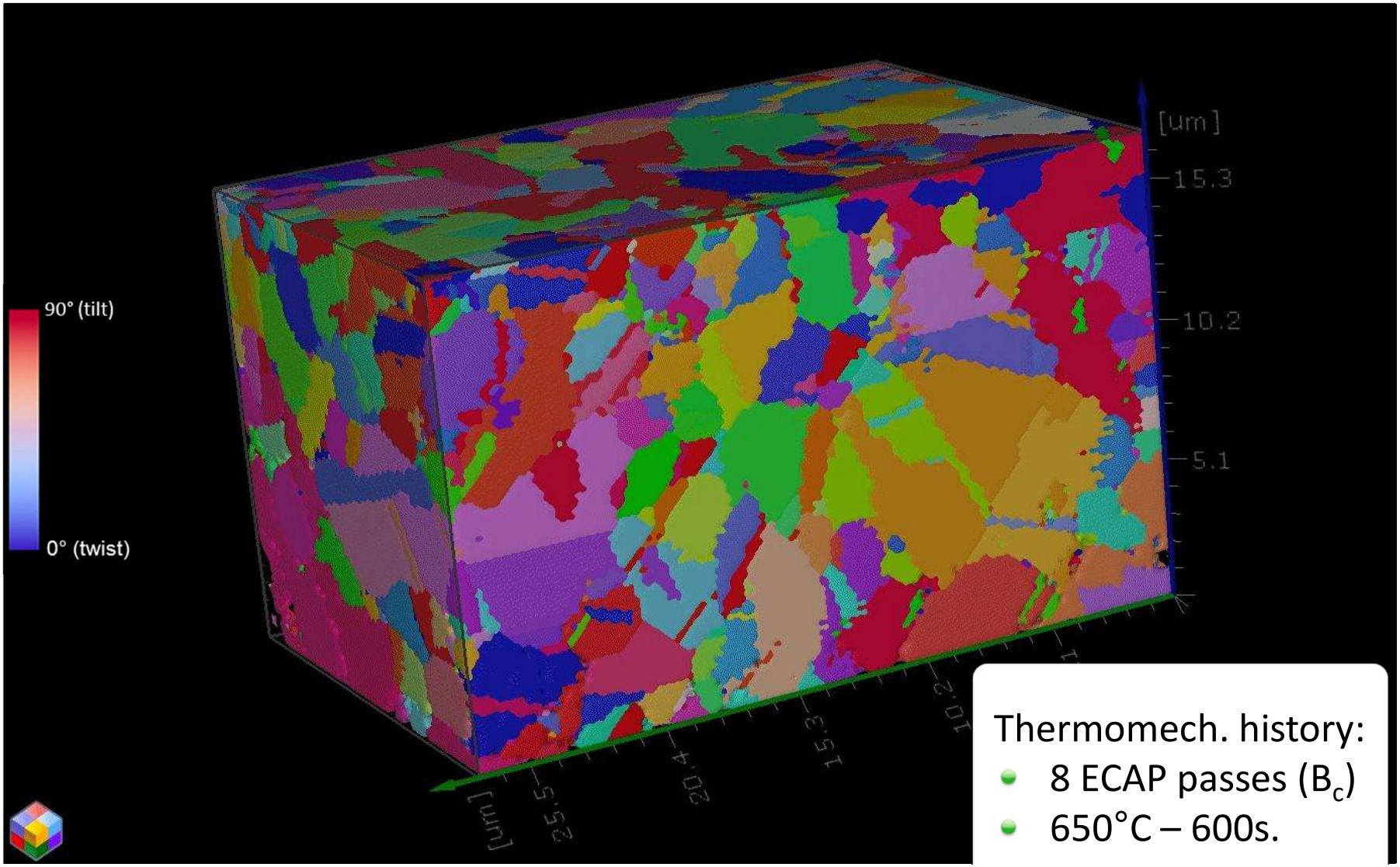
"tilt set-up"
Zaefferer, Wright, Raabe,
Mat. Trans. A (2008)





Example: grain boundaries in Cu-0.17wt%Zr

Misorientation: 60° <111> ± 5° <…>



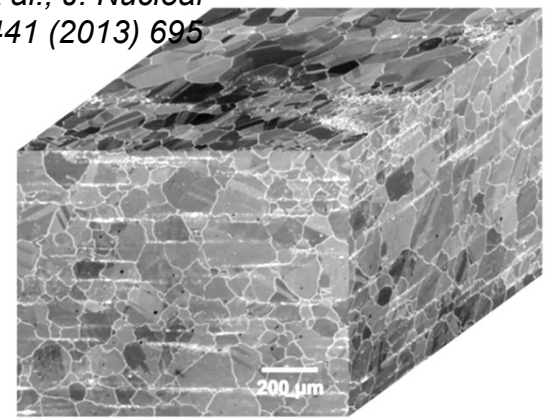
Thermomech. history:

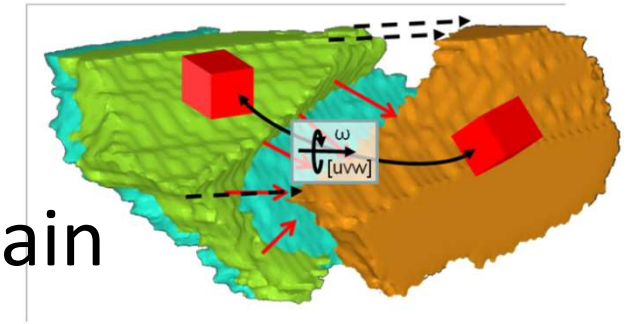
- 8 ECAP passes (B_c)
- 650°C – 600s.

Why 3D? What do we want/need to find out?

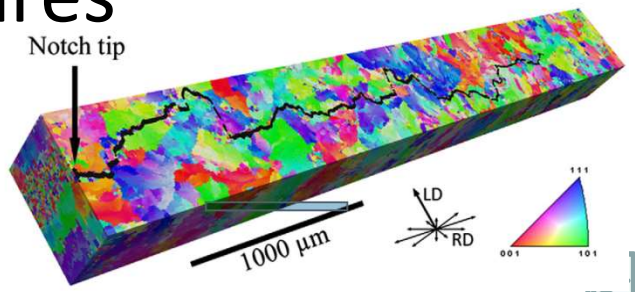
- Size, shape and arrangement of grains and phases in complex microstructures
- 3D-arrangement of defects (interfaces, dislocation, residual stresses)
- Comprehensive crystallographic grain boundary characterization
- Physical properties of microstructures
- Input data for simulation processes

Kun Mo, et al., J. Nuclear Materials 441 (2013) 695

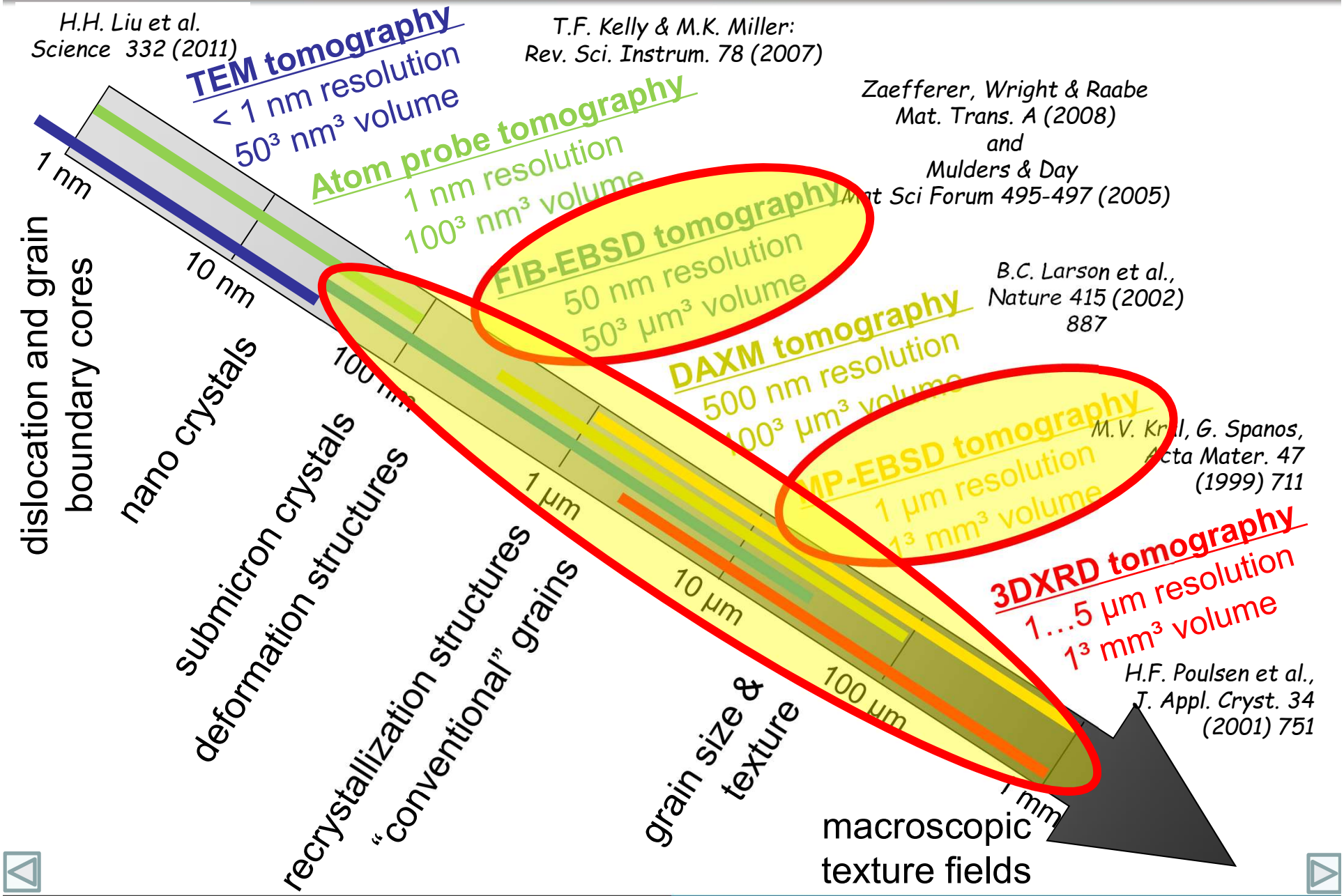




Pirgazi et al, Mat. Character. 90 (2014), 13



Length scale of 3D orientation microscopy



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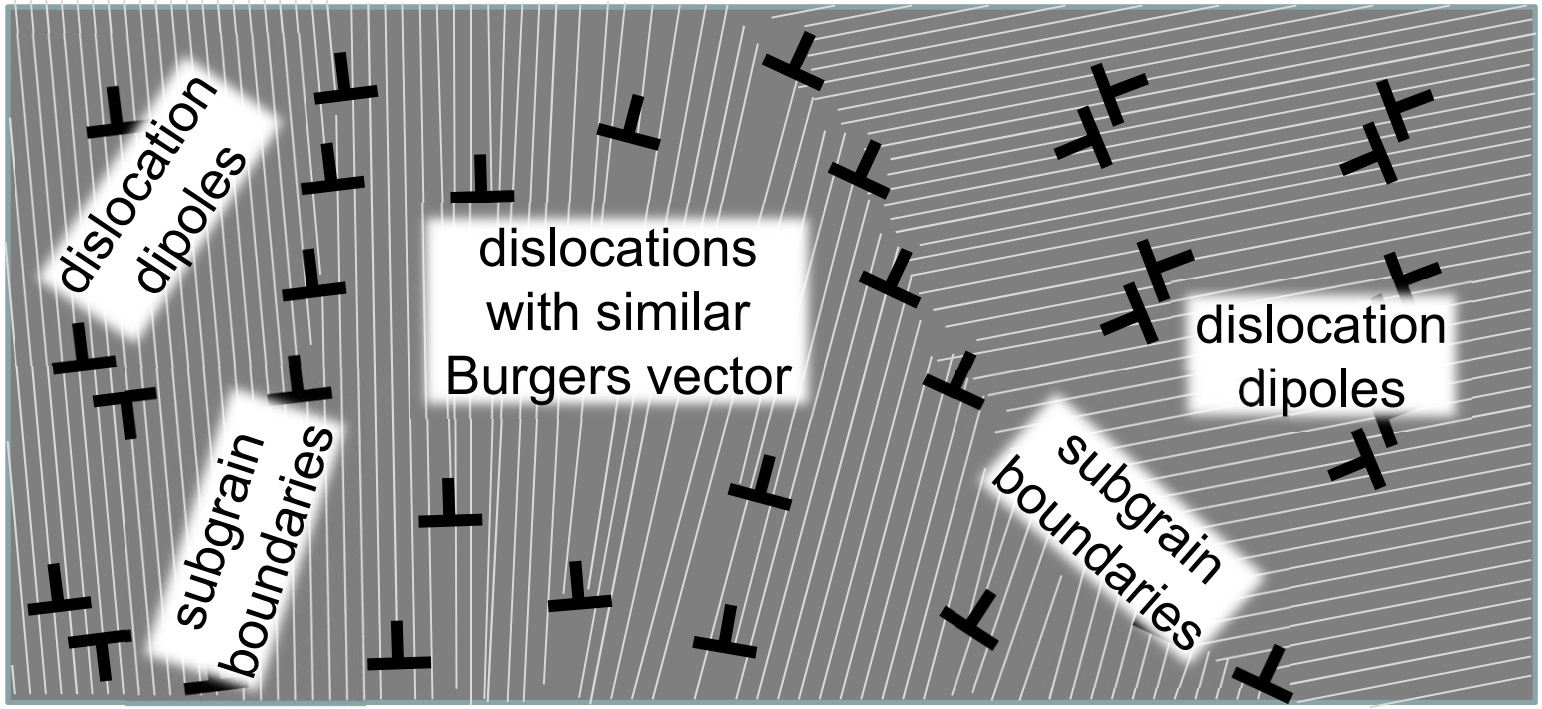
Classification of dislocations



Geometrically necessary dislocations: GND

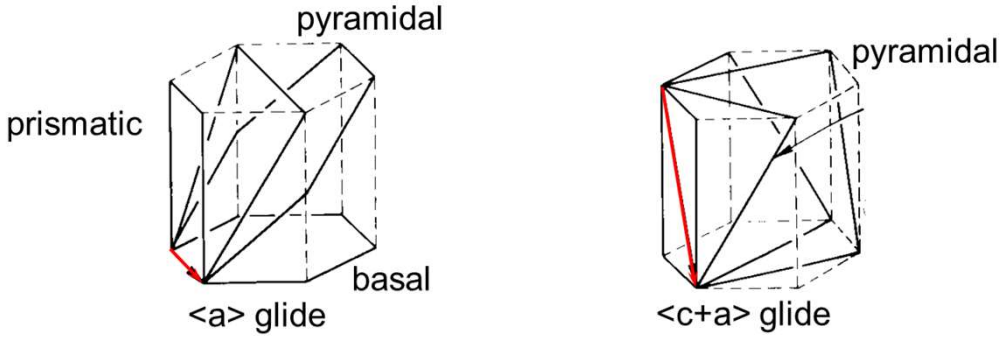
⇨ Create measurable lattice rotations

Statistically stored dislocations: SSD

⇨ Only lead to pattern blurring

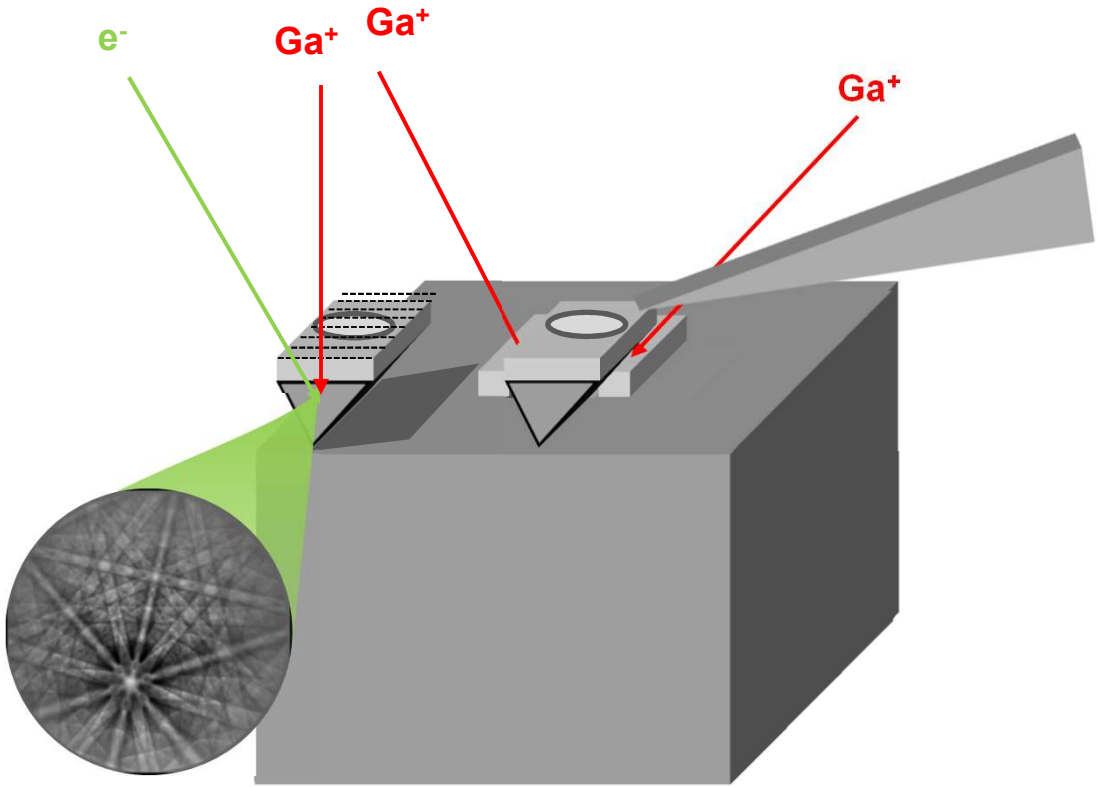
GND determination using 3D EBSD

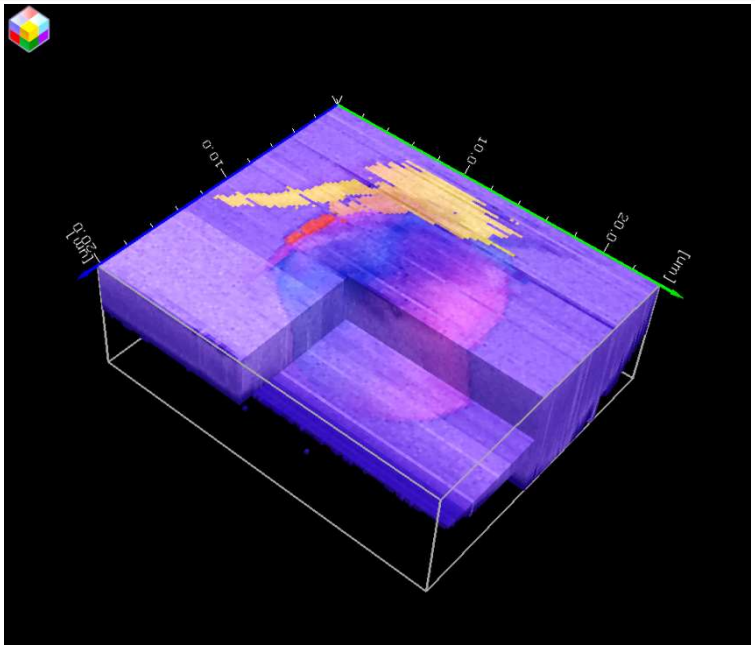
- GNDs quantify heterogeneity of plastic deformation
- 3D orientation data allow calculation of GND densities, separated for different types of dislocations

