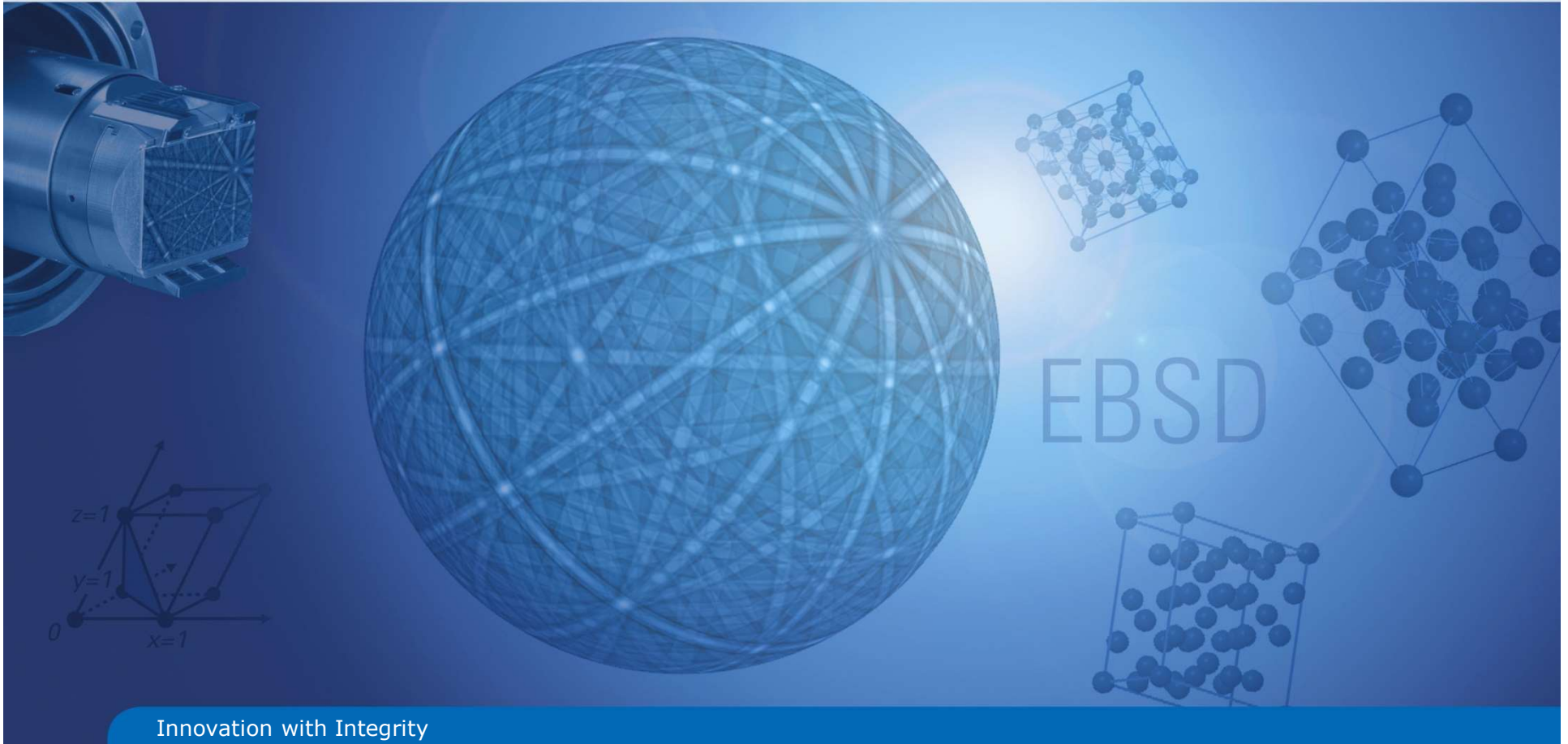


Advanced Material Characterization by Combined 3D EBSD/EDS Measurements and Post Processing with ESPRIT QUBE



Free online webinar – December 7th 2017
Bruker Nano Analytics, Germany



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



Max-Planck-Institut
für Eisenforschung GmbH

S. Zaefferer, P. Konijnenberg

D. An, G. Nayeri, A. Khorashadizadeh



- 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-rigid slice alignment
- Possibilities & Limitations

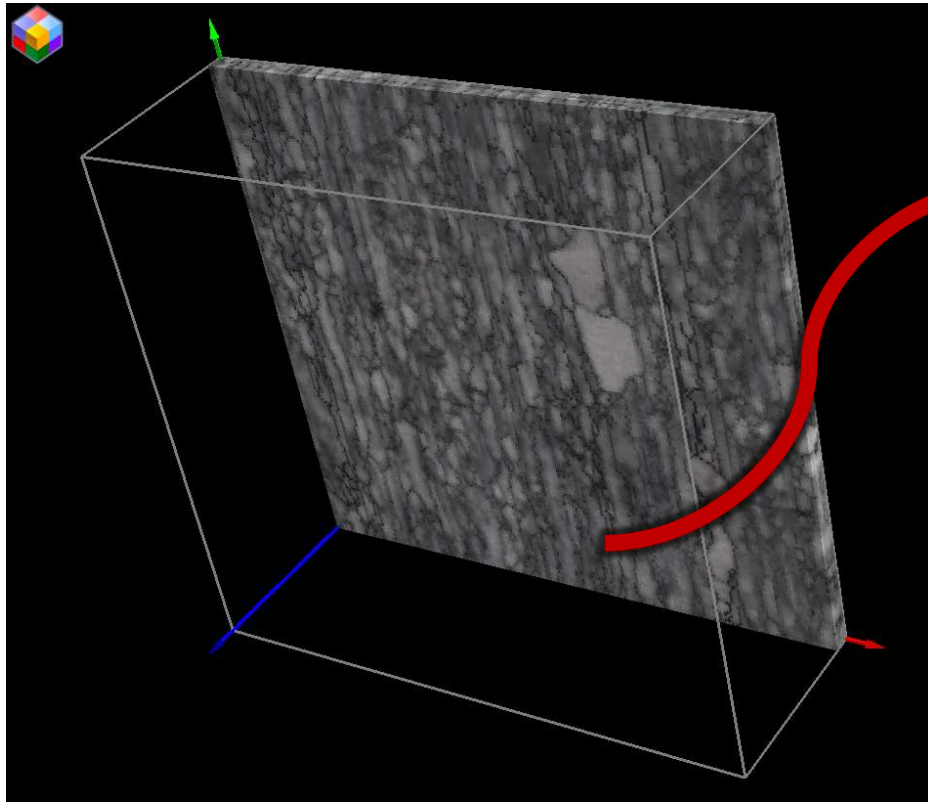




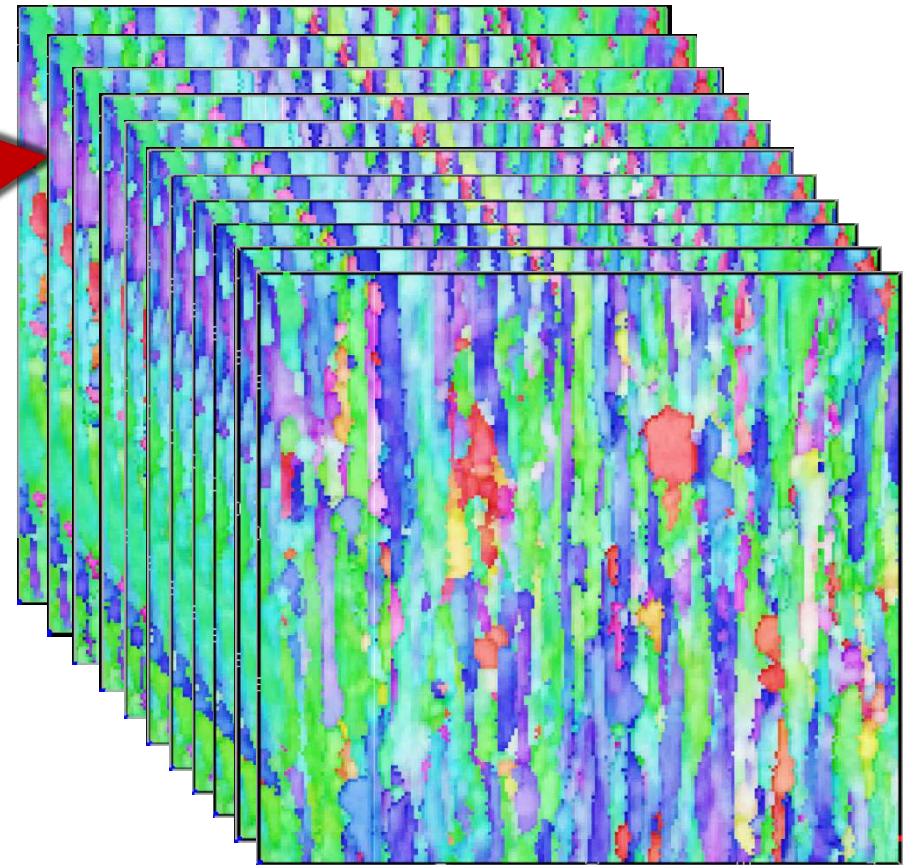
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What is 3D orientation microscopy or „3D EBSD“?