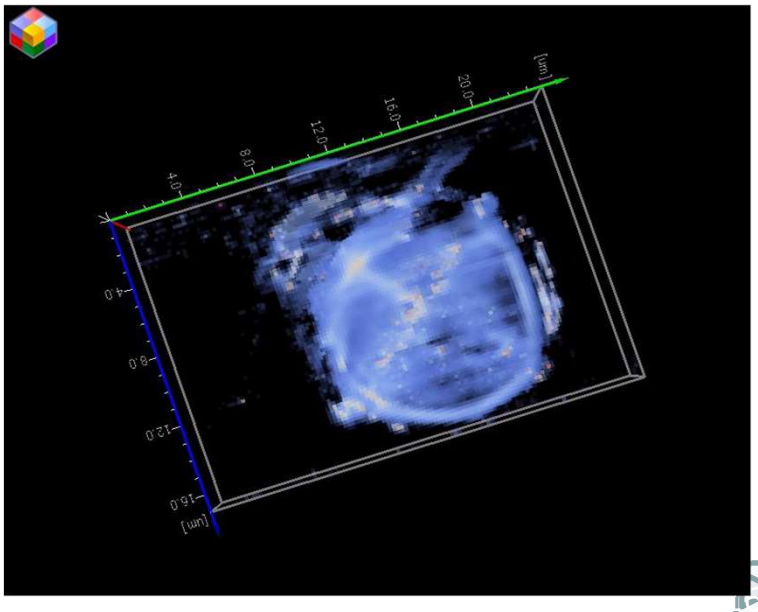


- The (slightly complicated) calculations make the following assumptions:
  - dislocations lead to lattice rotations („orientation gradients“)
  - each type of dislocation is associated with a characteristic rotation
  - from measurement of the rotations in space (via EBSD) the amount and type of dislocations can be determined

3D EBSD on nano indents in magnesium

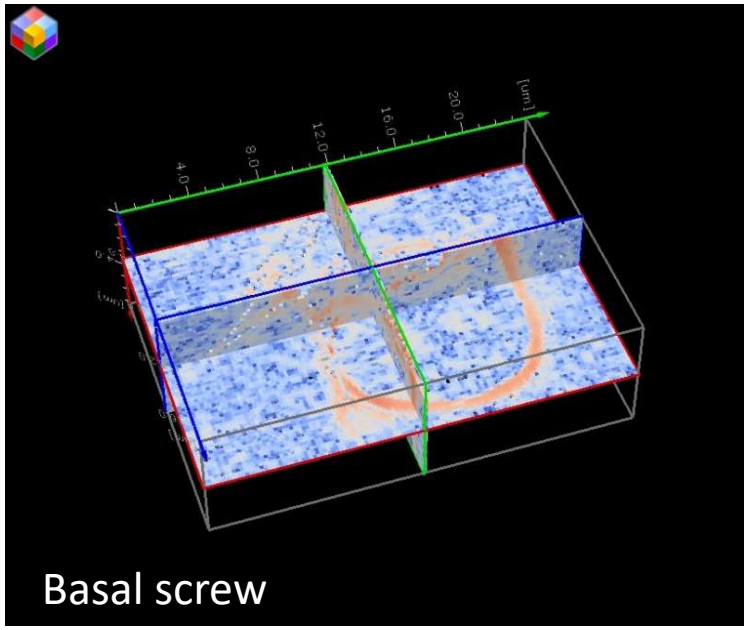


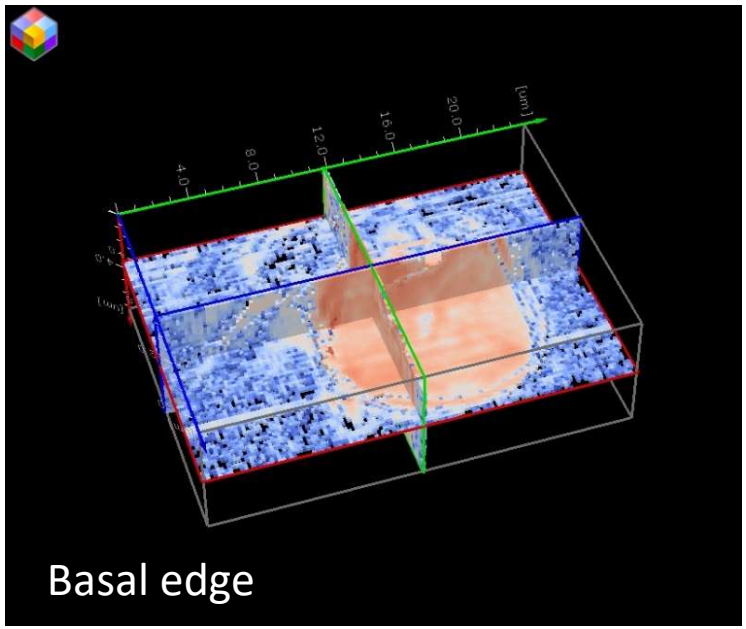




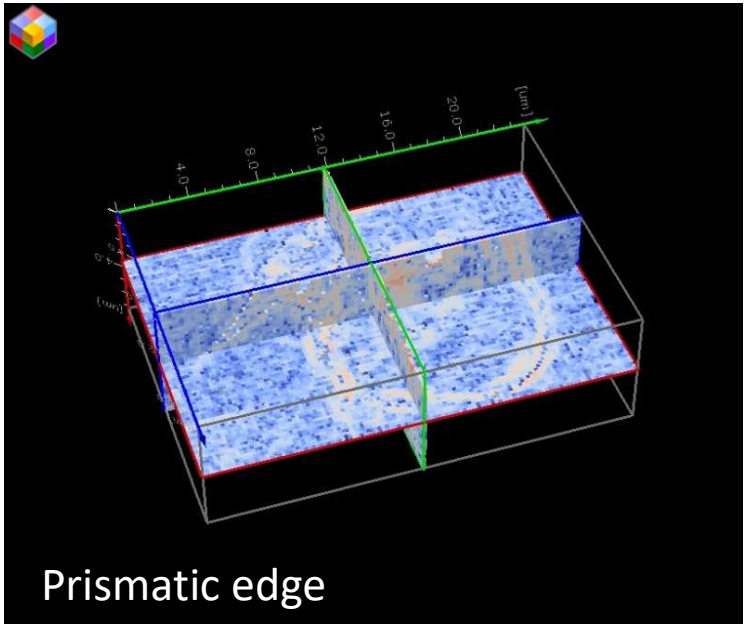
G. Nayyeri, et al. Mater Sci. Eng. A, **670** (2016), pp. 132-145

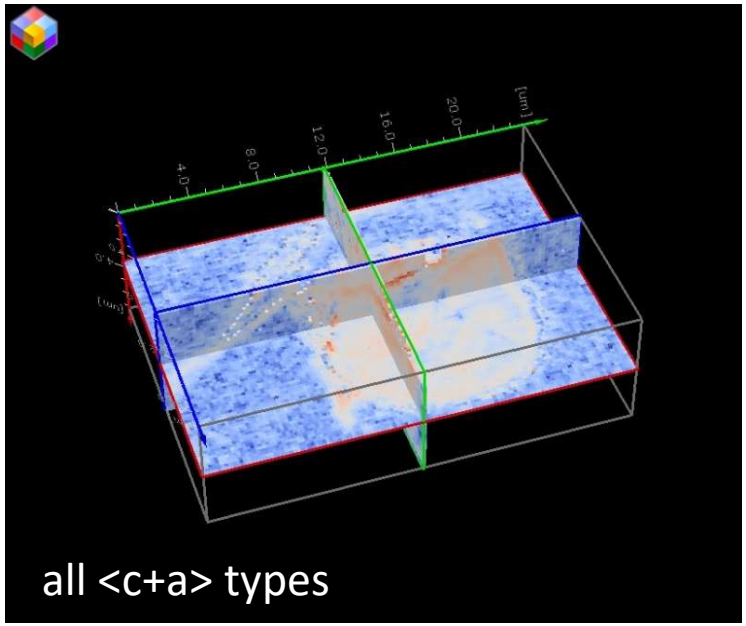
GNDs under (0001) indent





Dislocation density $1 \cdot 10^{12}$ to $2.7 \cdot 10^{15}$





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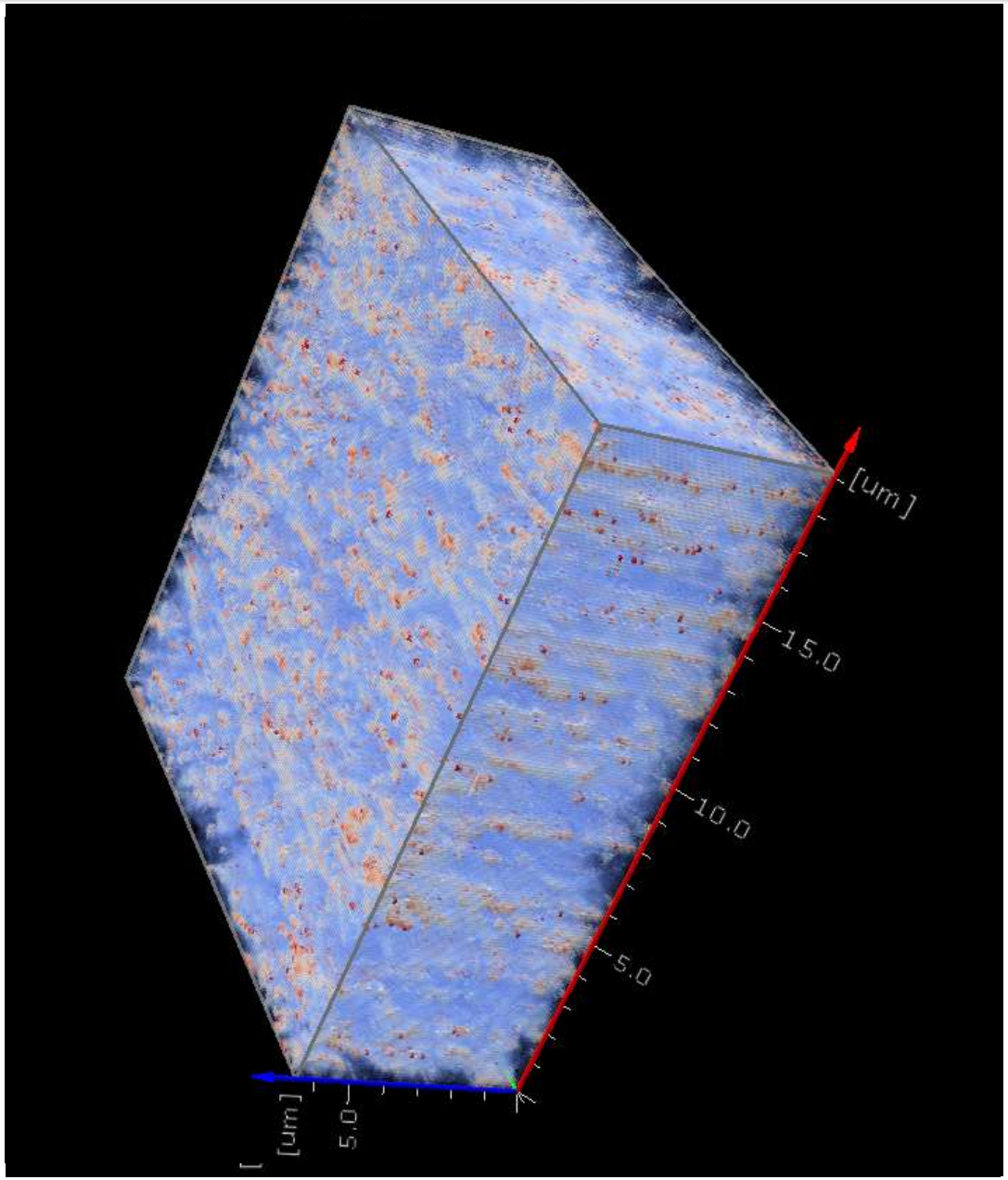
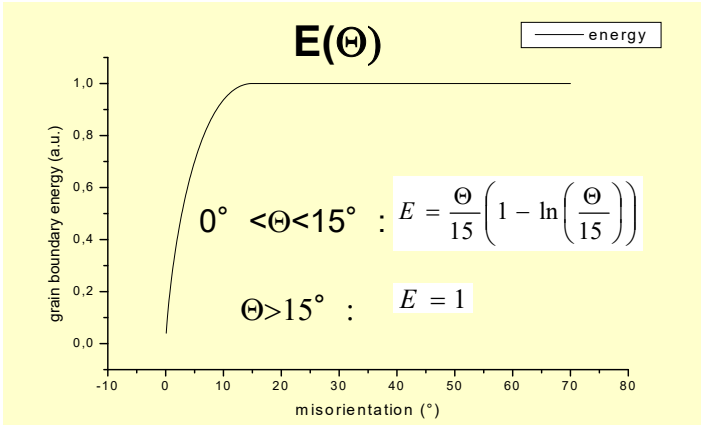
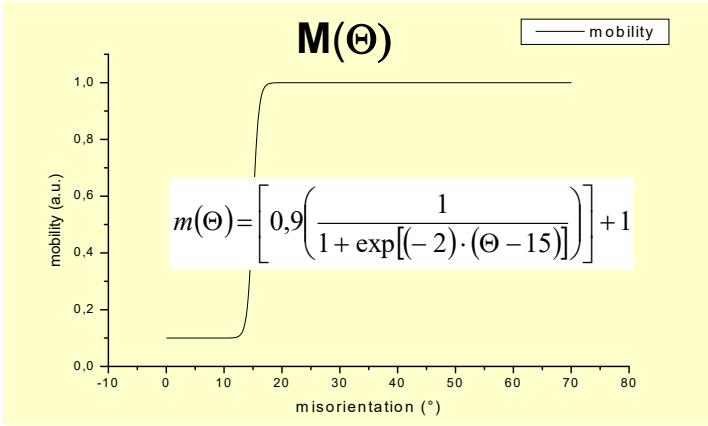
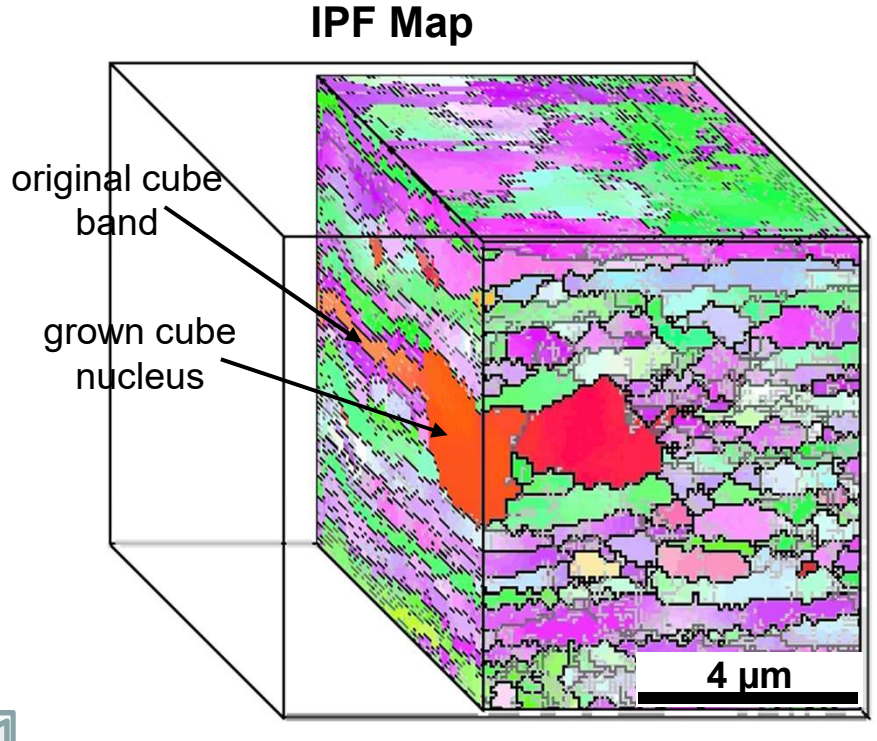
Observation of nucleation and stored energy

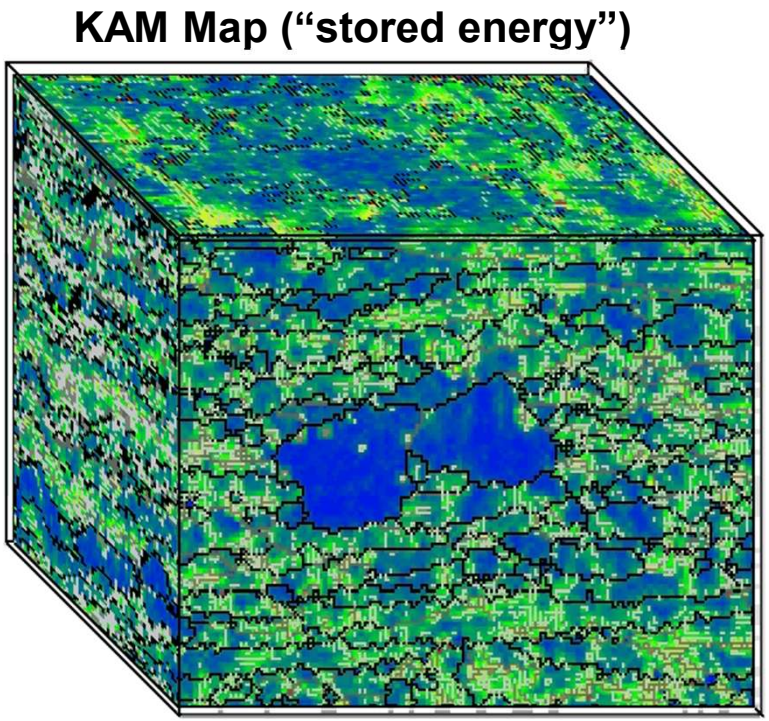


3D Monte-Carlo Potts model simulations









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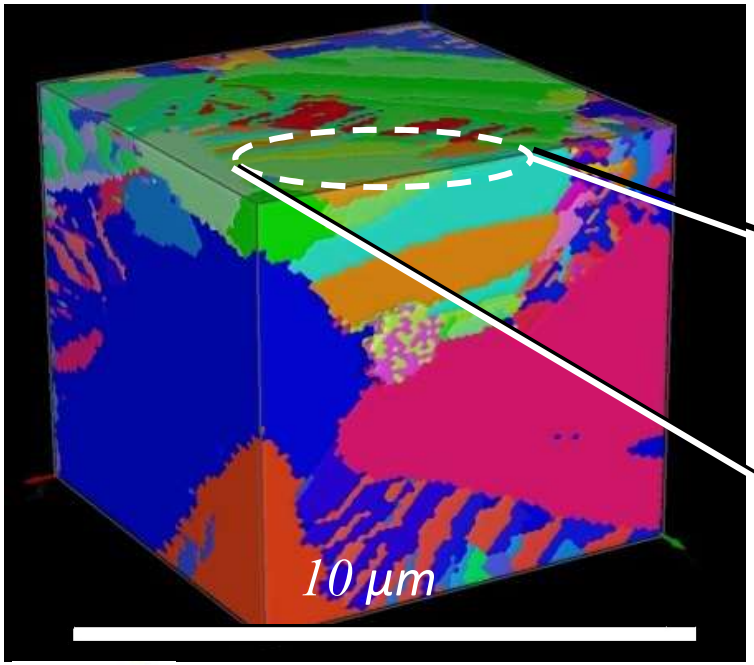
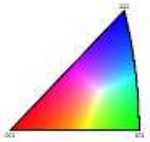
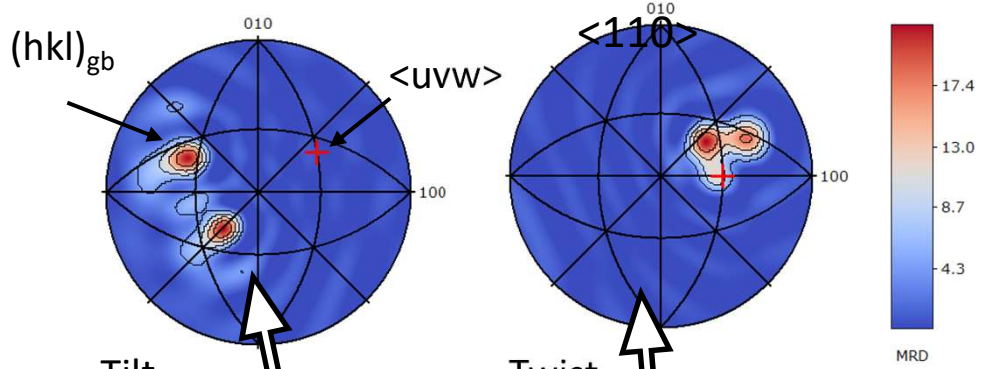
Comprehensive description of grain boundaries





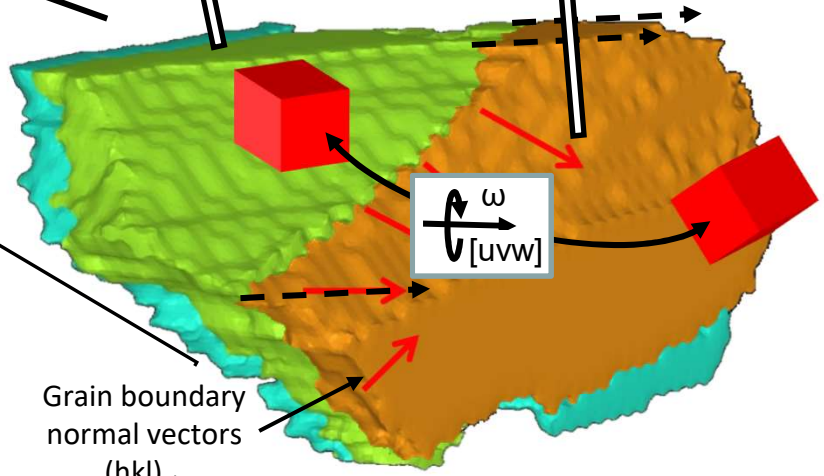
Mean misorientation:
53° <323>

57°
<110>



Tilt boundary

Twist boundary

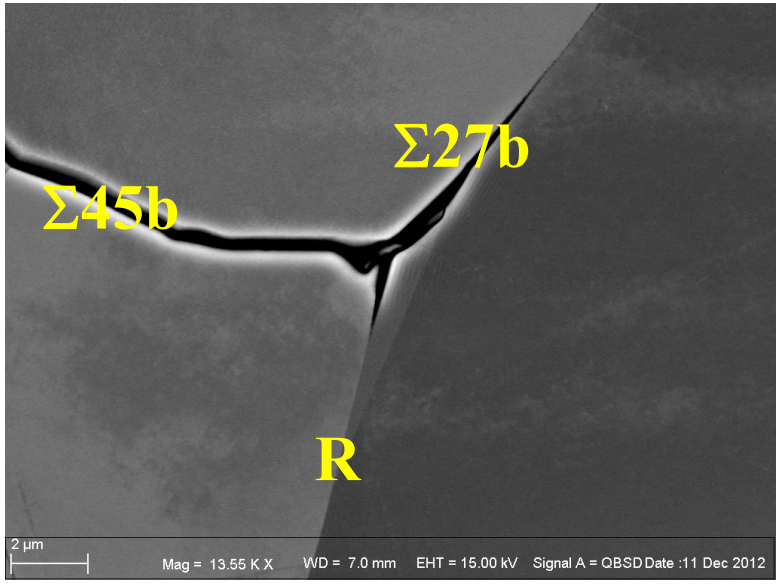


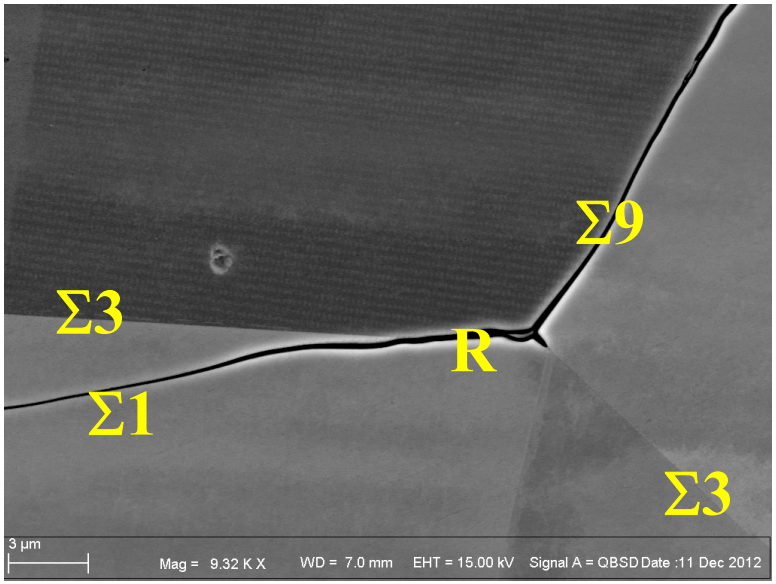
Grain boundary normal vectors $(hkl)_{gb}$

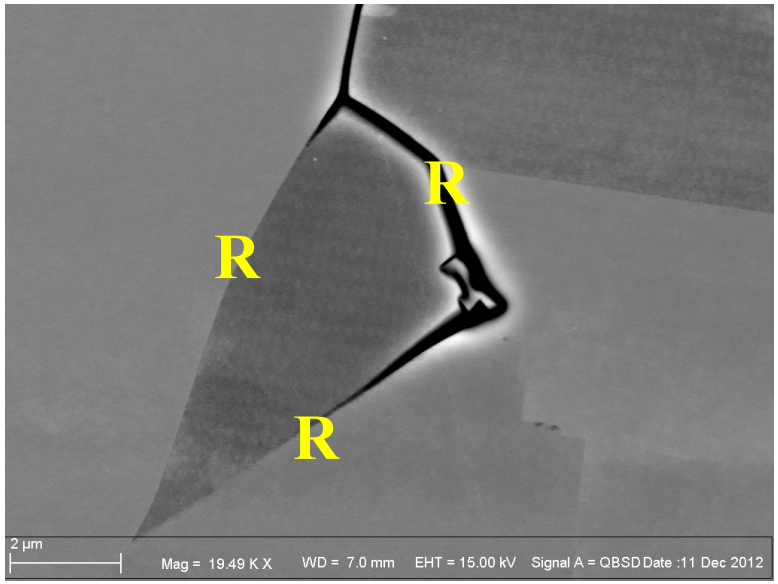
5 rotational parameters: ω (1), <uvw> (2), $(hkl)_{gb}$ (2)

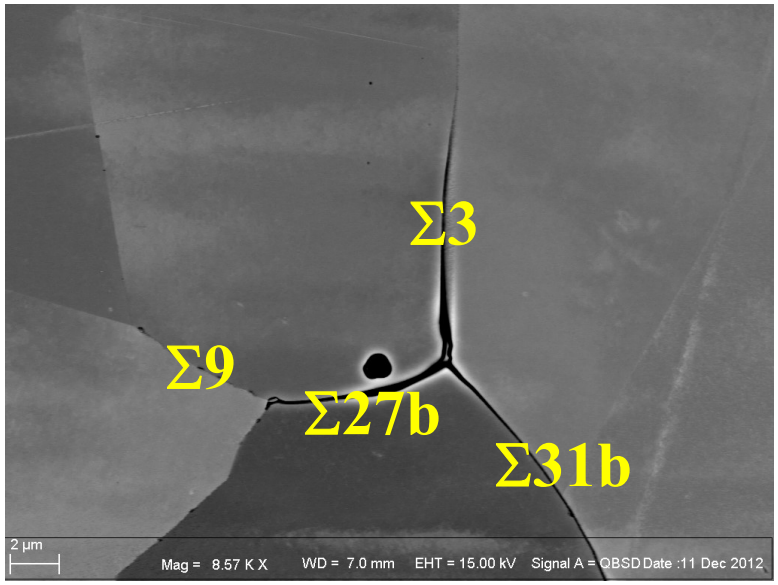
Measurement of grain boundary character requires 3D observation

Chemical properties: corrosion of boundaries



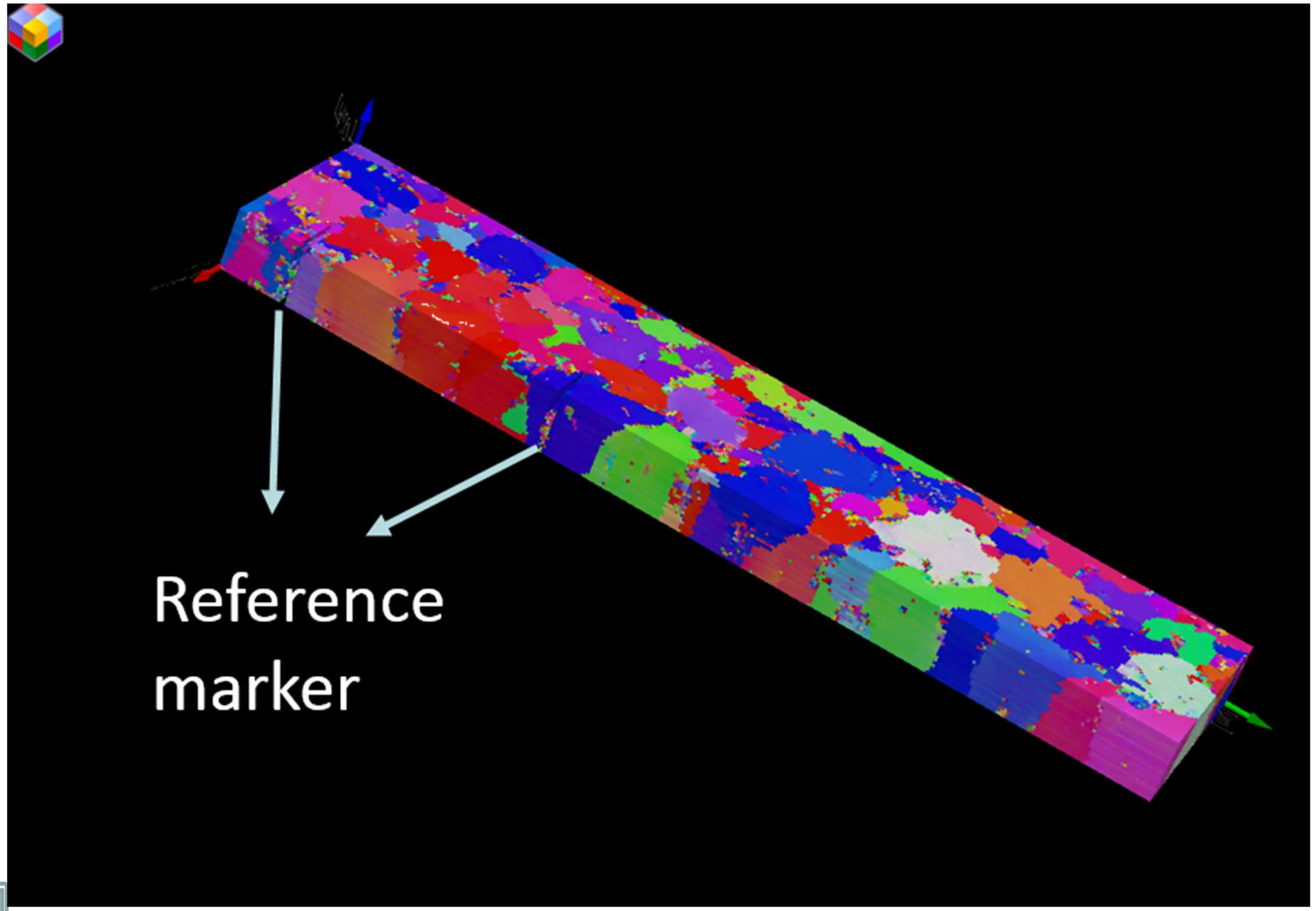




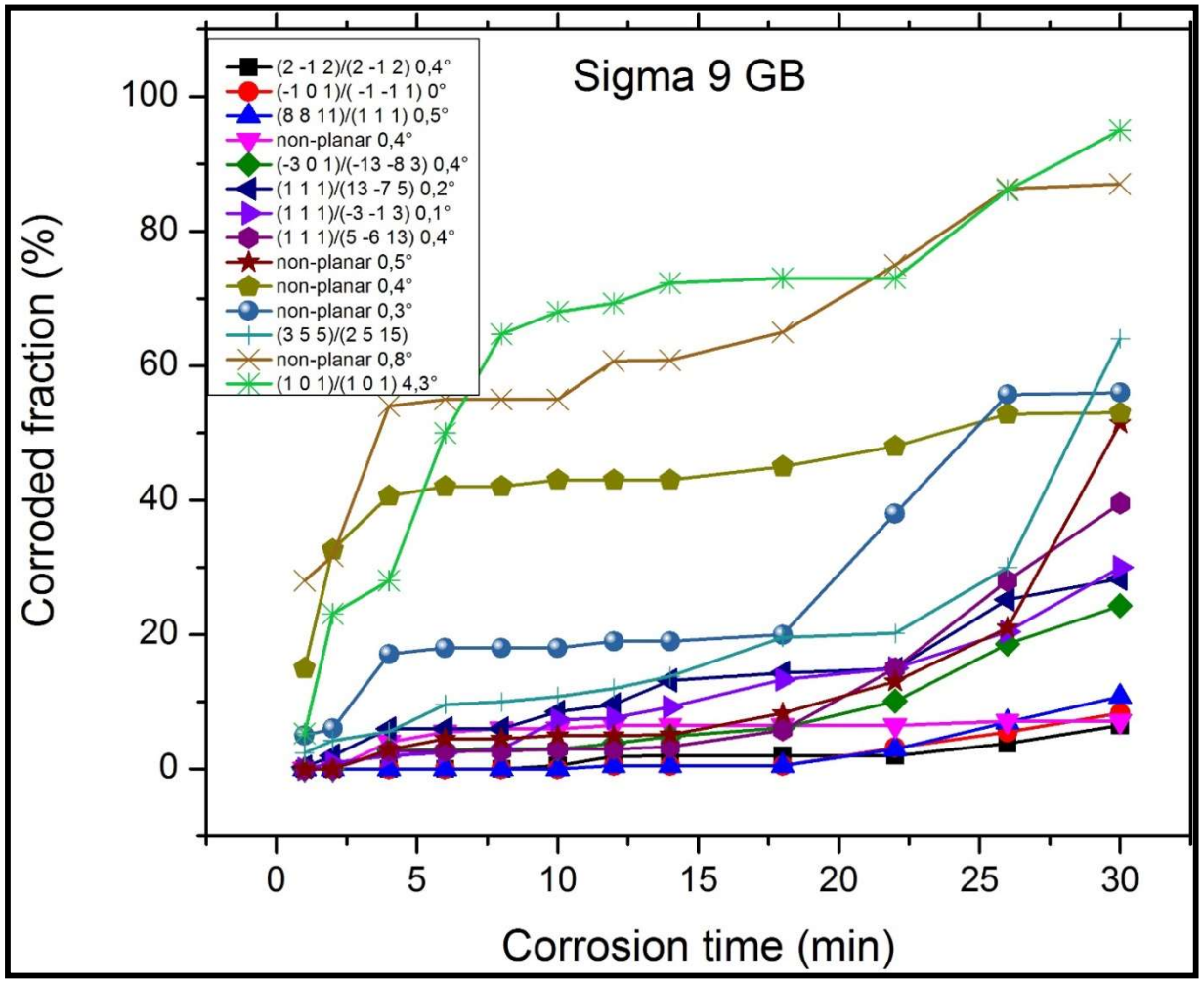


Corrosion behaviour is not only depending on misorientation

Large-size 3D EBSD map (mechanical sectioning)



Corrosion results of Σ3n GBs



- Σ9 with coherent or low Miller index GB plane have higher corrosion-resistance than those with high Miller index GB plane or non-planar GBs.
- Σ9 with large deviation angle are easily corroded.

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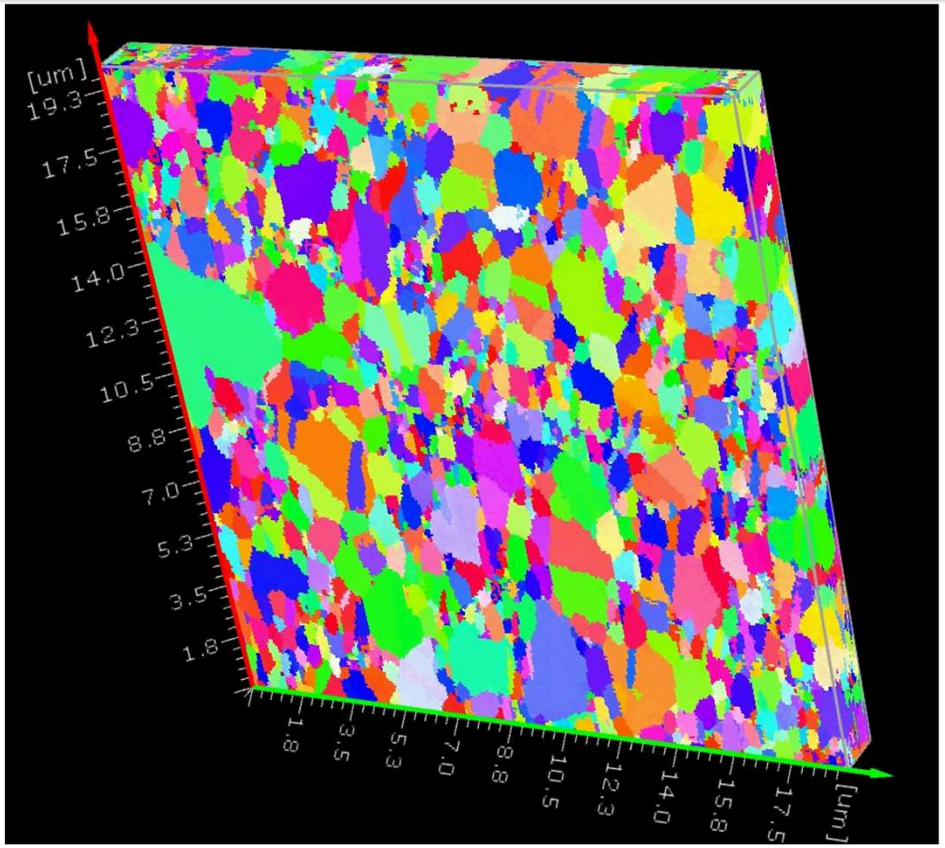
Slice misalignment

Sources of beam drift:

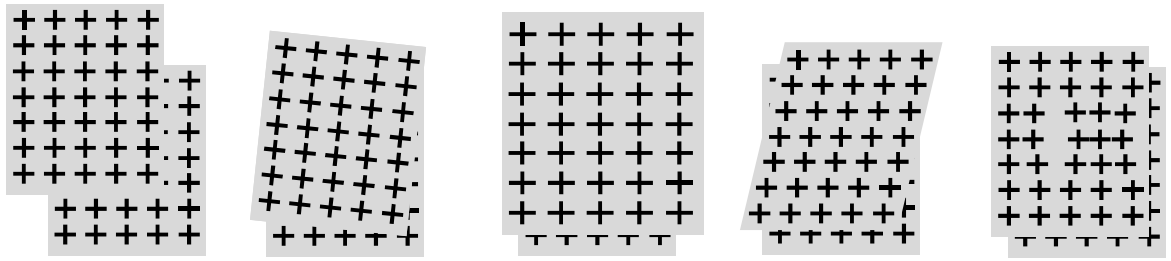
- Electro static (de)charging
- Thermal effects (e.g. lens cooling water)
- Mechanical relaxation (stage)
- Magnetic fields (lens)
- Surface roughness (e.g. curtaining)
- Incorrect sample repositioning



Post acquisition correction methods:

- Methods using reference features
- Minimization of total slice misorientation
- Minimization of scan line misorientation
- Relaxational methods (e.g. Monte Carlo Potts)