Original material



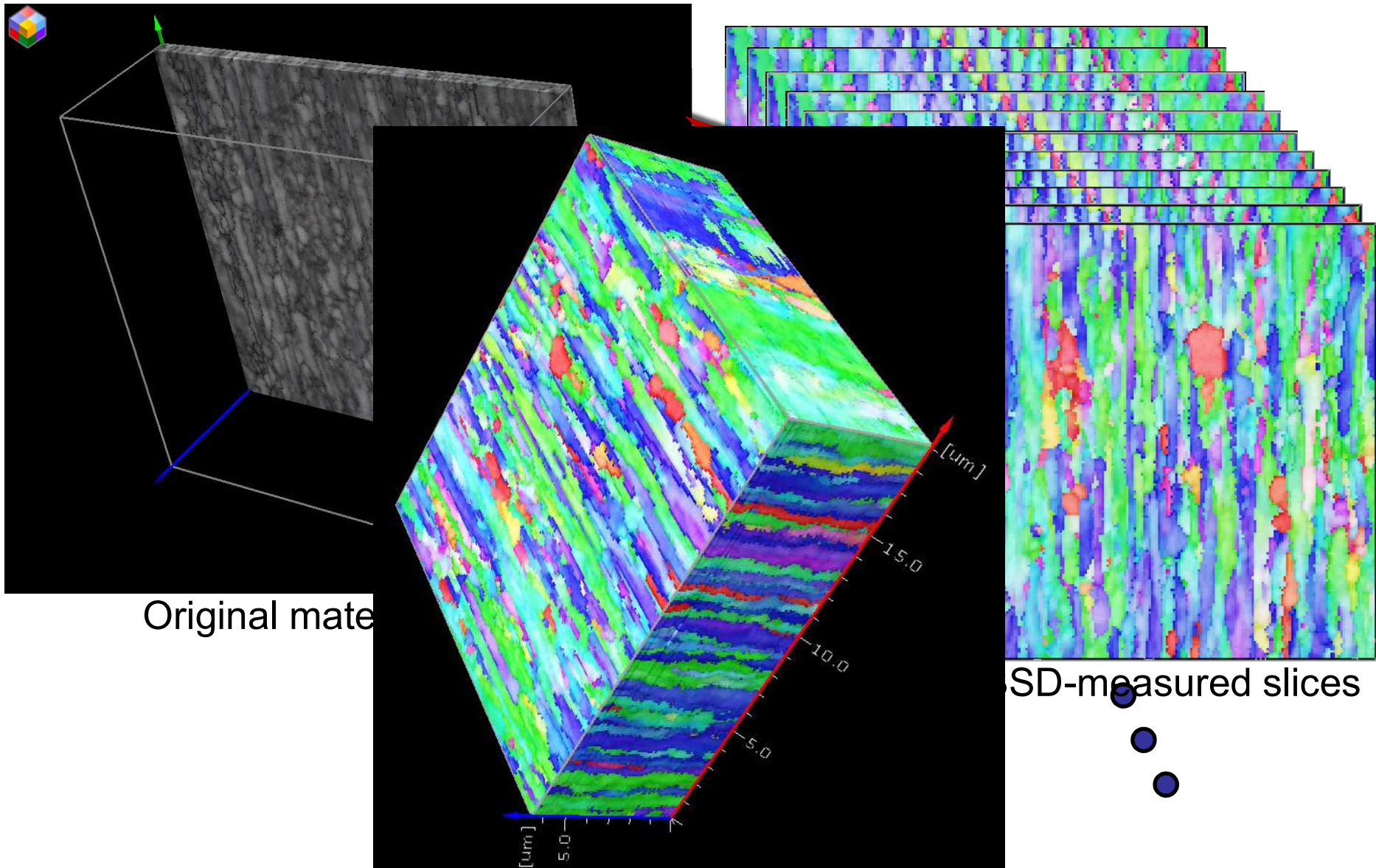
EBSD-measured slices



Reconstructed microstructure



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



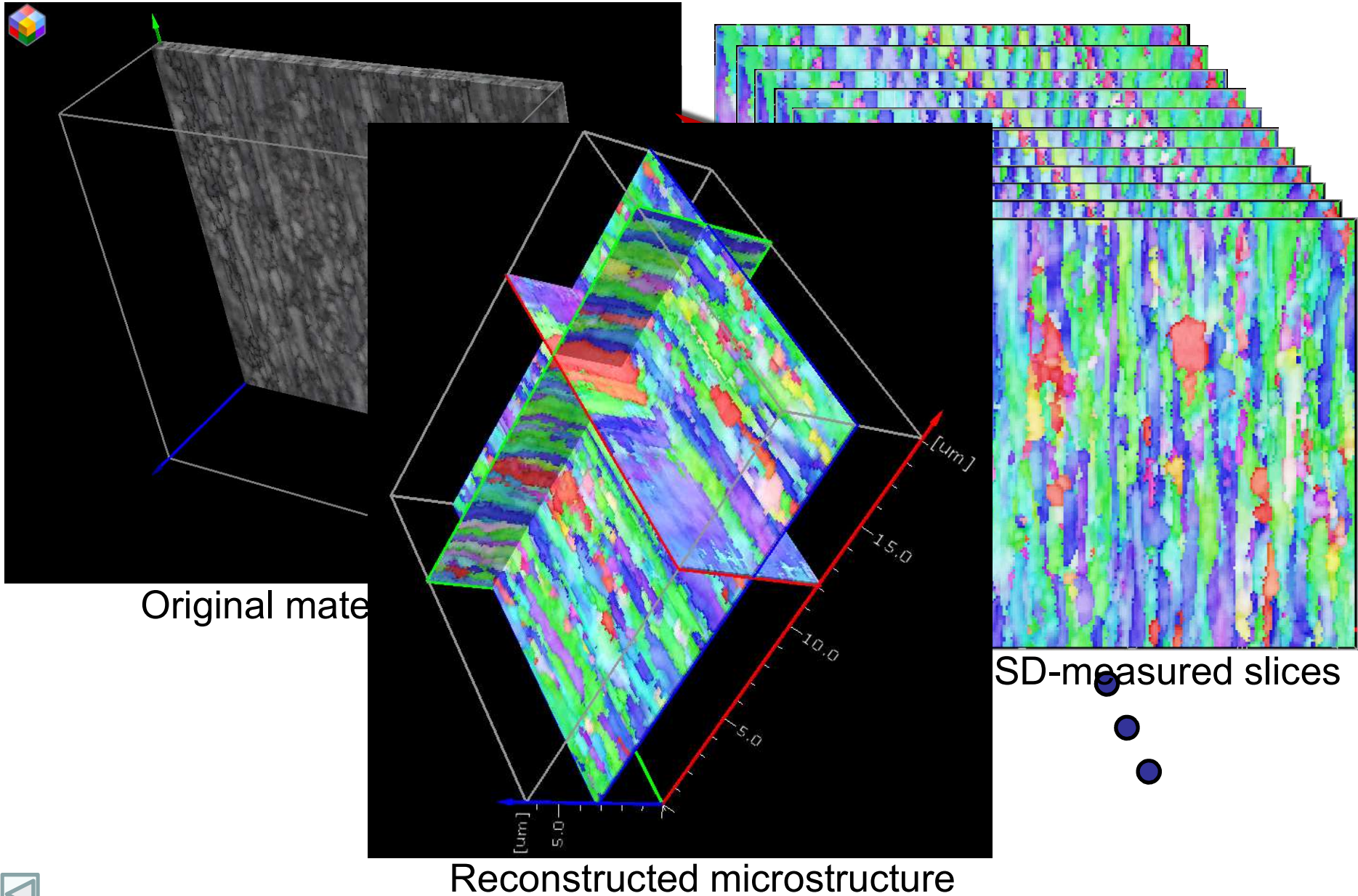
Original material

Reconstructed microstructure

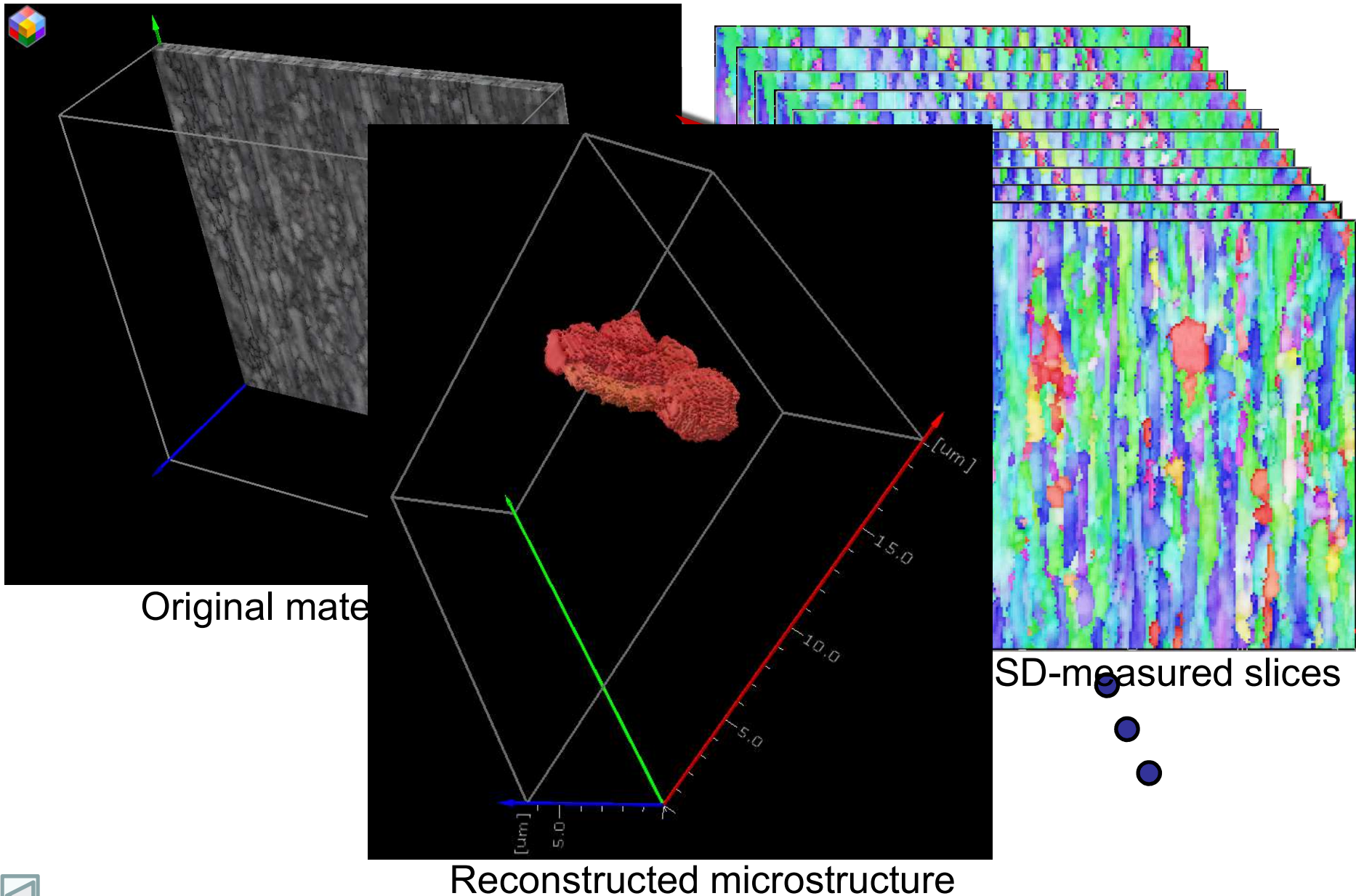
SD-measured slices



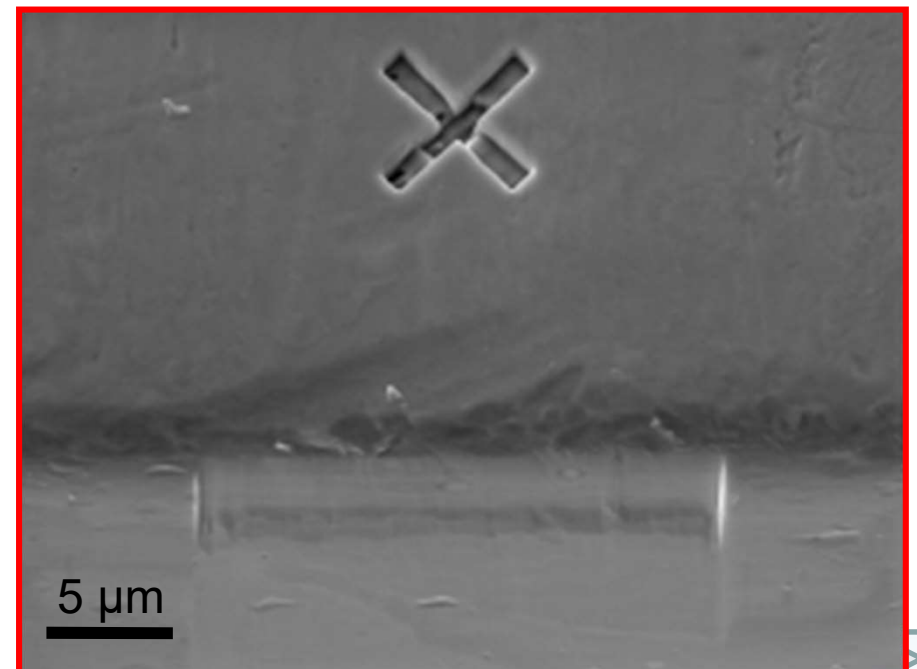
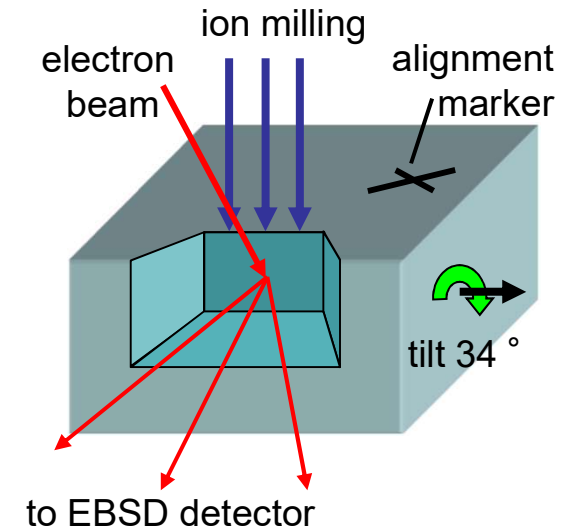
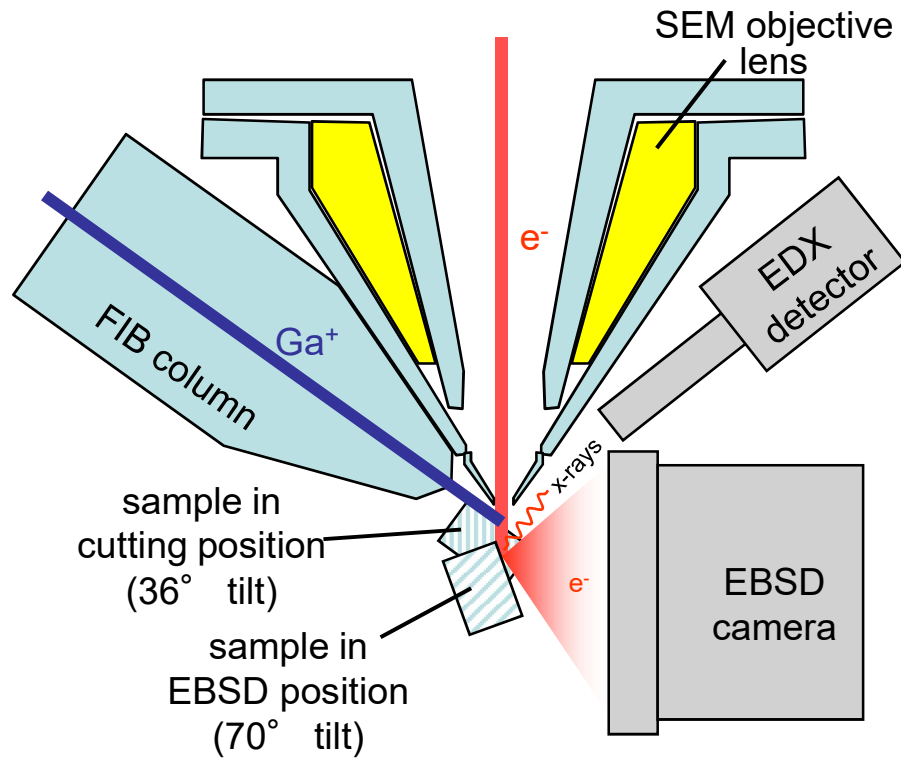
What is 3D orientation microscopy or „3D EBSD“?



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



How to do 3D EBSD?