Errors by type



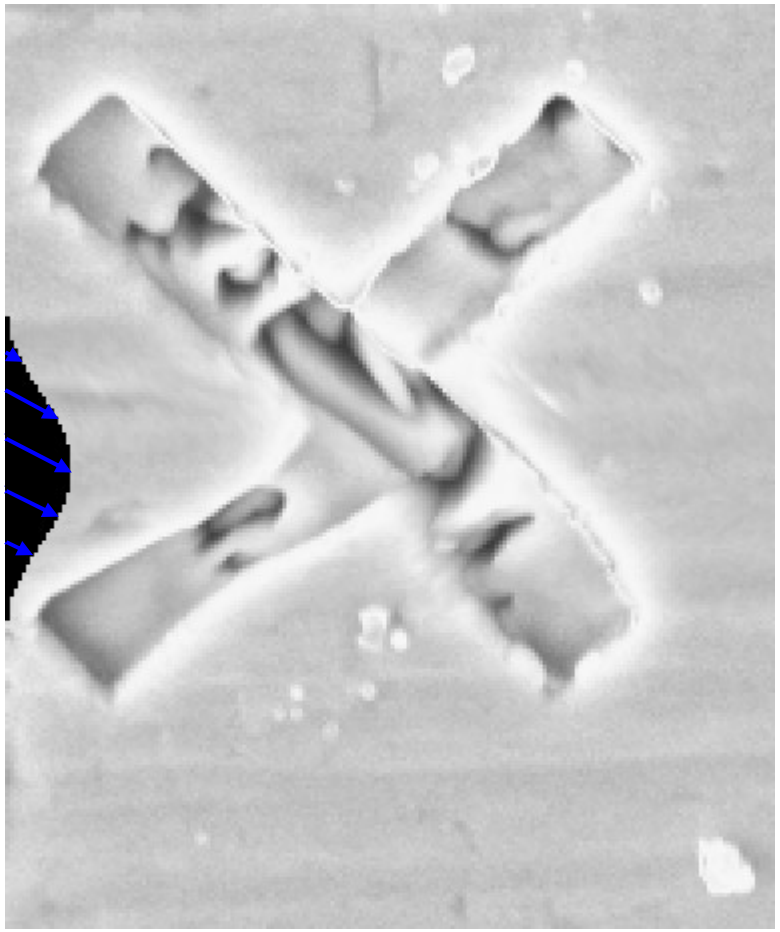
	Error	Translation	Rotation	Scaling	Shearing	Warping
Transform	Rigid	✓	✓			
	Similarity	✓	✓	✓		
	Affine	✓	✓	✓	✓	
	Non-rigid					✓

Parametric

Non-rigid alignment of fundamental importance for the quality of all subsequent analysis:

- Feature or intensity based
- Spatial or frequency domain
- Unimodal / multimodal

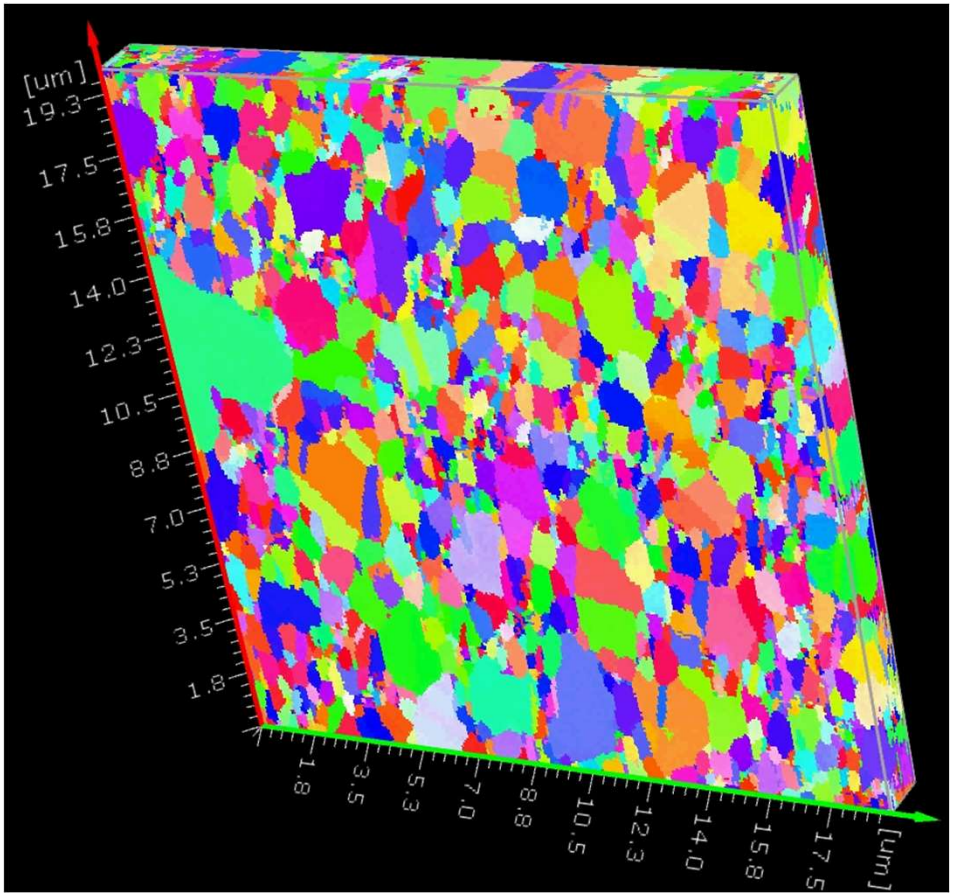
Validation 2(2)



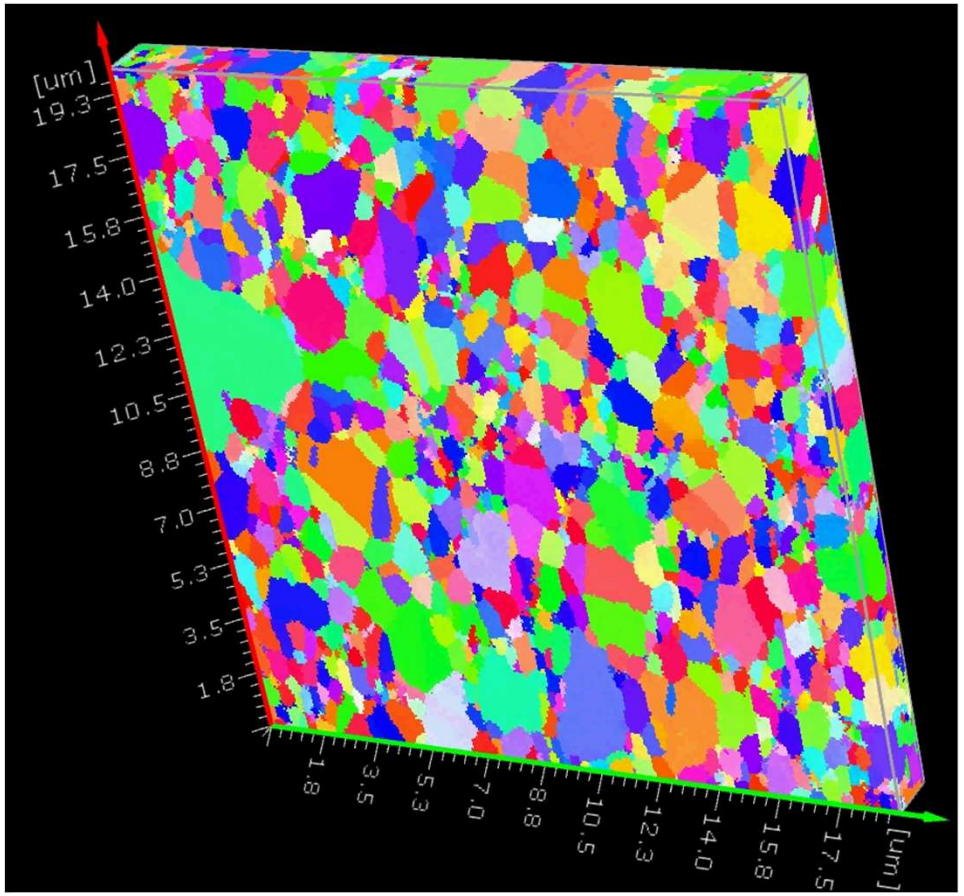
Generic row-wise drift (X and Y)

Original

Example: non-ridgid alignment



before alignment



after alignment

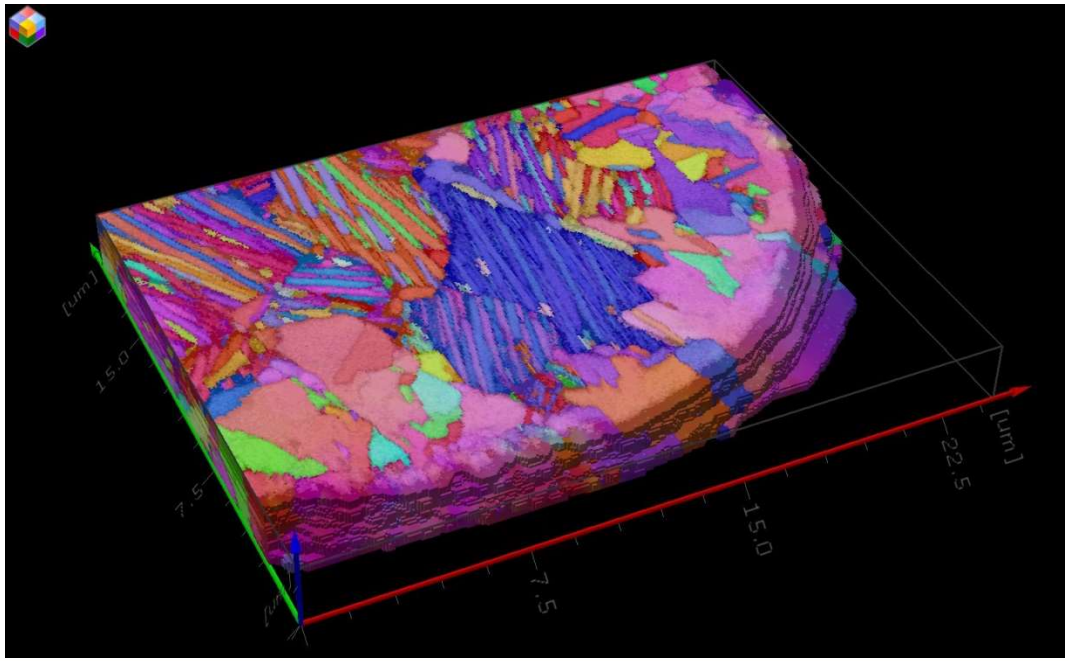
Possibilities & Limitations

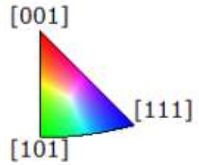
- 3D EBSD is a versatile and powerful technique
  - Crystallographic characterization of grains, phases, interfaces, dislocations, cracks, etc
  - 5-parameter grain boundary characterization is unique
  - Combination with compositional characterization via EDX
  - Ideal combination with simulation tools
- Time consuming technique (measurement & analysis):
  - one order of magnitude more data usually means one order of magnitude longer measurements
- Statistical relevance of measured data?
- Artefacts and imperfections:
  - slice alignment and depth removal rates with serial sectioning
- Destructive technique: only static microstrctrs observable

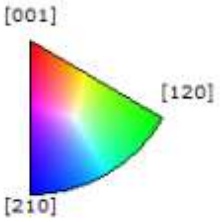
ESPRIT QUBE

Data postprocessing – Subsetting & visualization

- Powder metallurgy particle
- Phases: Brass & intermetallic phase (trigonal)
- Serial sectioning using a Ga FIB-SEM with the static method
- Slice preparation time: ~5min/slice
- 3D EBSD/EDS data acquisition time: ~9min/slice



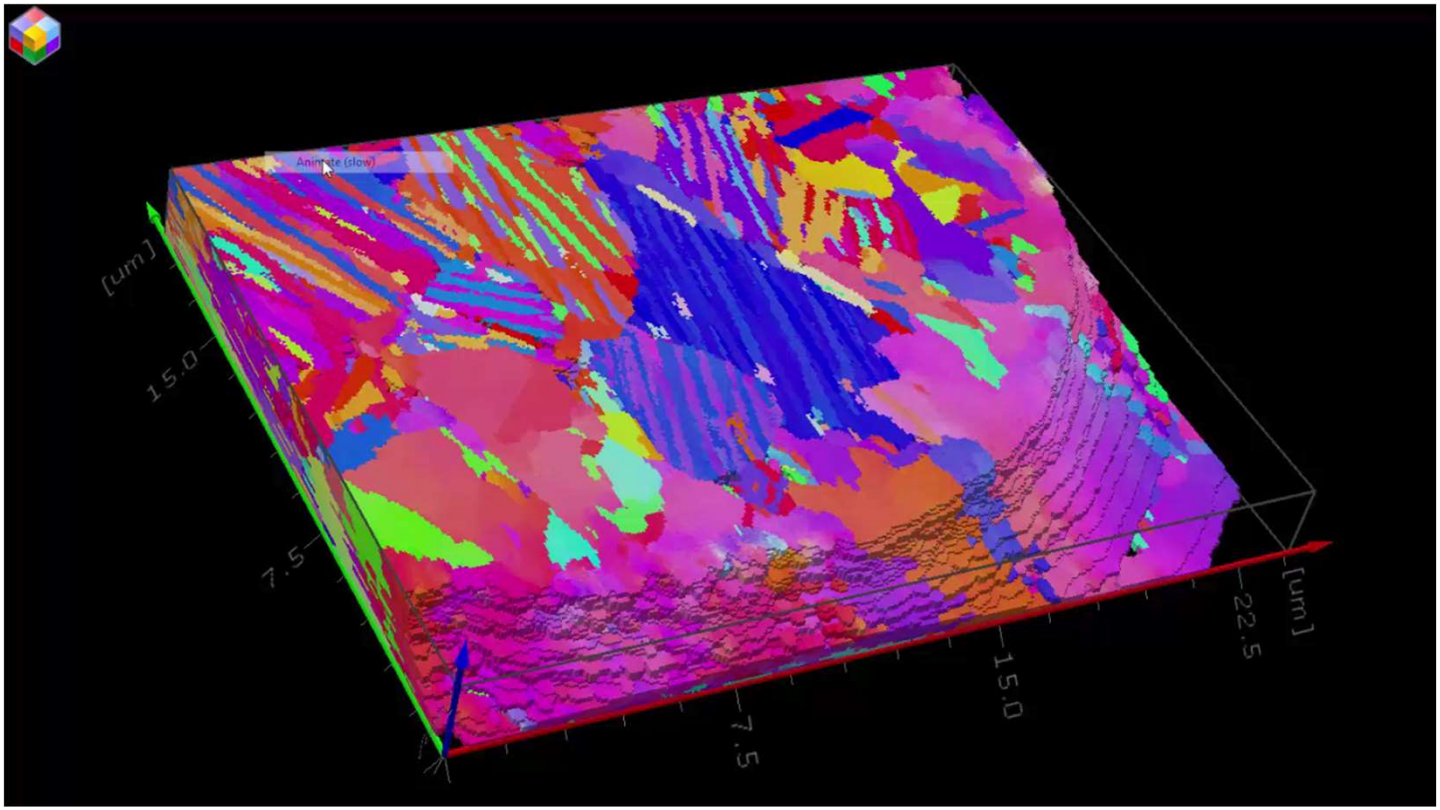


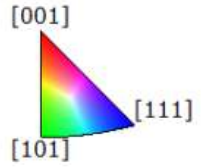


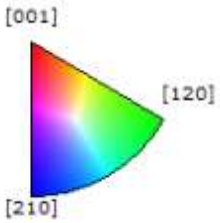
ESPRIT QUBE

Data postprocessing – slice realignment

3D EBSD – 27 slices – no realignment applied





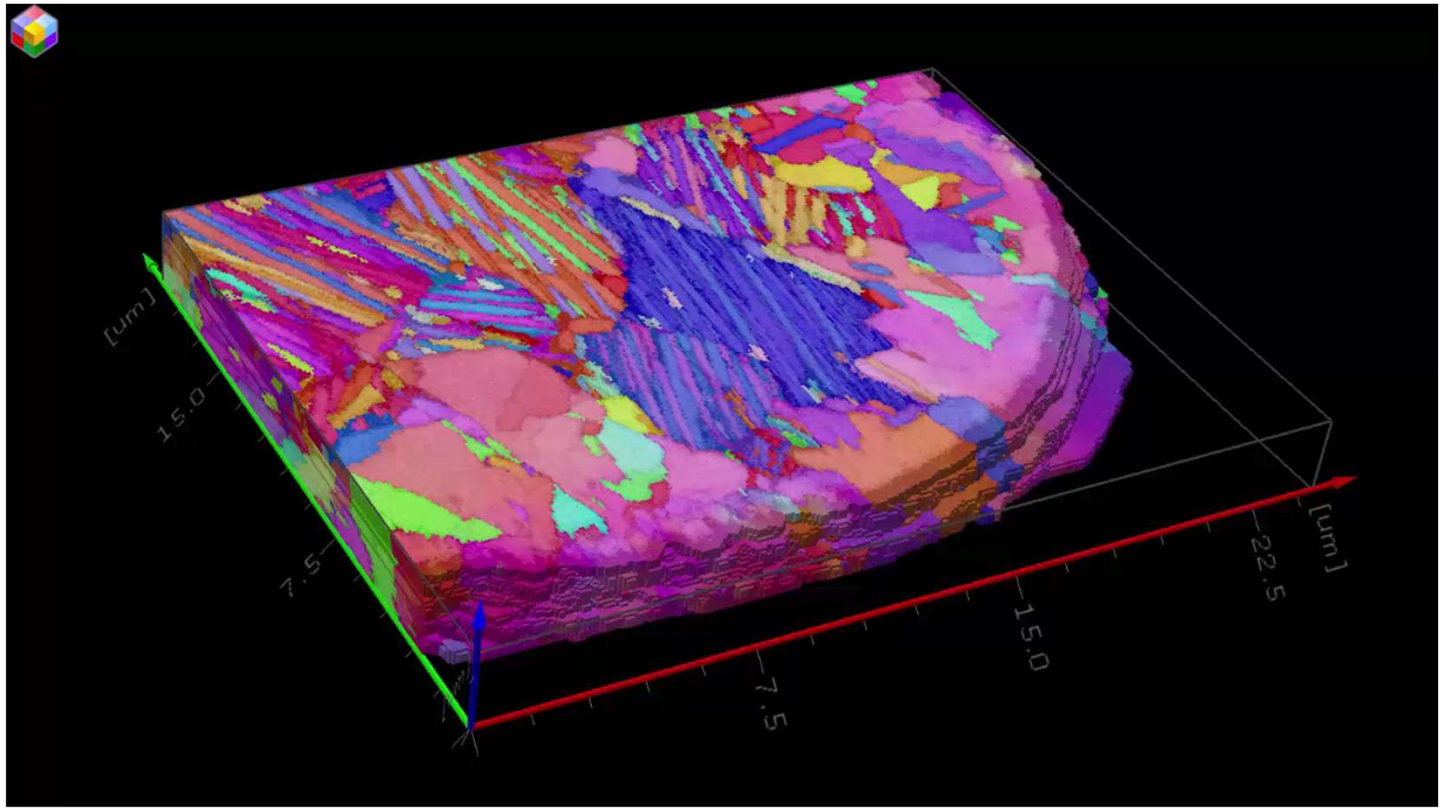


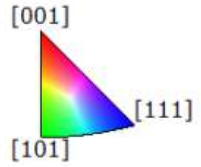
- Strong initial misalignment due to particle instability