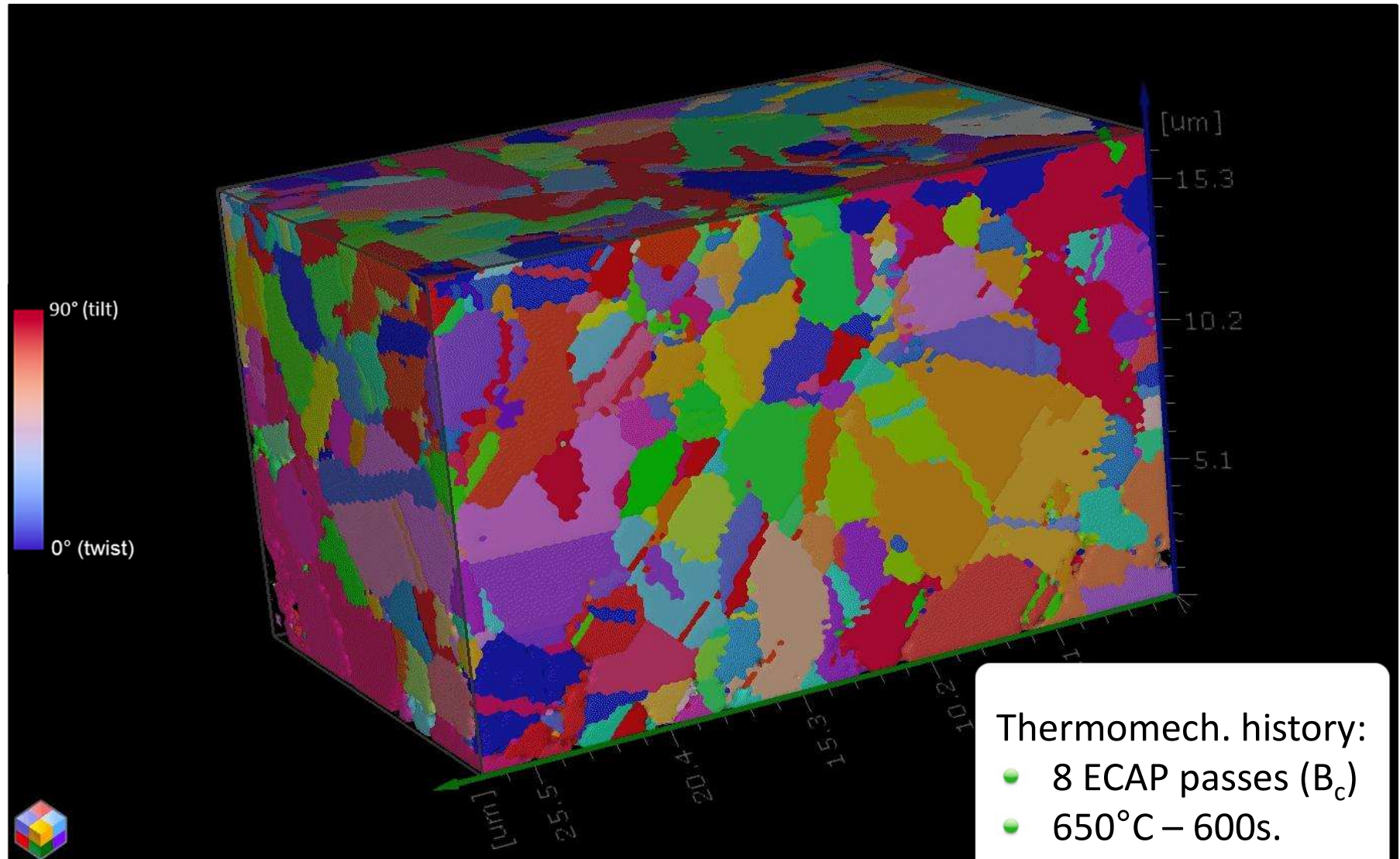
"tilt set-up"
Zaefferer, Wright, Raabe,
Mat. Trans. A (2008)



Example: grain boundaries in Cu-0.17wt%Zr



Misorientation: $60^\circ \langle 111 \rangle \pm 5^\circ \langle \dots \rangle$

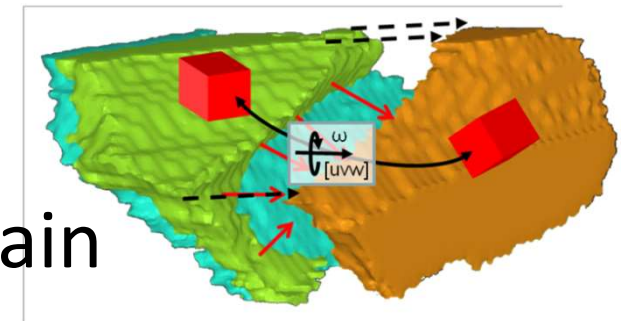
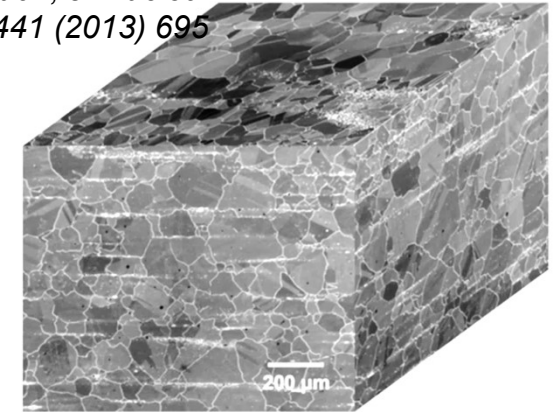


Why 3D? What do we want/need to find out?

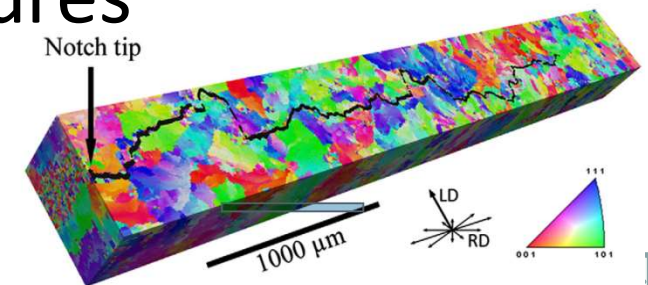


- Size, shape and arrangement of grains and phases in complex microstructures
- 3D-arrangement of defects (interfaces, dislocation, residual stresses)
- Comprehensive crystallographic grain boundary characterization
- Physical properties of microstructures
- Input data for simulation processes

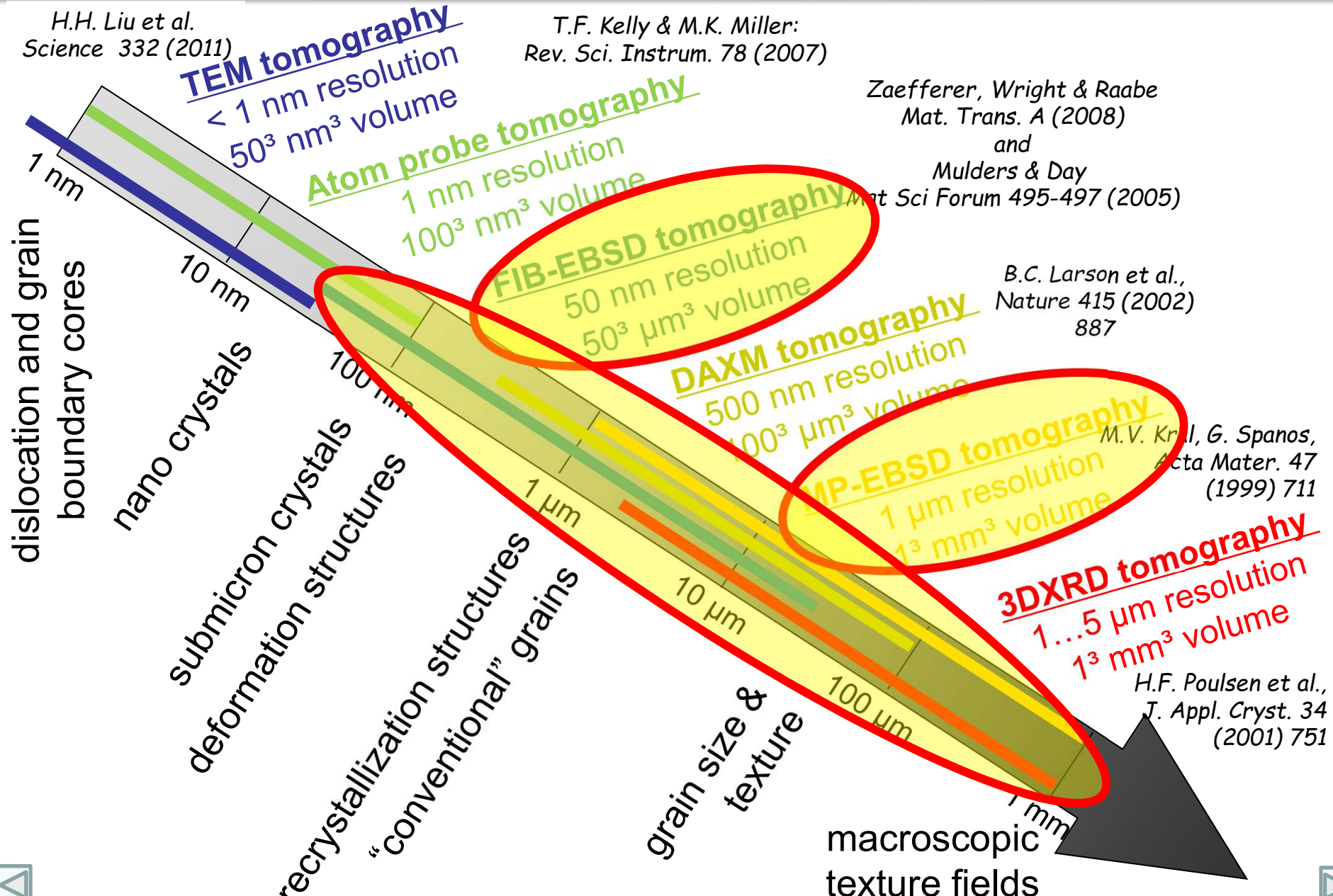
Kun Mo, et al., J. Nuclear Materials 441 (2013) 695



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

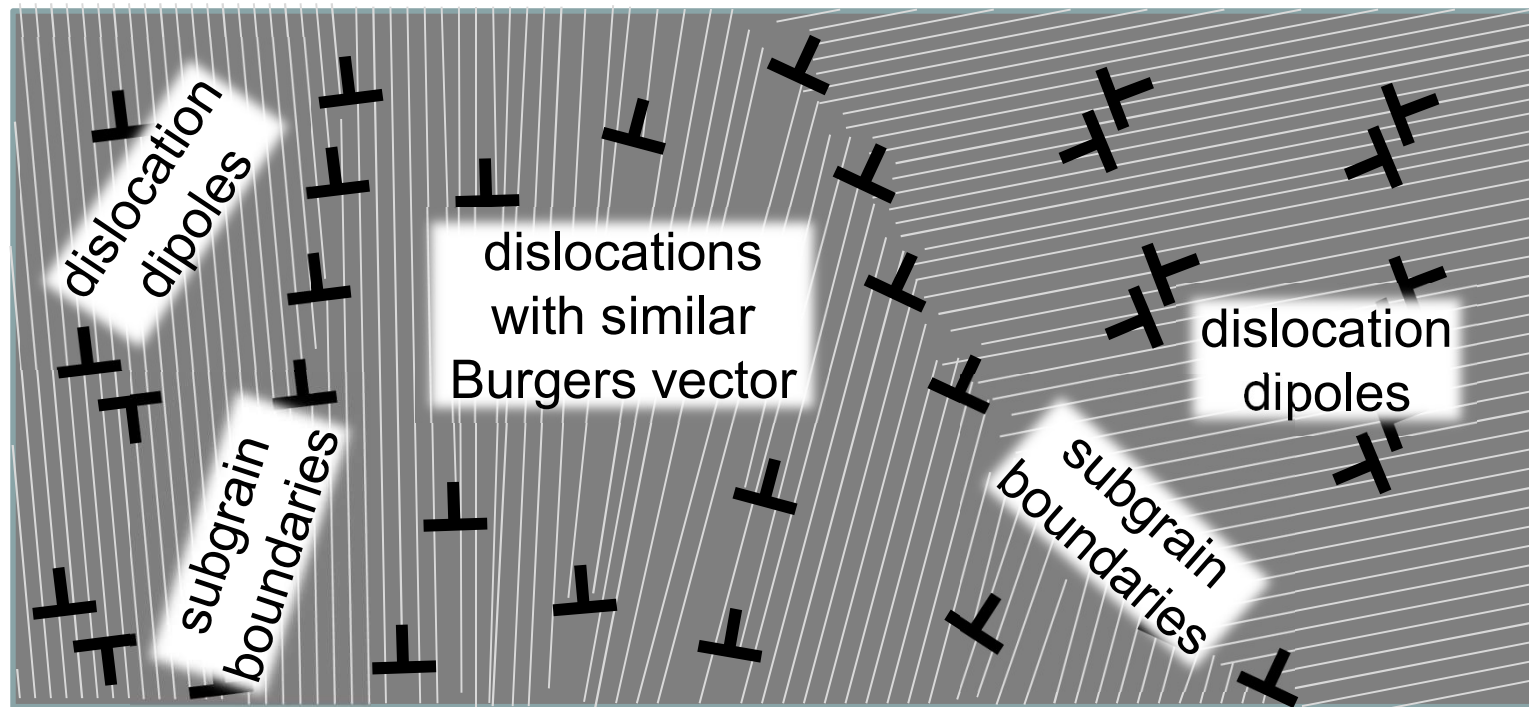


Length scale of 3D orientation microscopy



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Geometrically necessary dislocations: GND