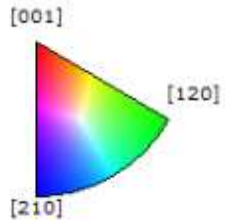
ESPRIT QUBE

Data postprocessing – slice realignment

3D EBSD – 27 slices – Euler angles based slice realignment



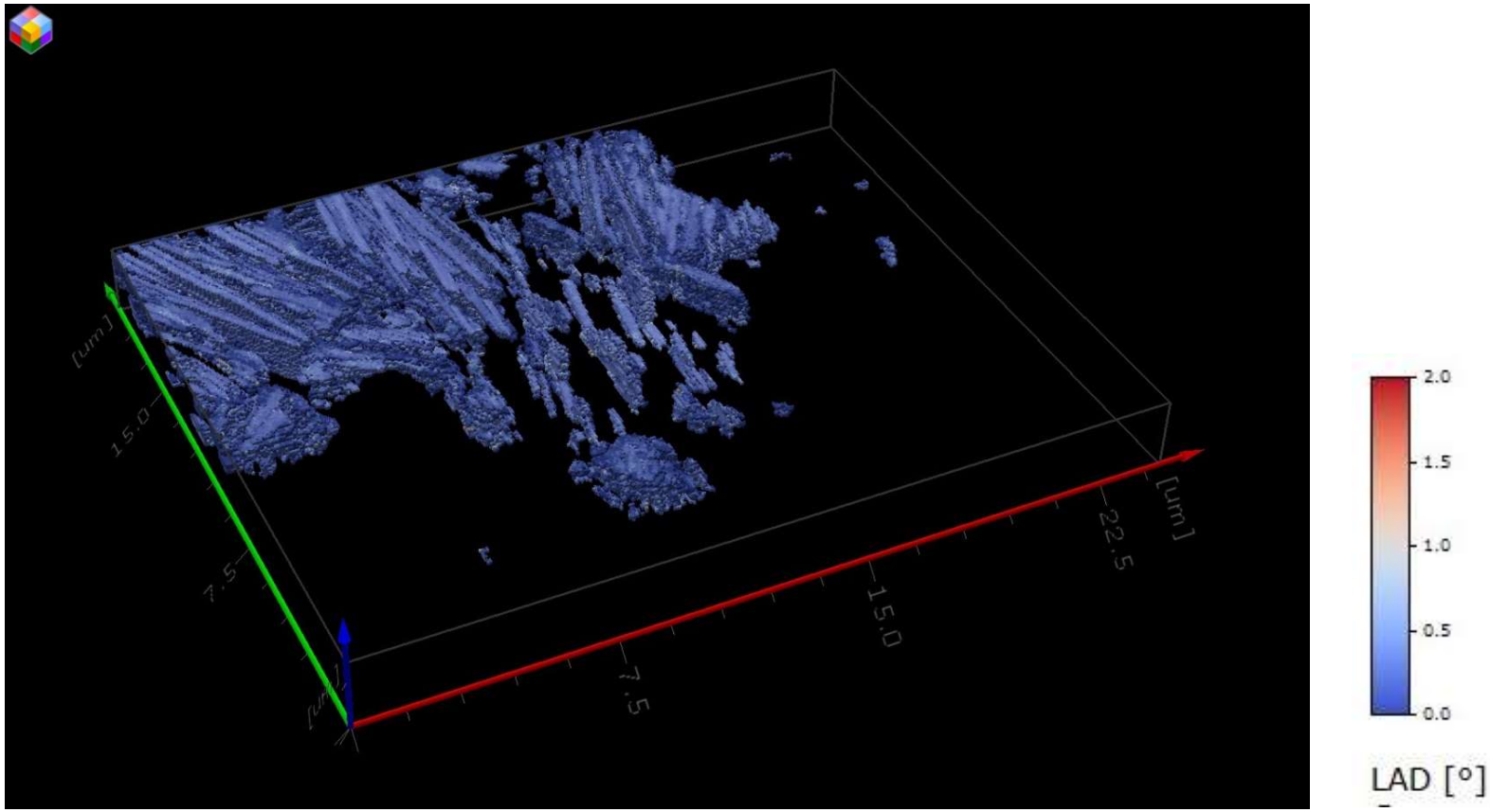




- Data cube after realignment and cropping (on two sides only)

ESPRIT QUBE
Data postprocessing – LAD

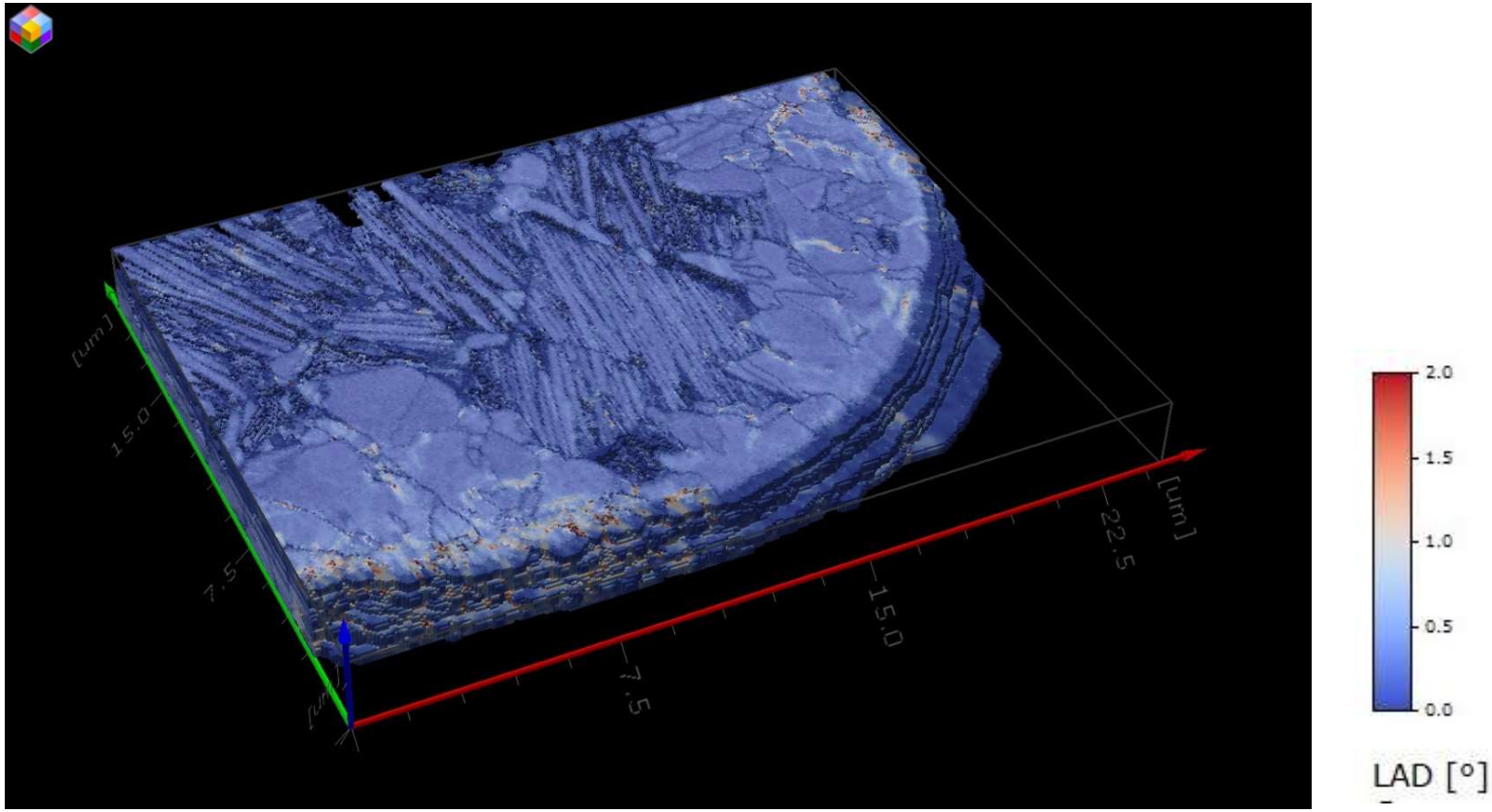
Local Average Disorientation (LAD) inside the intermetallic phase



- Very low LAD values inside the hard intermetallic phase

ESPRIT QUBE
Data postprocessing – LAD

Local Average Disorientation (LAD) inside the CuZn alloy

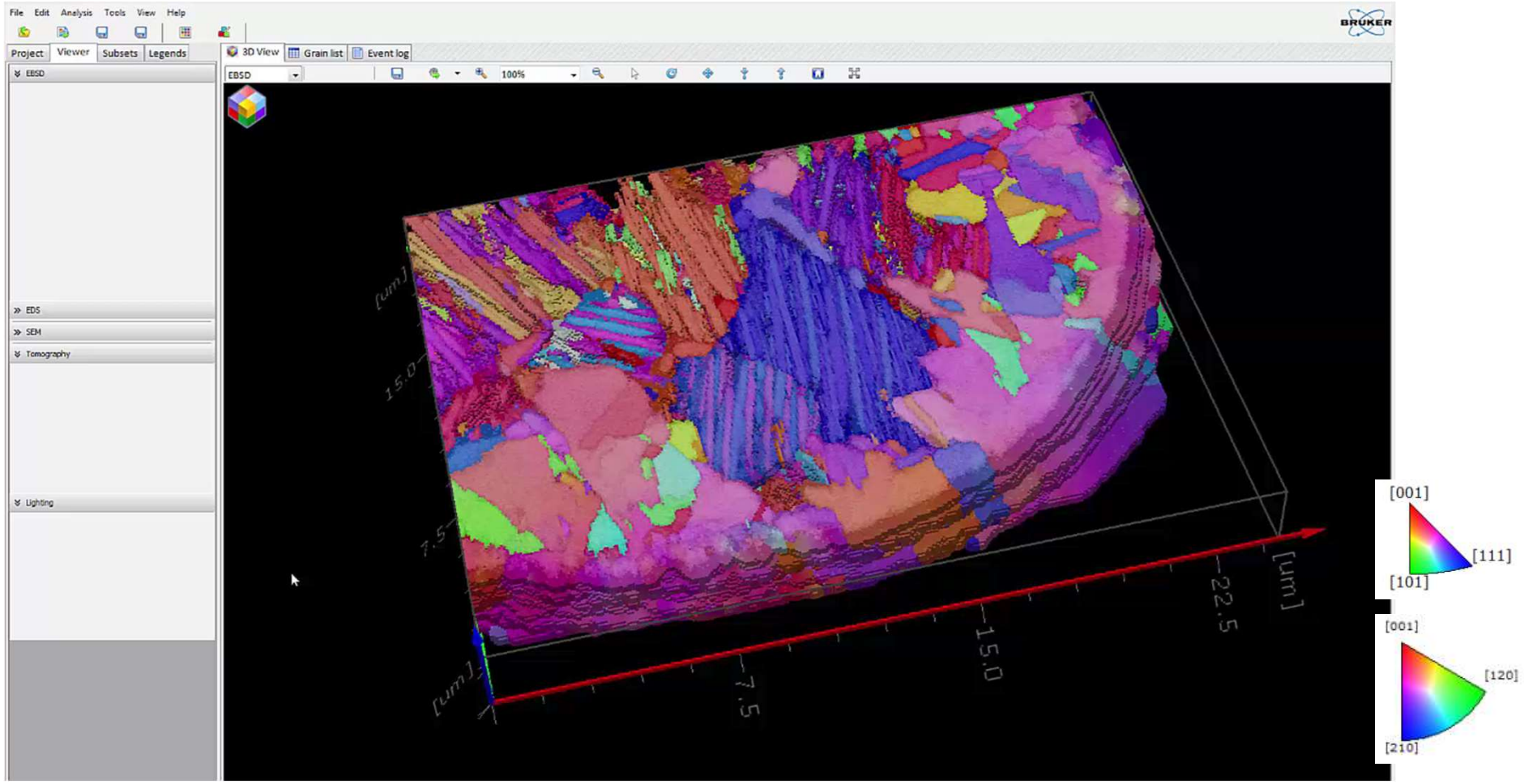


- Significantly higher low LAD values inside the soft CuZn alloy

ESPRIT QUBE

Data postprocessing – Subsetting & visualization

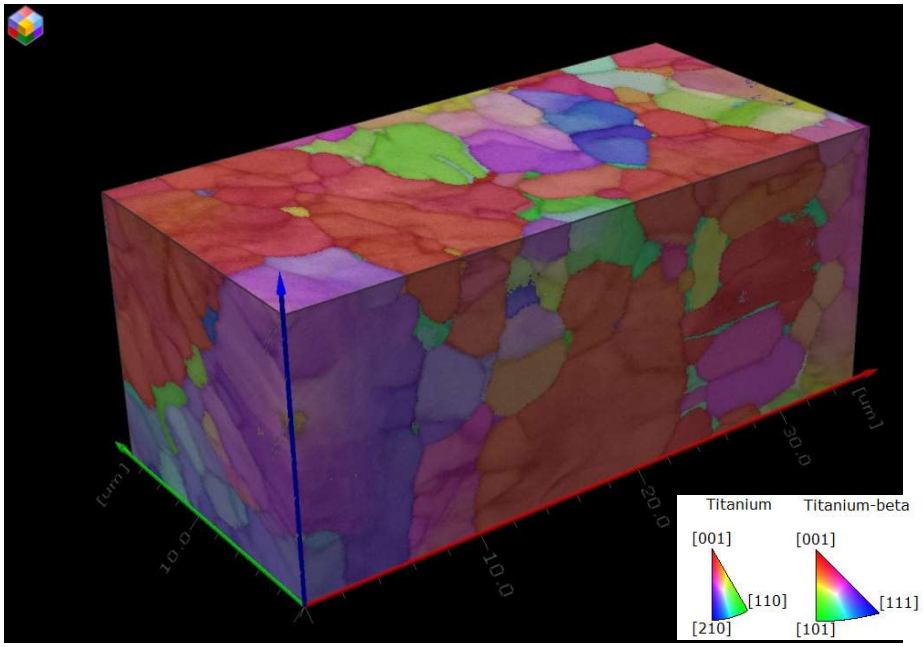
Advanced subsetting and interactive 3D data visualization

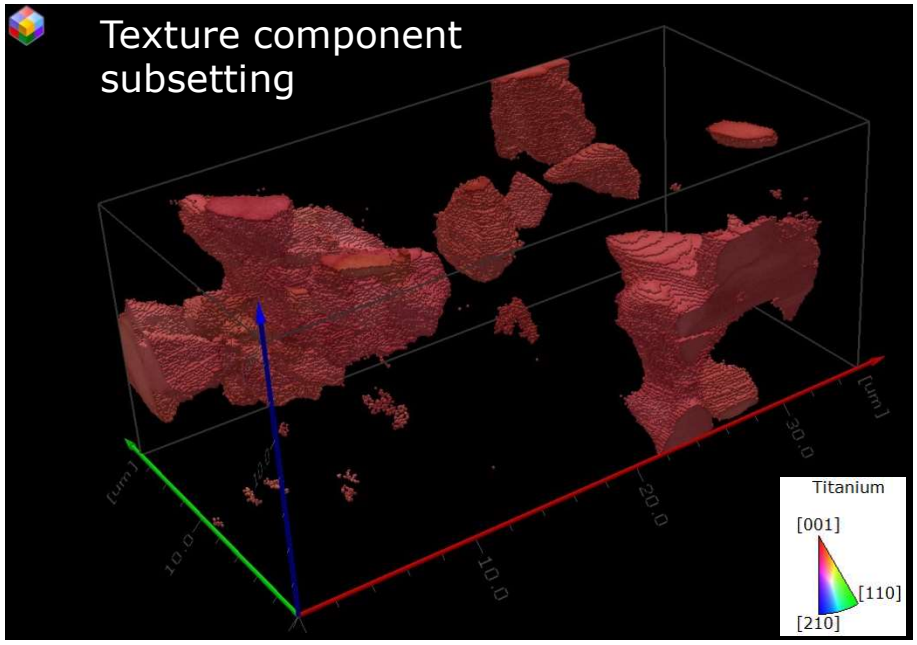


ESPRIT QUBE

Data postprocessing – Subsetting & visualization

- Deformed Ti alloy (alpha and beta phases)
- Large data cube – 245 slices (35x20x20µm^3)
- Serial sectioning using a PFIB-SEM with the standard "rotation" method
- Slice preparation time: ~2min/slice
- 3D EBSD data acquisition time: ~7min/slice

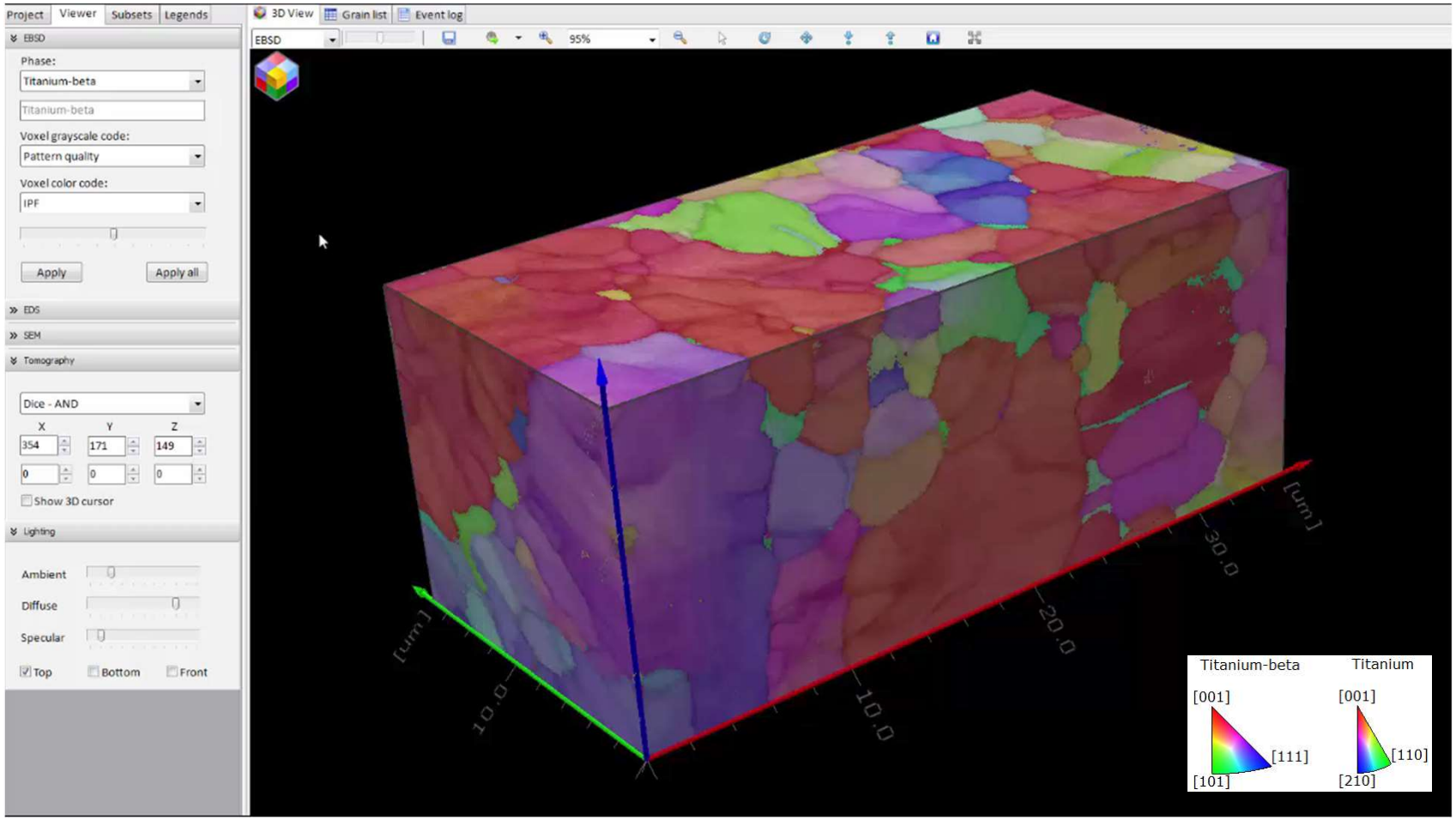




ESPRIT QUBE

Data postprocessing – Subsetting & visualization

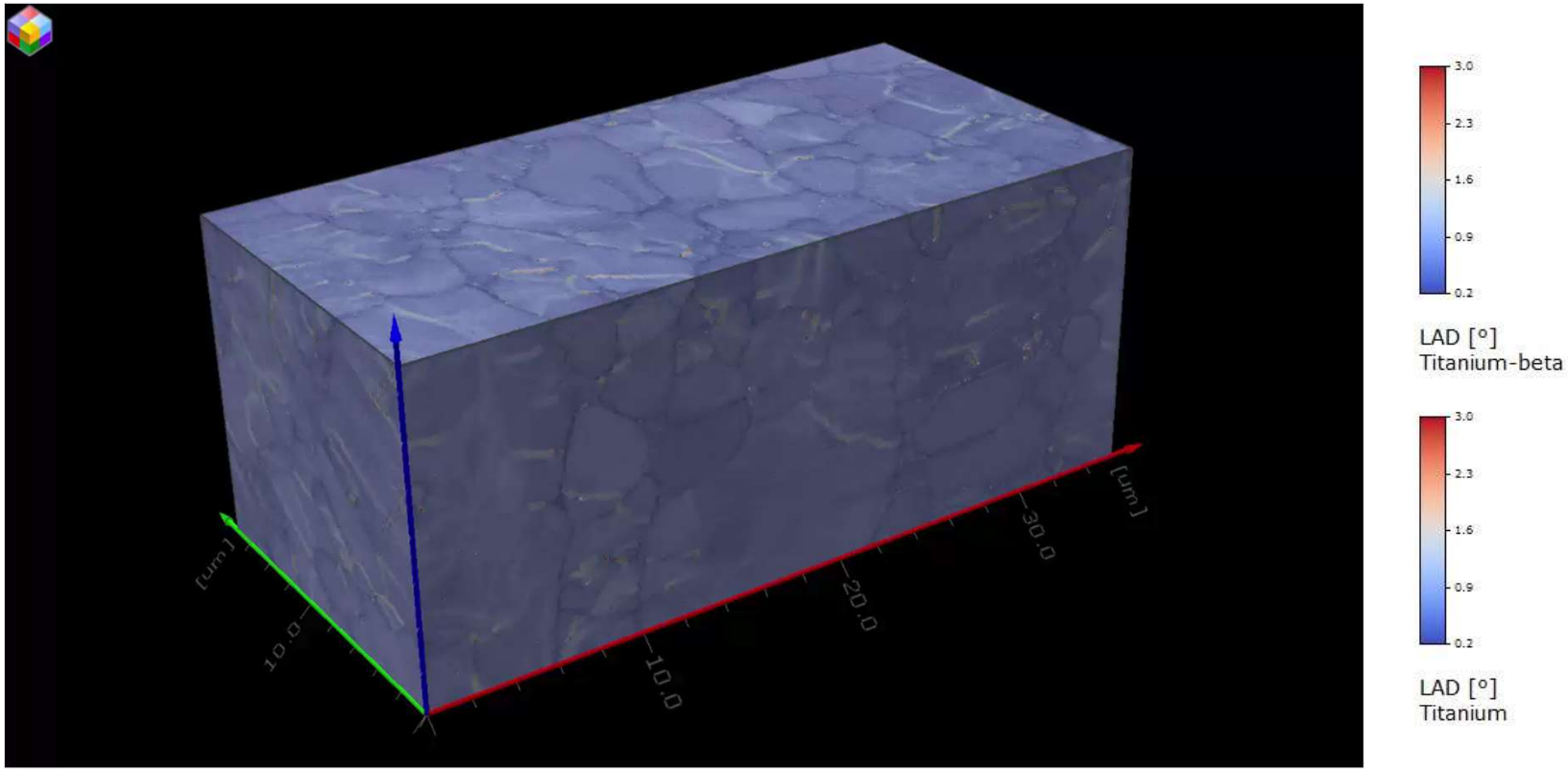
3D EBSD on deformed Ti alloy – phase subsetting