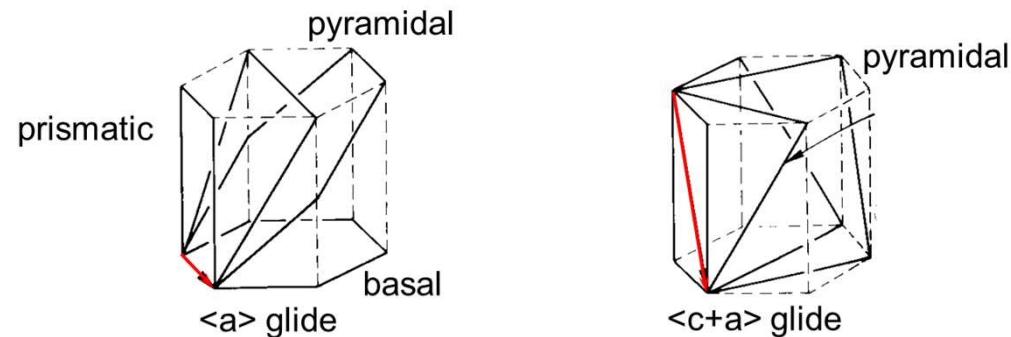
⇒ Create measurable lattice rotations

Statistically stored dislocations: SSD

⇒ Only lead to pattern blurring

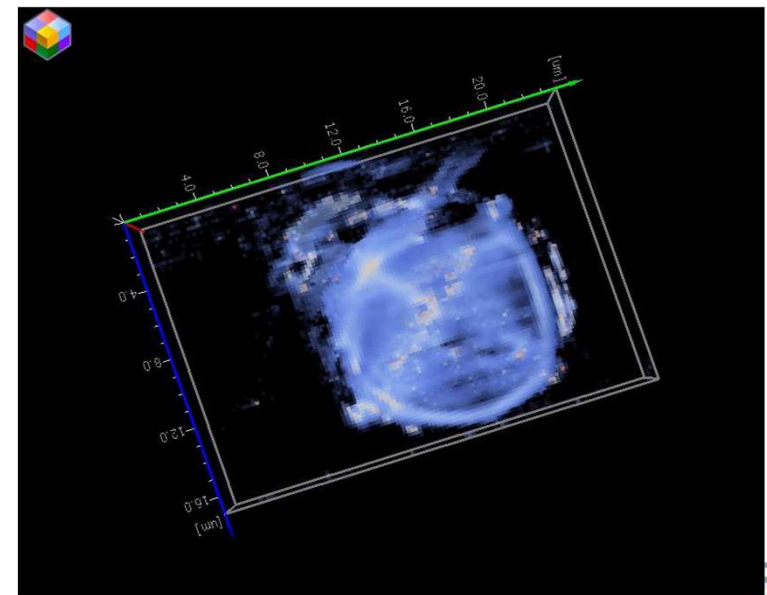
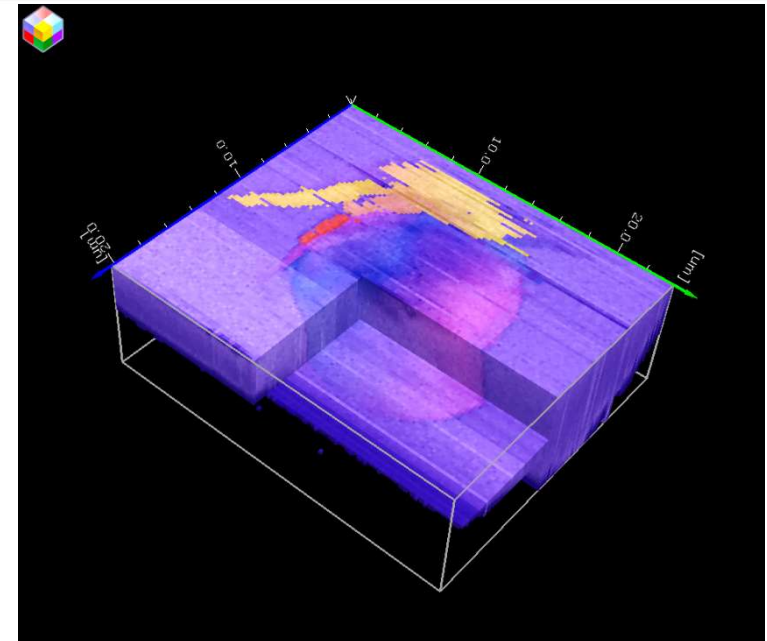
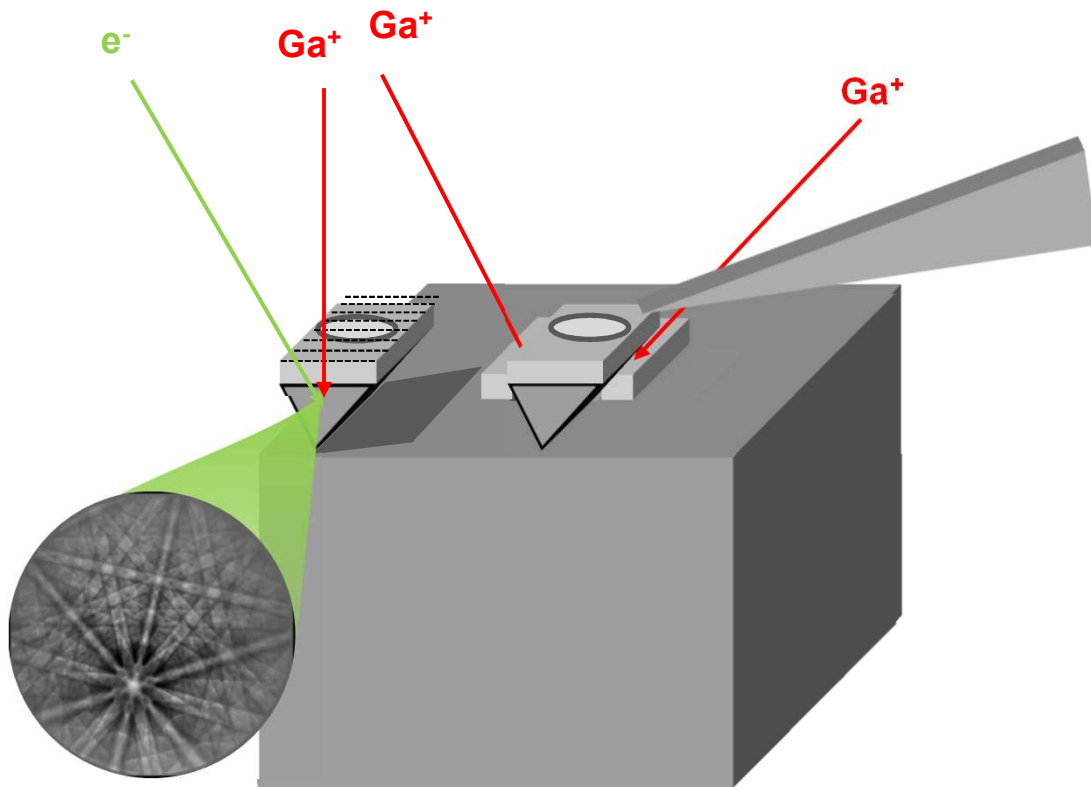


- GNDs quantify heterogeneity of plastic deformation
- 3D orientation data allow calculation of GND densities, separated for different types of dislocations



- 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

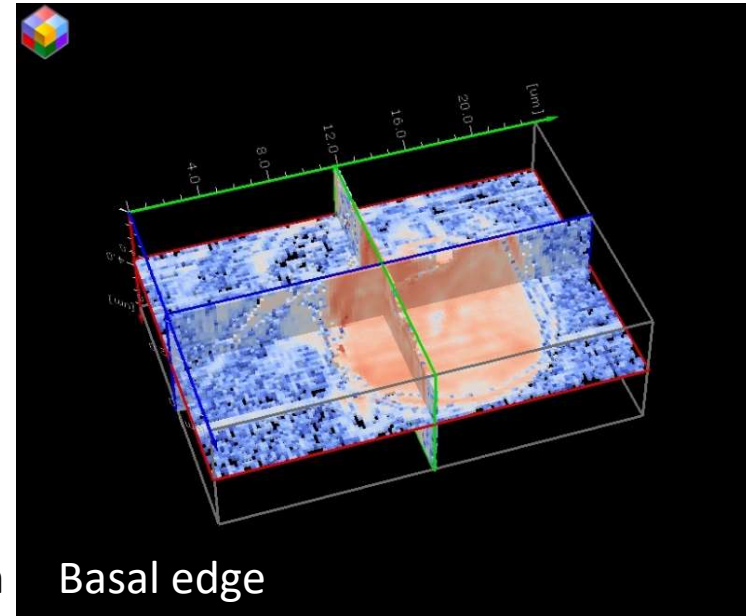
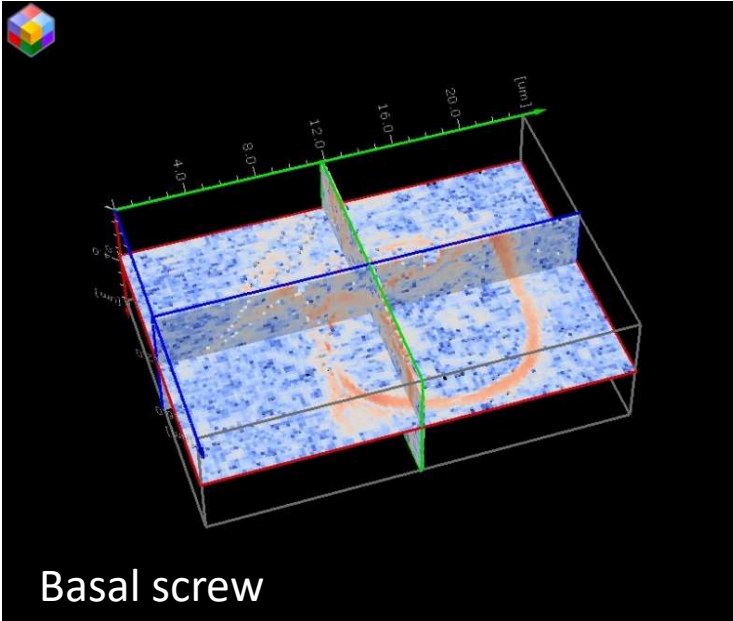
3D EBSD on nano indents in magnesium



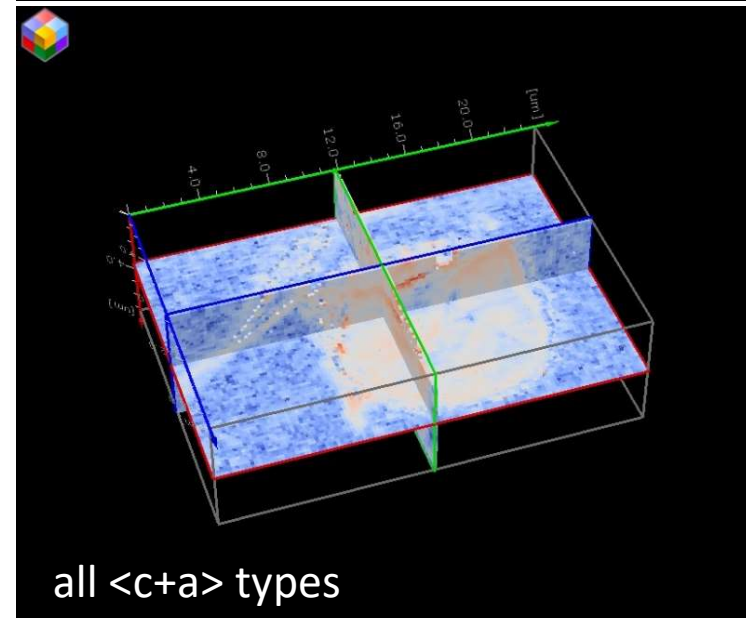
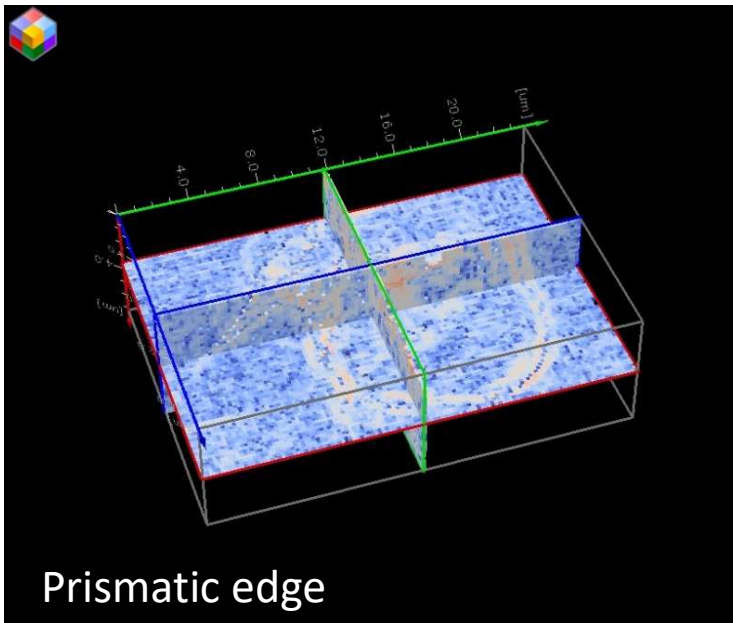
G. Nayyeri, et al. Mater Sci. Eng. A, 670 (2016), pp. 132-145



GNDs under (0001) indent



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

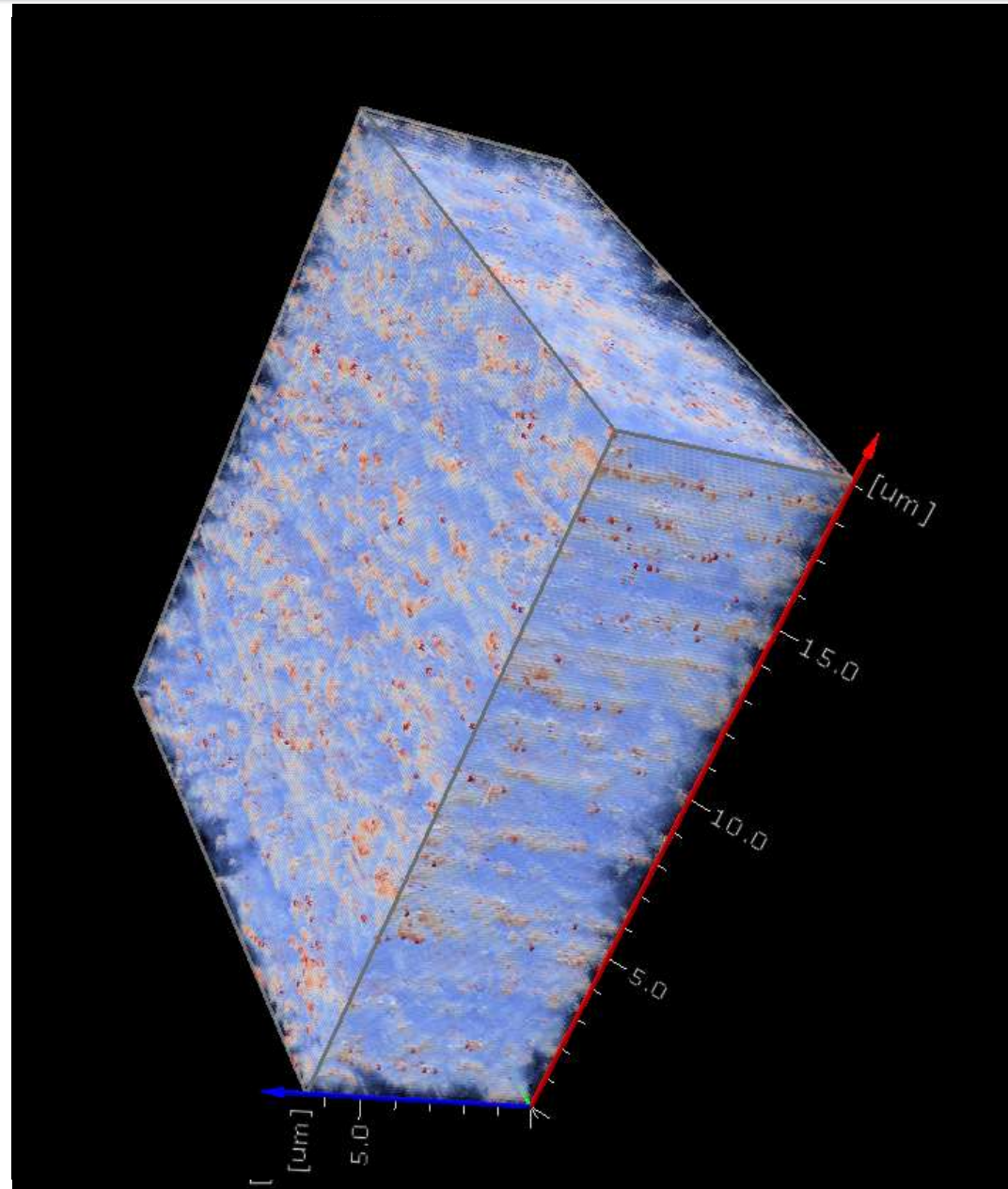




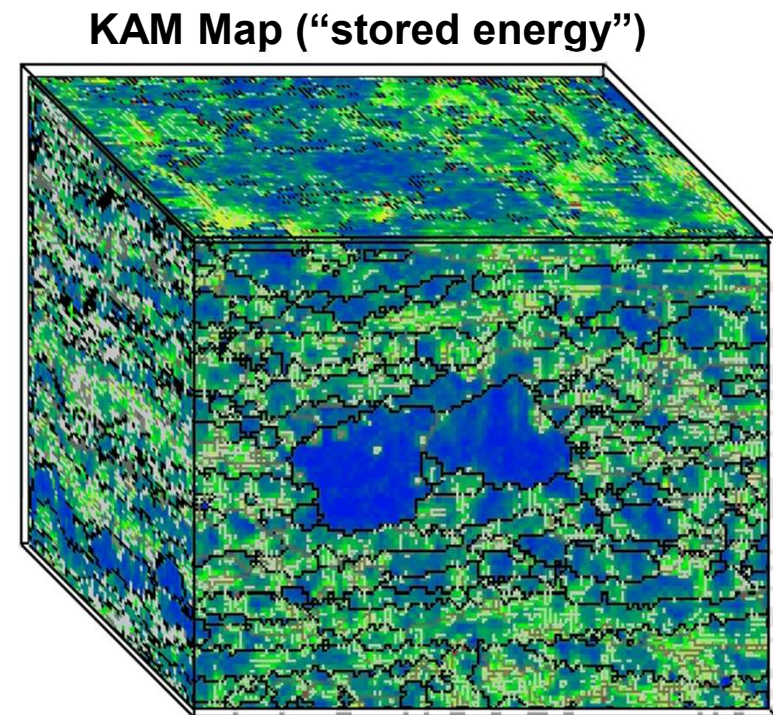
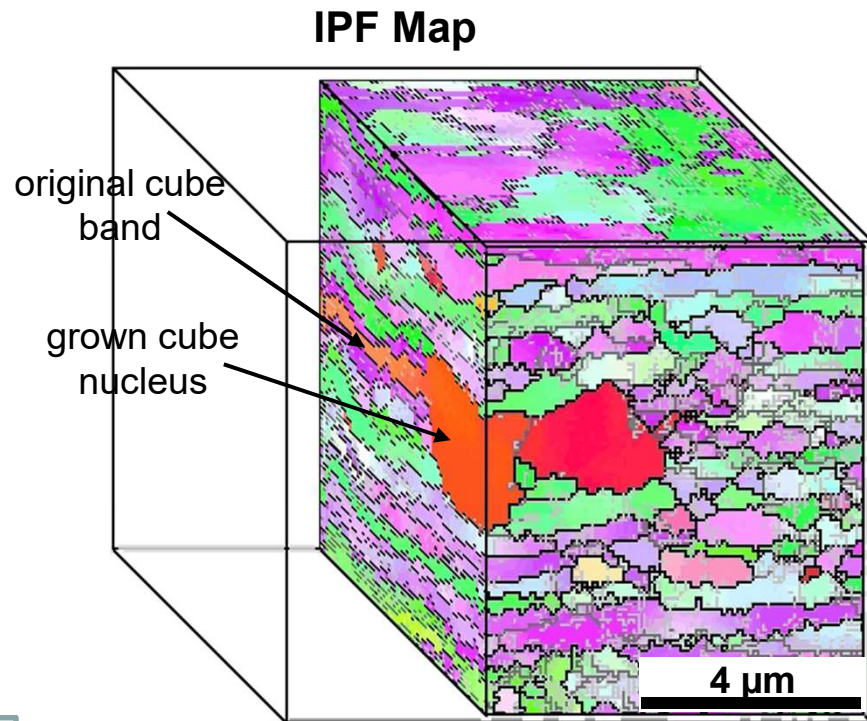
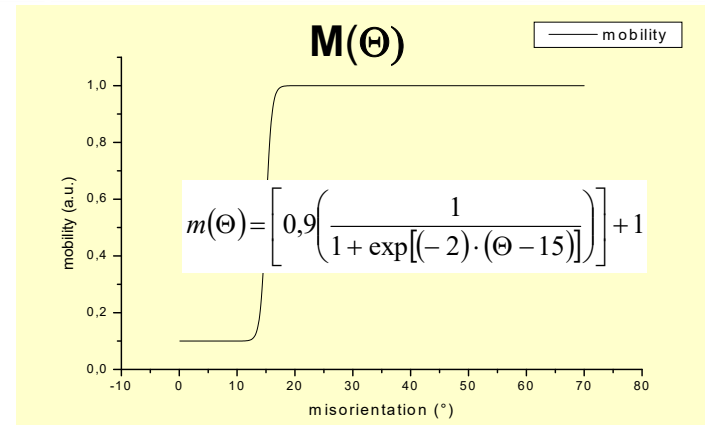
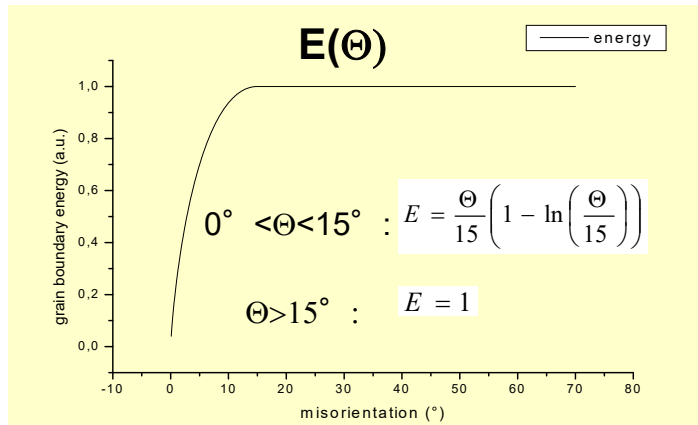
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Observation of nucleation and stored energy