ESPRIT QUBE

Data postprocessing – LAD visualization

3D EBSD on deformed Ti alloy – LAD visualization

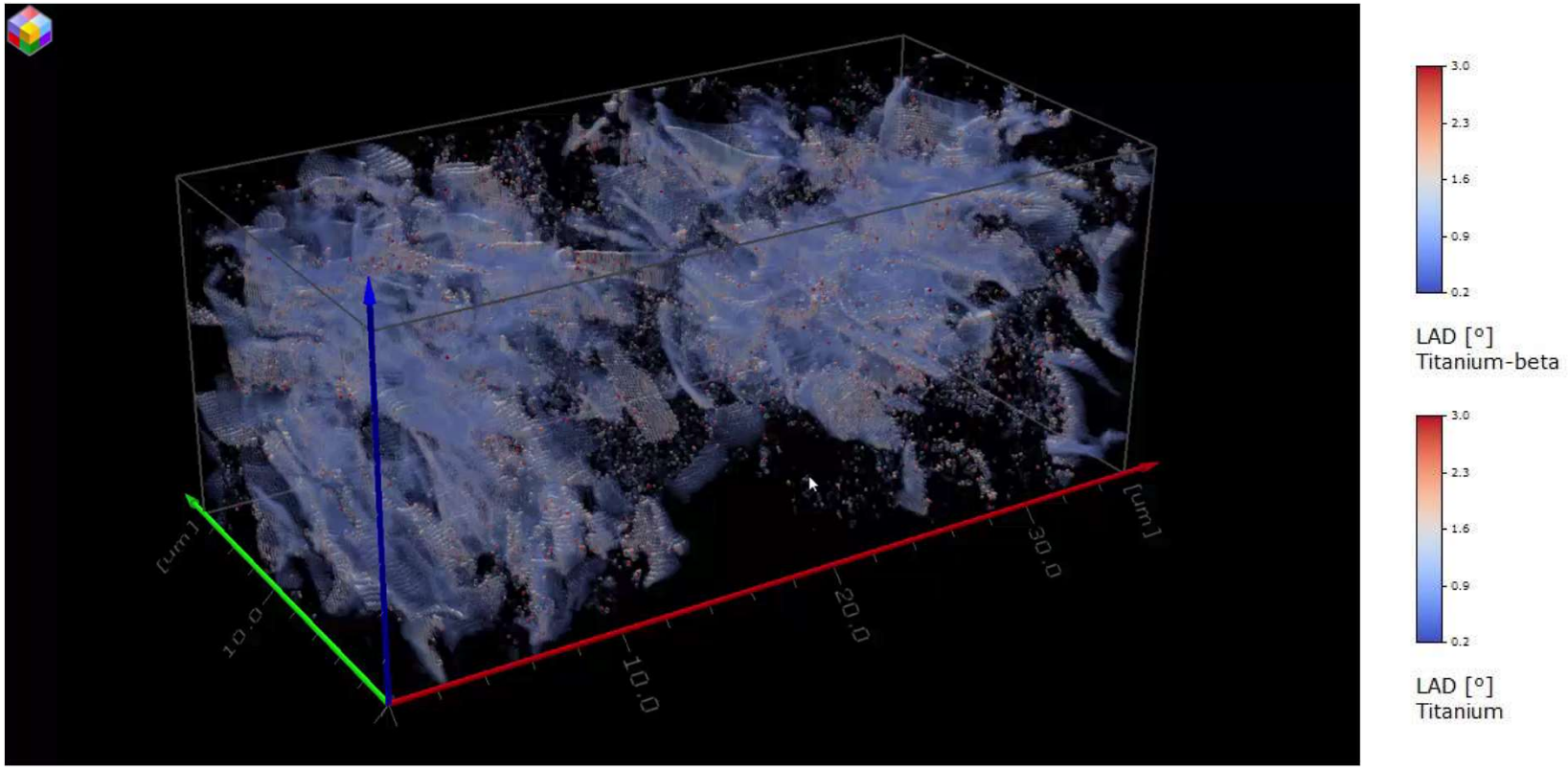


- LAD after slice realignment & cropping + data filtering
- Slice & dice visualization

ESPRIT QUBE

Data postprocessing – LAD visualization

3D EBSD on deformed Ti alloy – LAD visualization

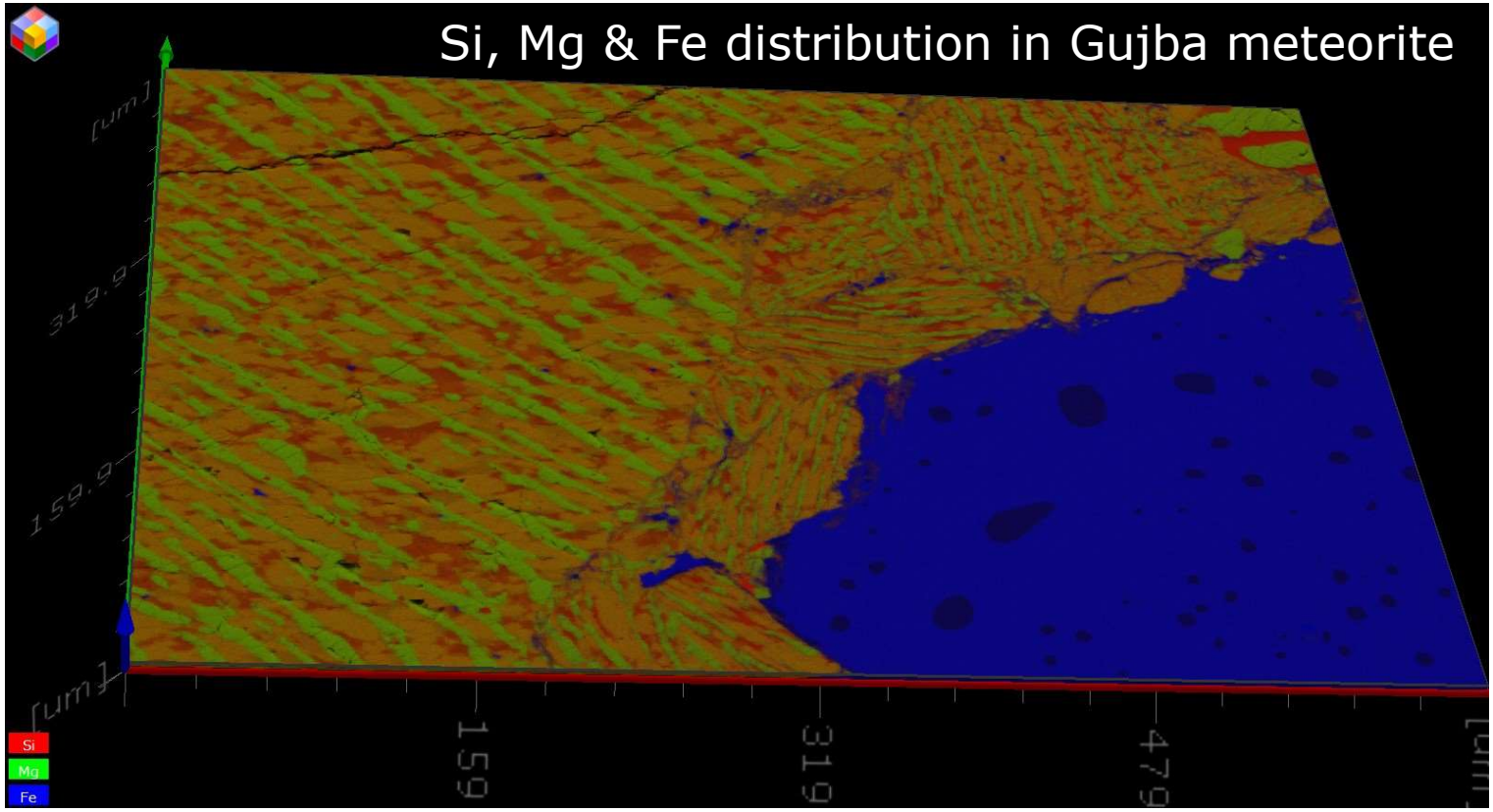


- LAD after slice realignment & cropping + data filtering
- Advanced visualization: Transparency increases with decreasing LAD values

ESPRIT QUBE
3D EDS – visualization

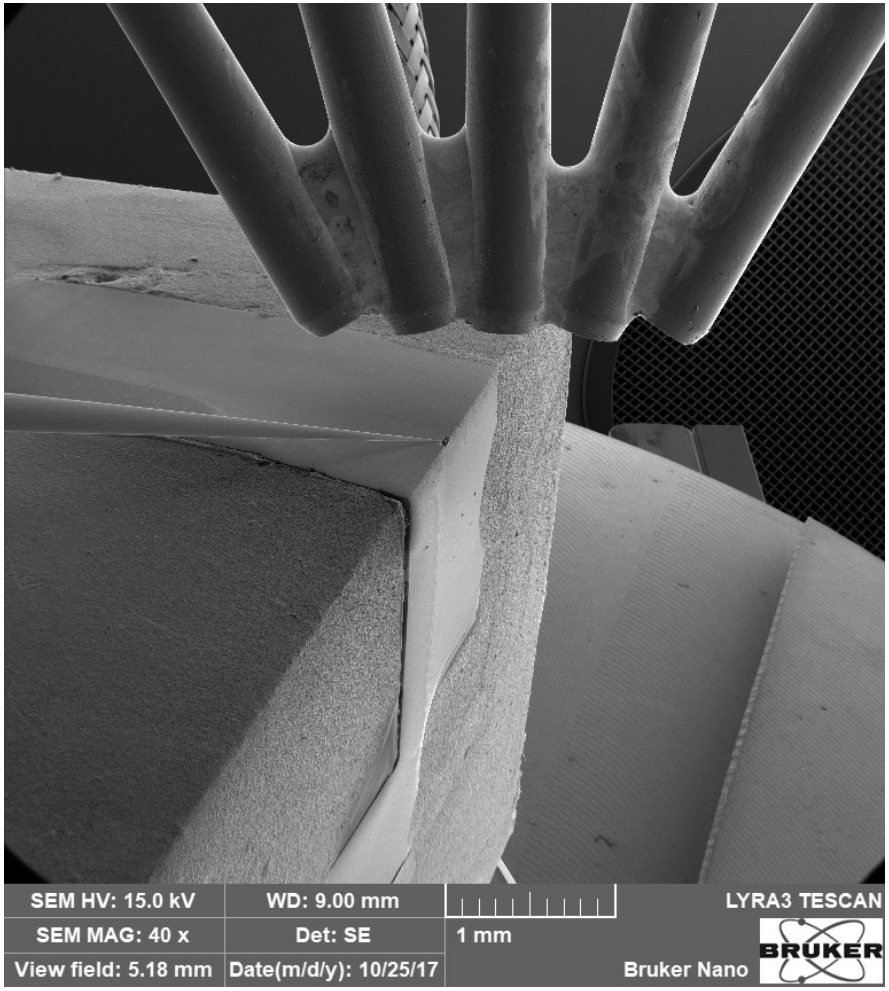
Extensive data subsetting with visual, interactive data exploration:

- EDS based subsetting (counts & quantified data)



ESPRIT QUBE

3D EBSD data processing: application example on stainless steel



stainless steel

Phases:
Ferrite, Austenite, Sigma

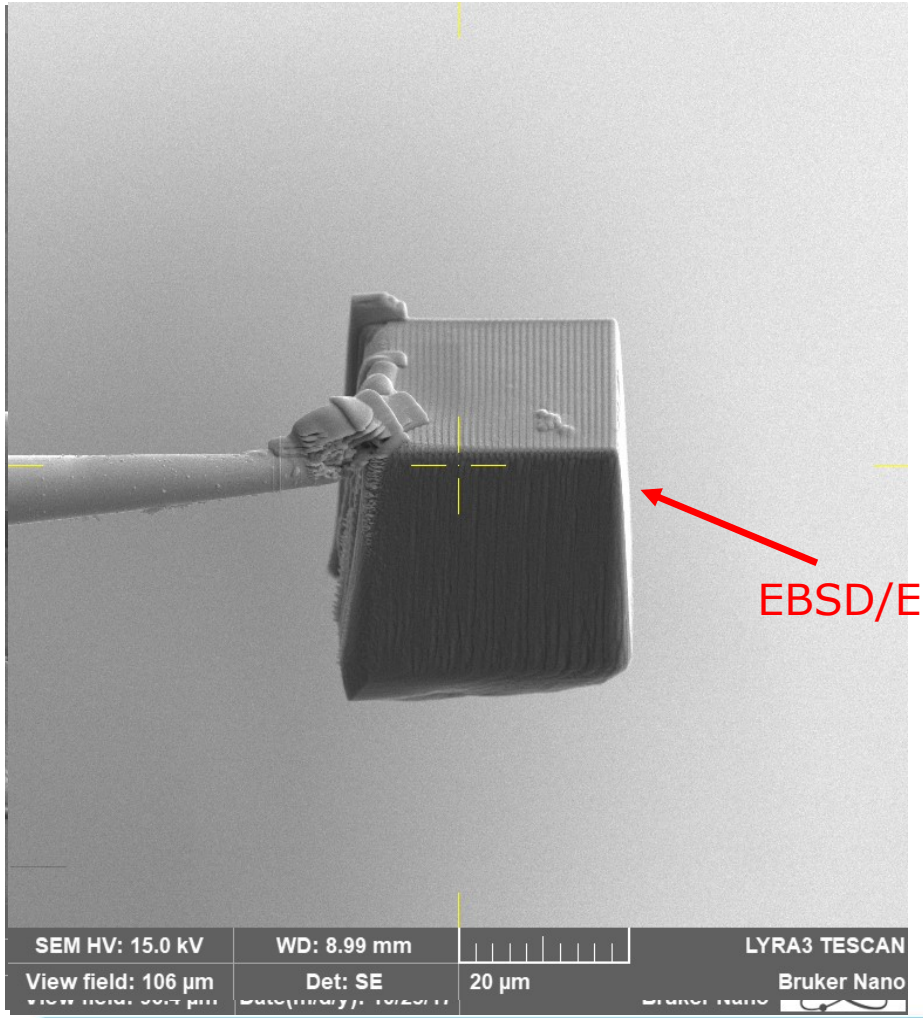
Parameters:

FIB (Ga+)
30 kV, 1.5nA

EBSD
e-Flash^{FS}
EBSP quality: 160*120 px
Exposure time 3ms

ESPRIT QUBE

3D EBSD data processing: application example on stainless steel

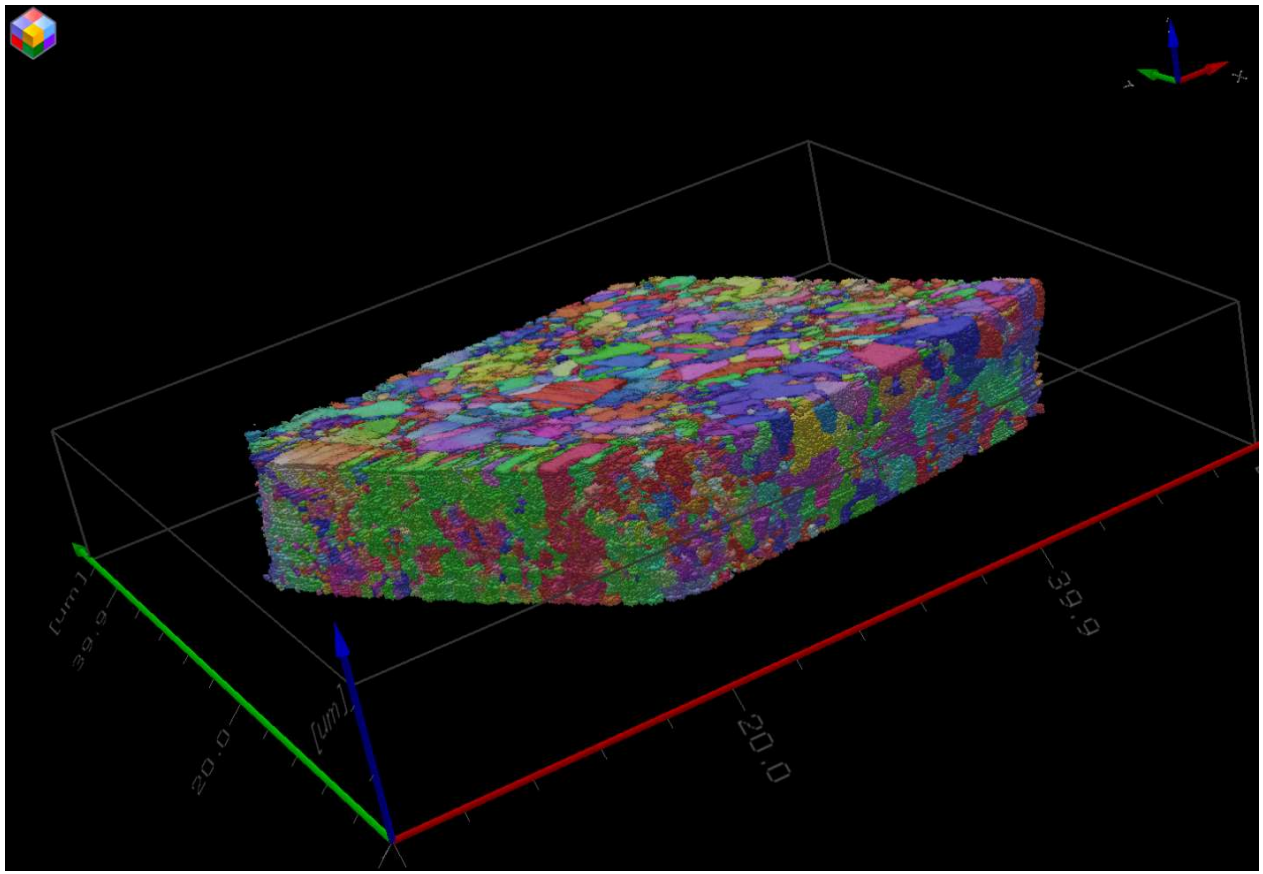


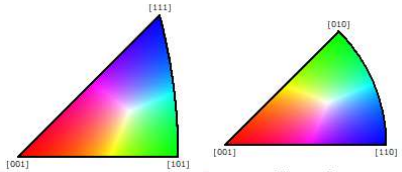
EBSD/EDS measurement

FIB setup

1. Trenches
2. undercut
3. Cube lift out

ESPRIT QUBE
3D EBSD processing: data realignment





stainless steel
90 slices

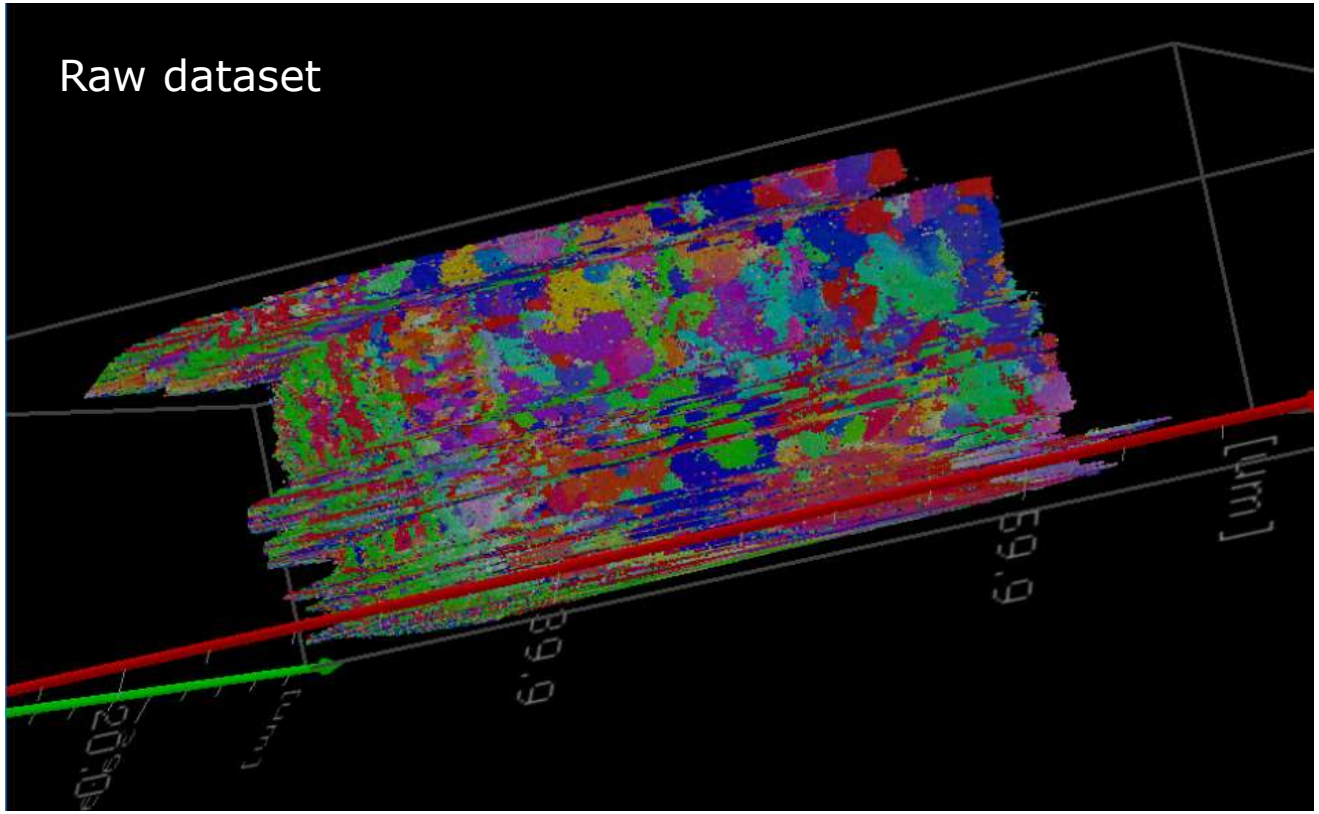
FIB (Ga^+)
100 nm slice
1.5nA, 30kV
7 min

EBSD:
e-Flash^{FS}
160*120px resolution
100nm step size
~27*30µm
320 fps (3 min)

~18 M pixels

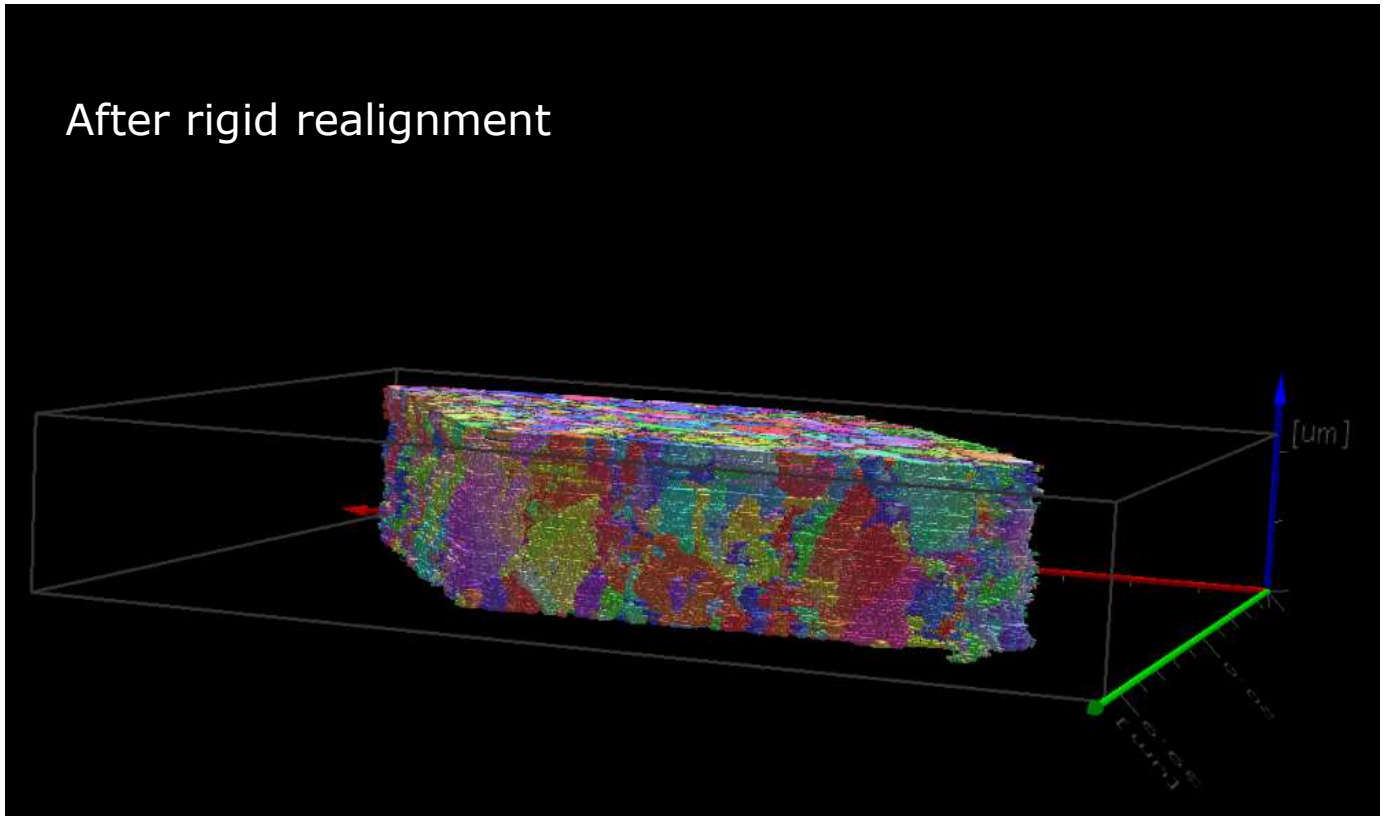
ESPRIT QUBE
3D EBSD processing: data realignment

- Dataset editing: rigid realignment



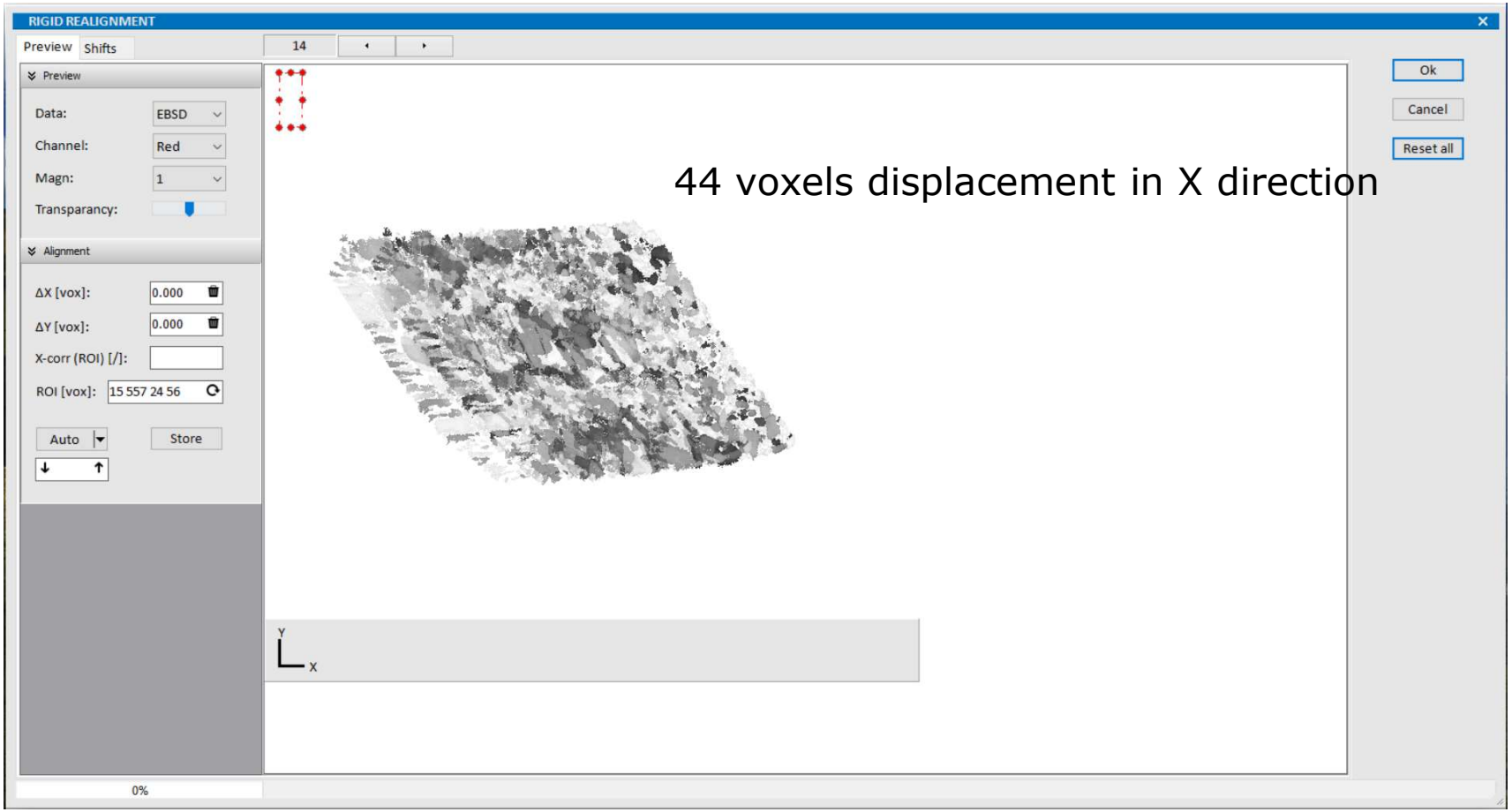
ESPRIT QUBE
3D EBSD processing: data realignment

- Dataset editing: rigid realignment



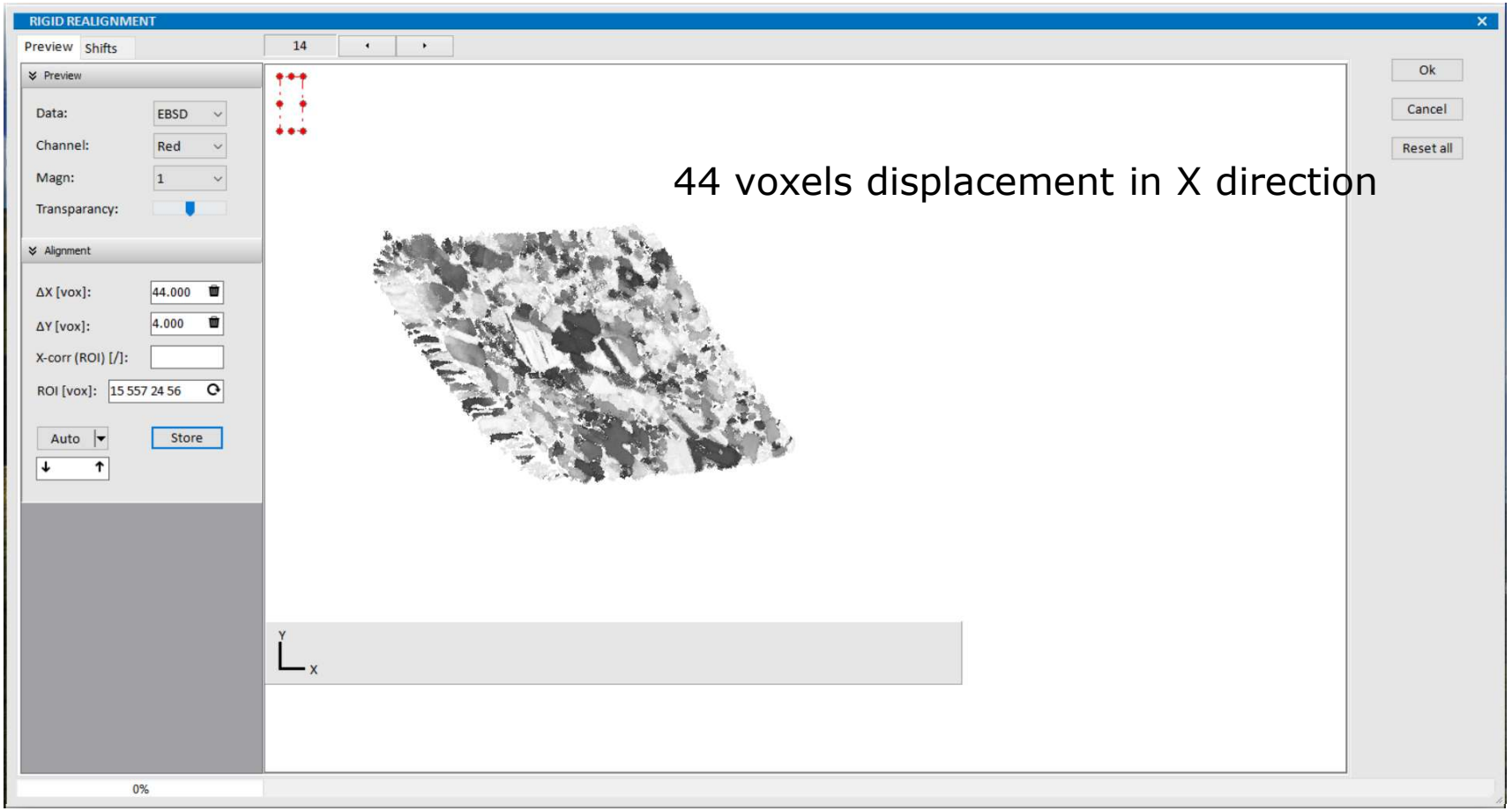
ESPRIT QUBE

3D EBSD processing: data realignment



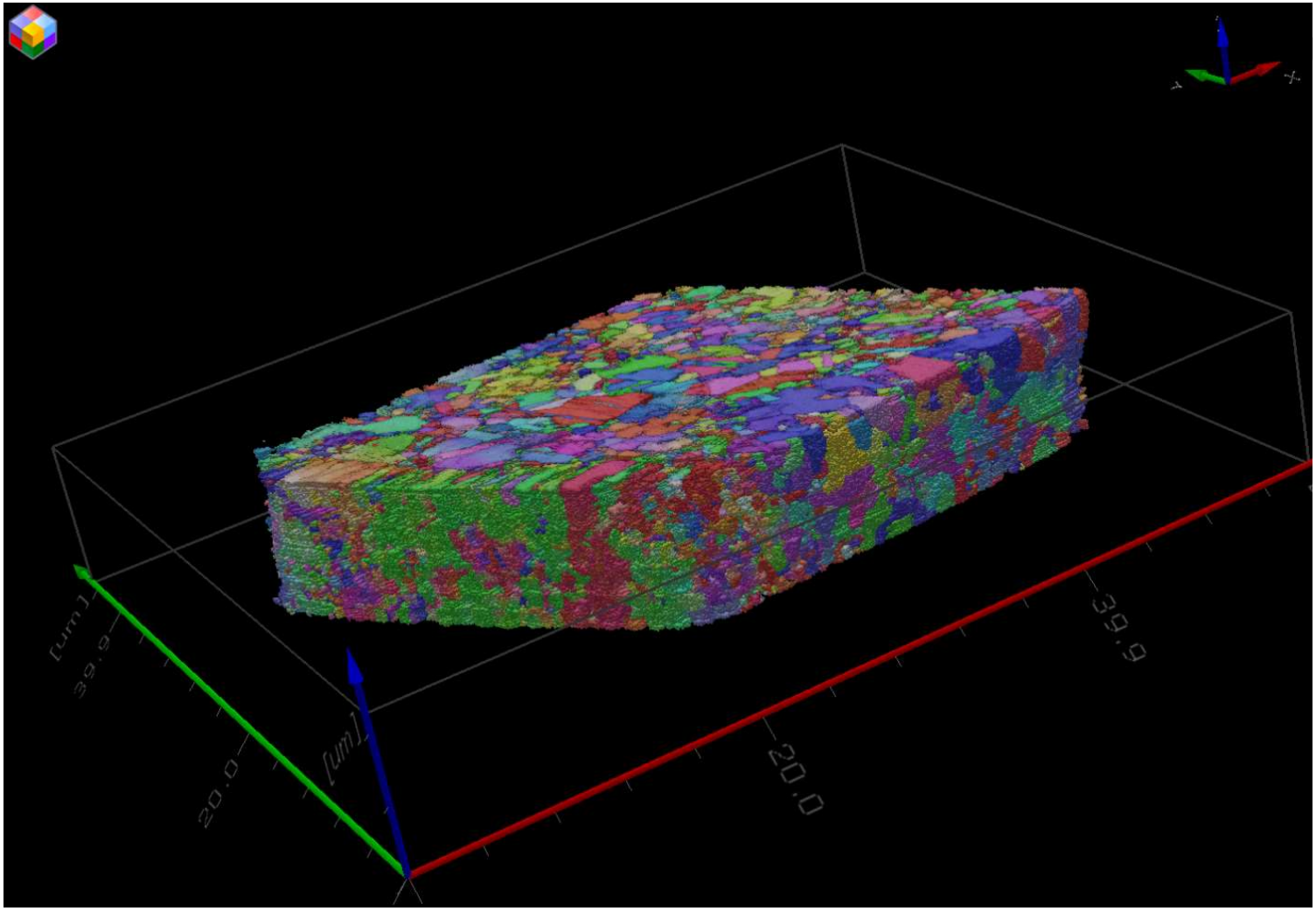
ESPRIT QUBE

3D EBSD processing: data realignment



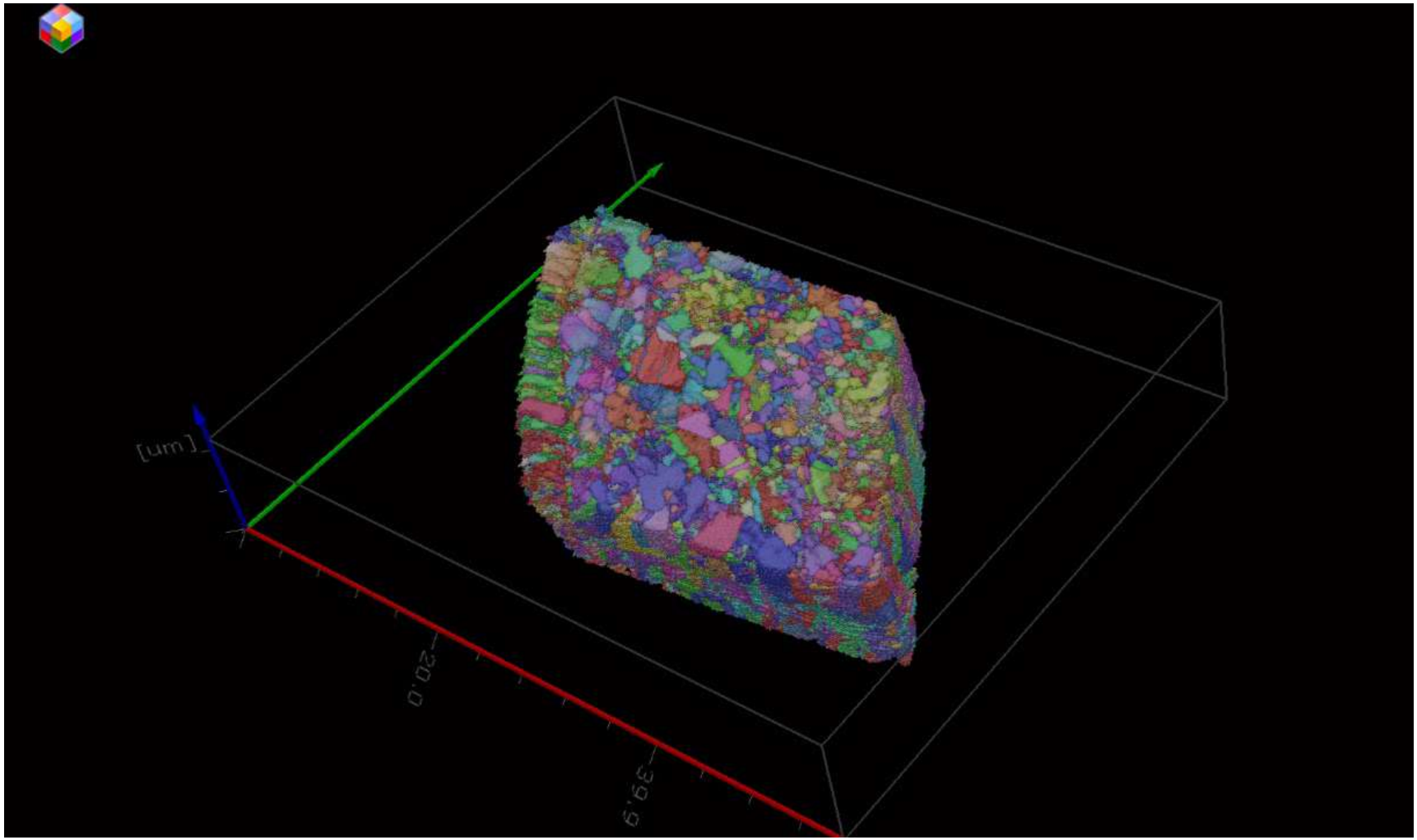
ESPRIT QUBE

3D EBSD processing: data realignment



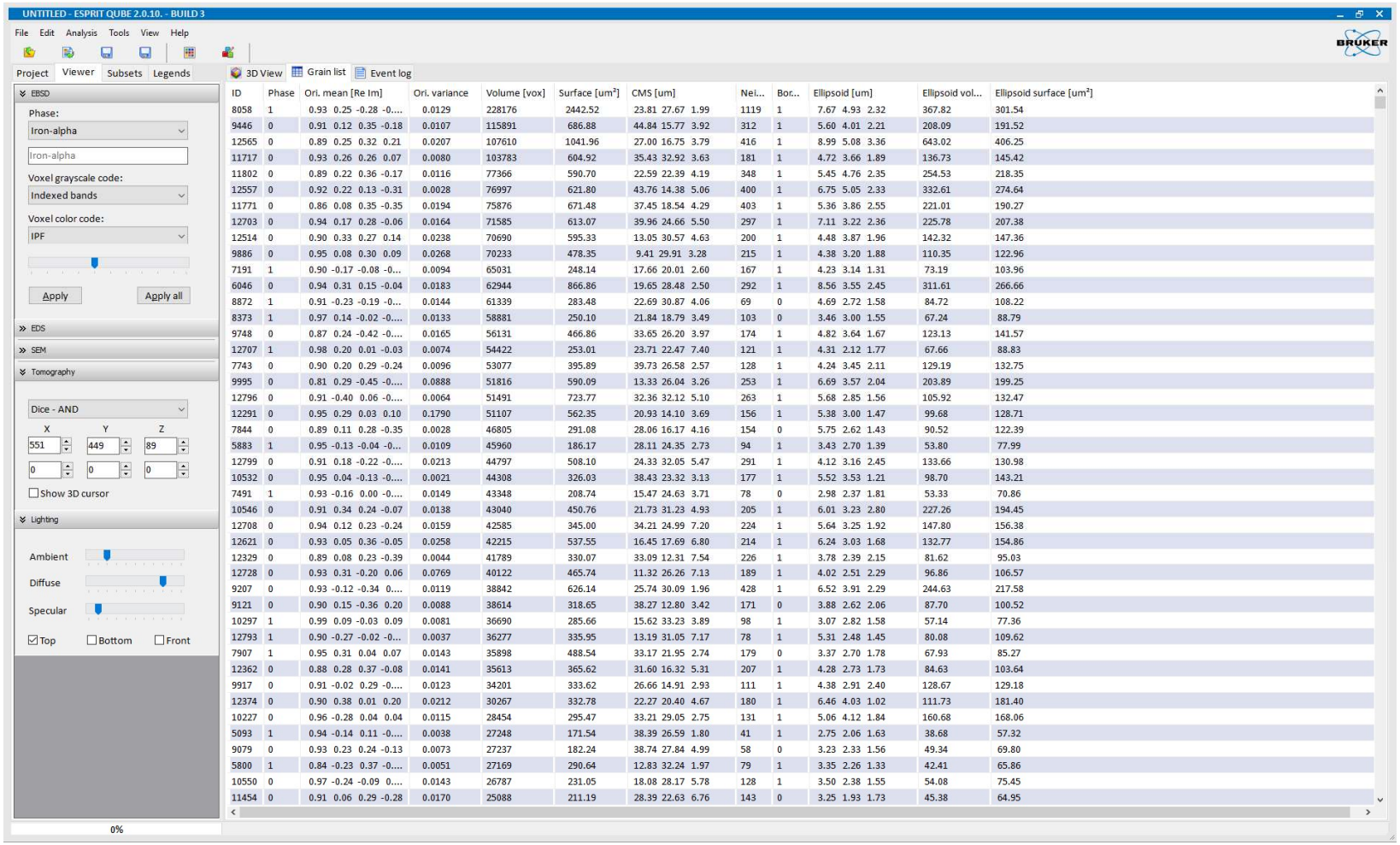
ESPRIT QUBE

3D EBSD processing: data filtering



ESPRIT QUBE

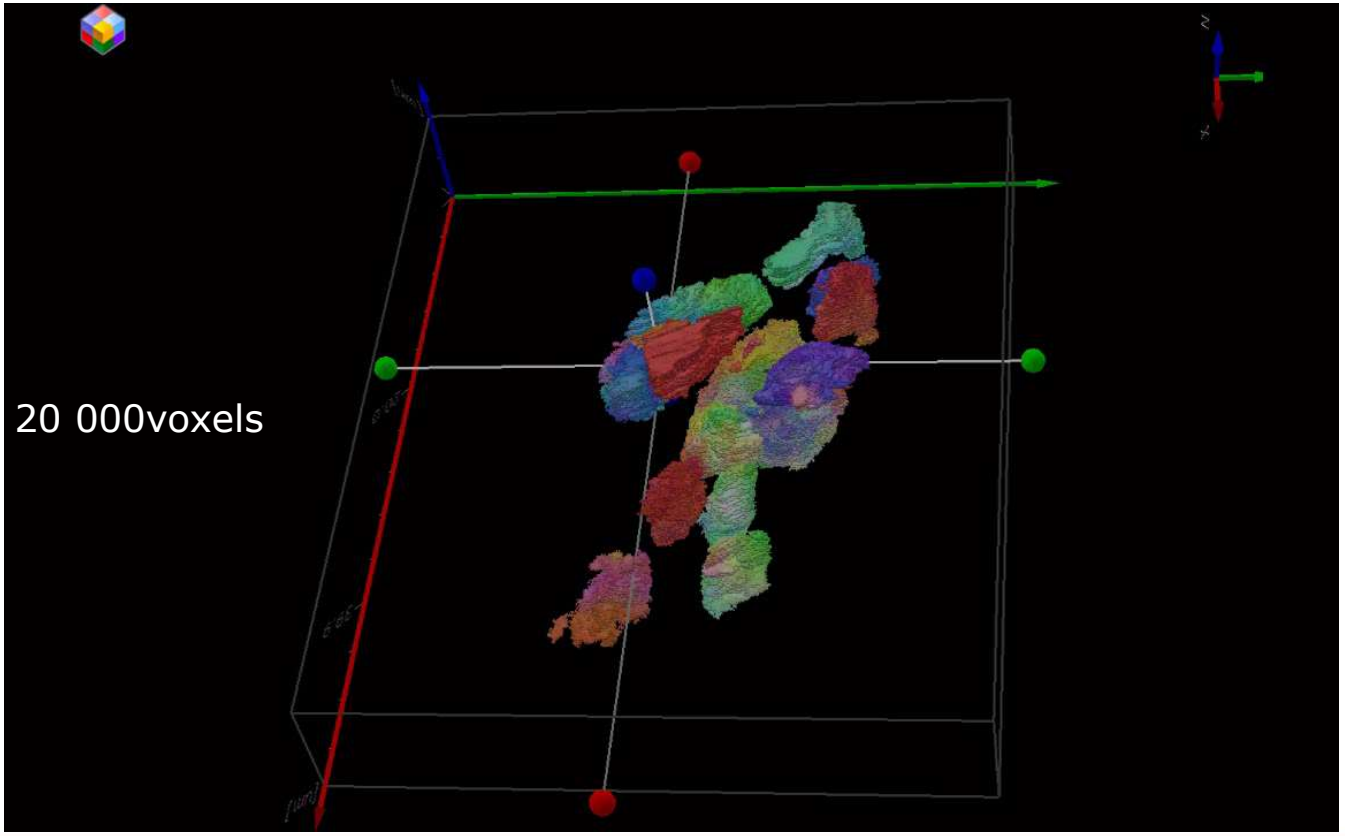
3D EBSD processing: grain list



ESPRIT QUBE
3D EBSD processing

- Extensive data subsetting with visual, interactive data exploration:
  - Subsetting based grain metrics (volume, area, shape, neighbours, etc.)

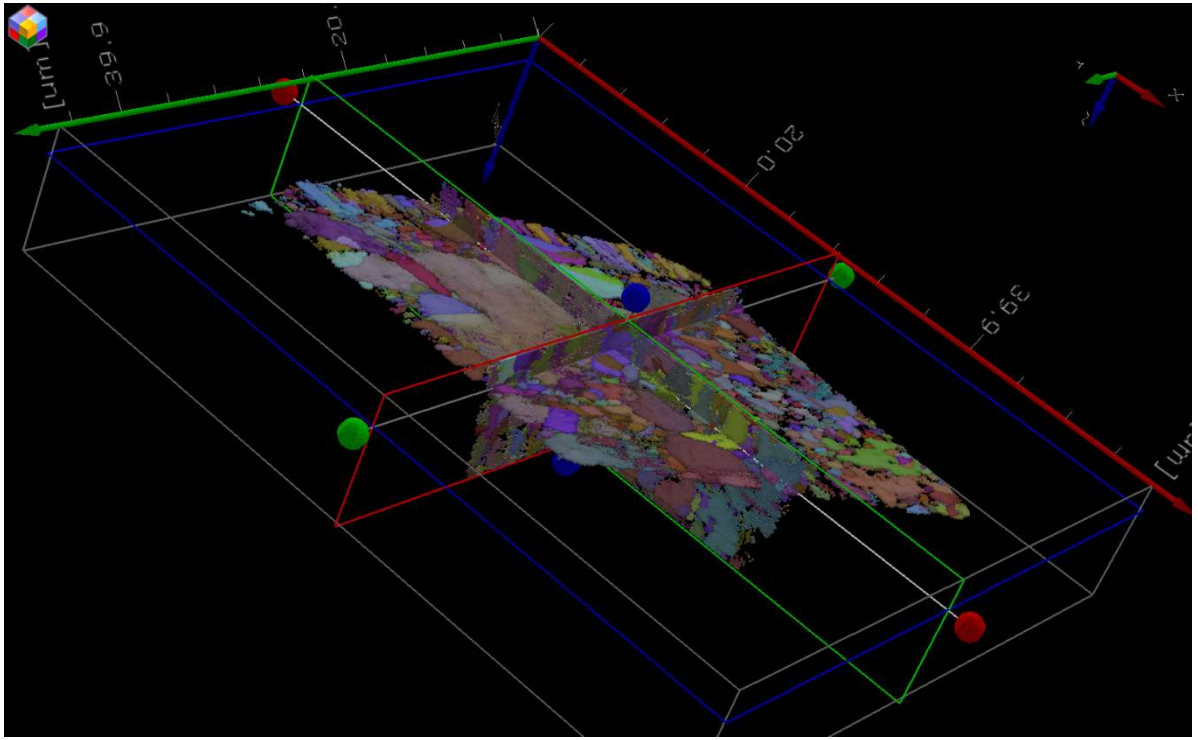
Grains containing > 20 000voxels



ESPRIT QUBE
3D EBSD processing

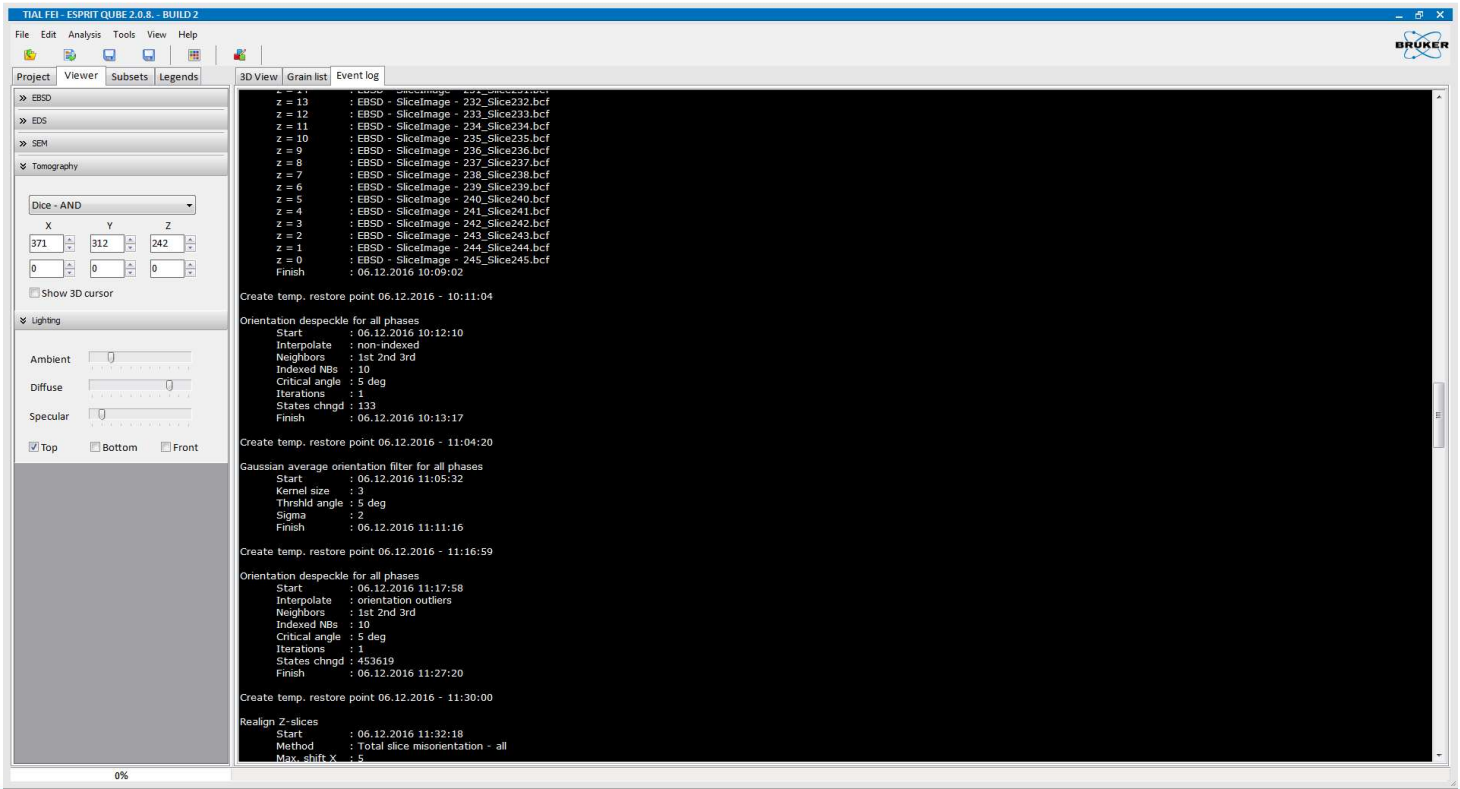
Main features:

- Visualization, slicing & dicing of EBSD data cubes



ESPRIT QUBE
3D EBSD processing

- "Event log" record all processing steps applied to the data cube *:* allowing to go back to any previous step in the data processing!

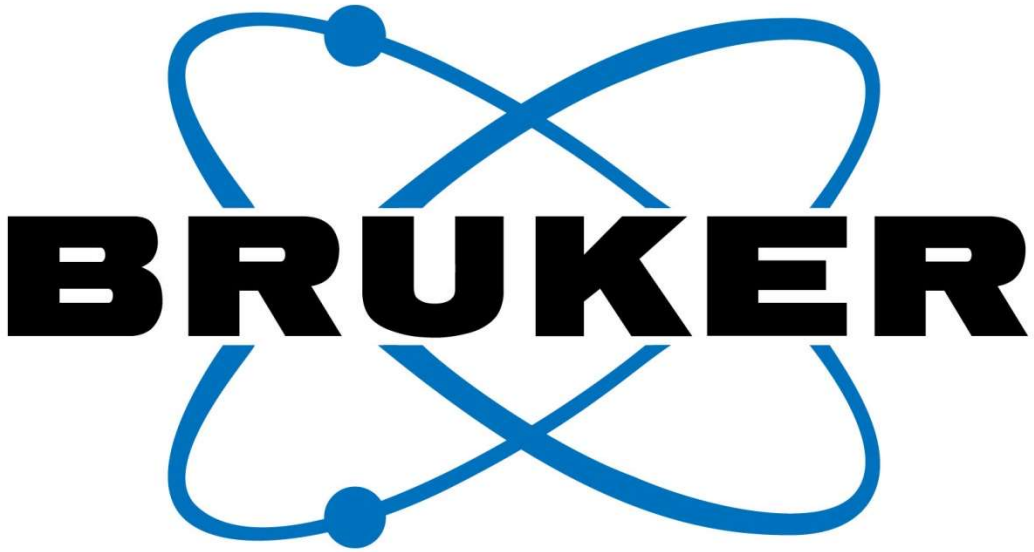


ESPRIT QUBE

Advanced 3D data postprocessing & visualization

Summary

- Unit **Qu**aternion based core
- Multiple slice realignment options
- Advanced data postprocessing capabilities (for crystal plasticity studies)
- Multitude of EBSD data subsetting options
- 3D EDS subsetting and visualization
- 3D EBSD/EDS data cube simulation
- Supports multiple file formats (import & export)



Innovation with Integrity