3D Monte-Carlo Potts model simulations





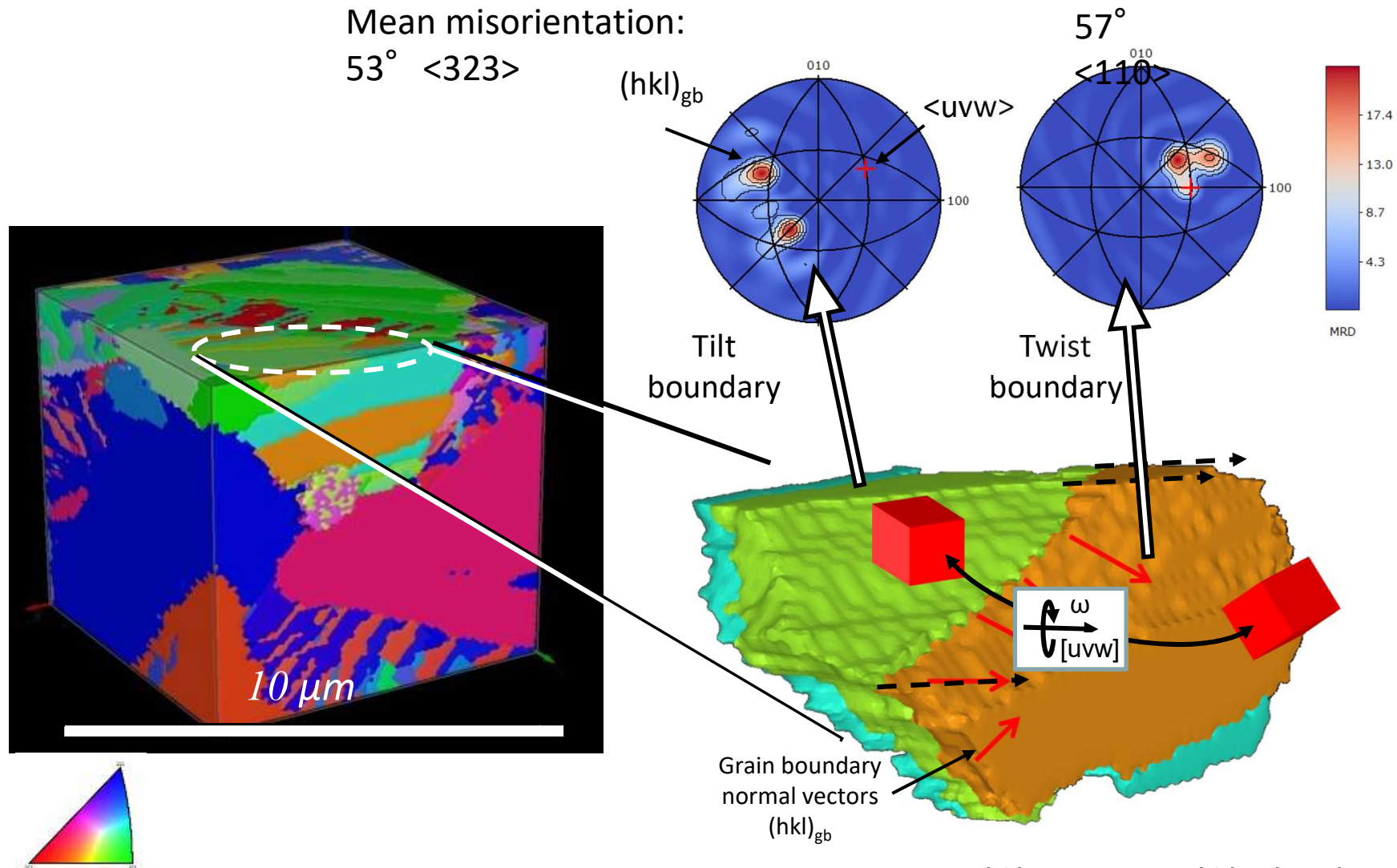
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Comprehensive description of grain boundaries



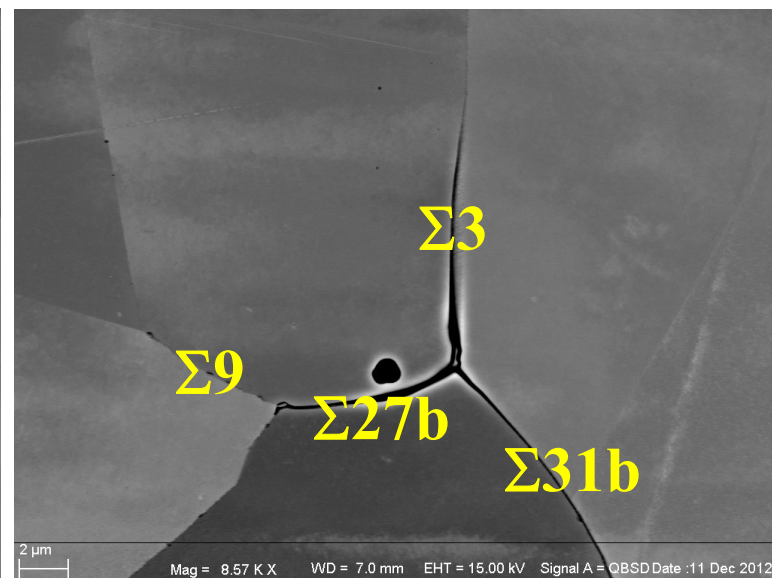
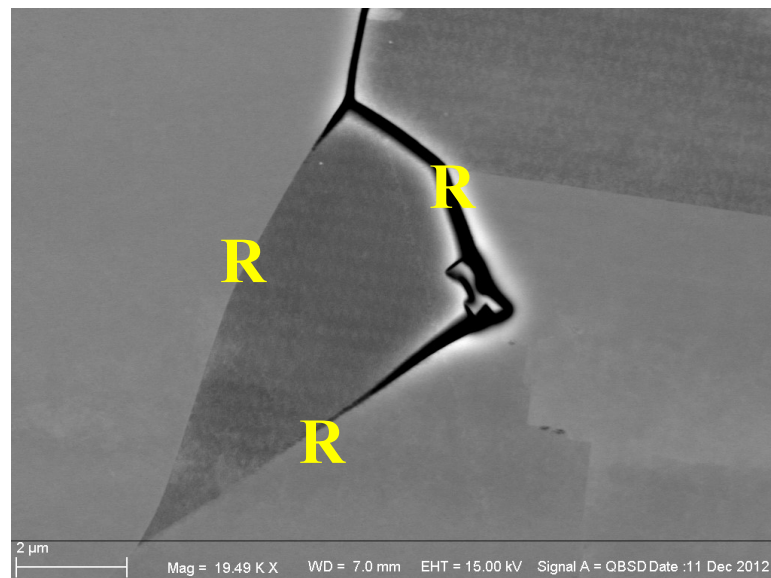
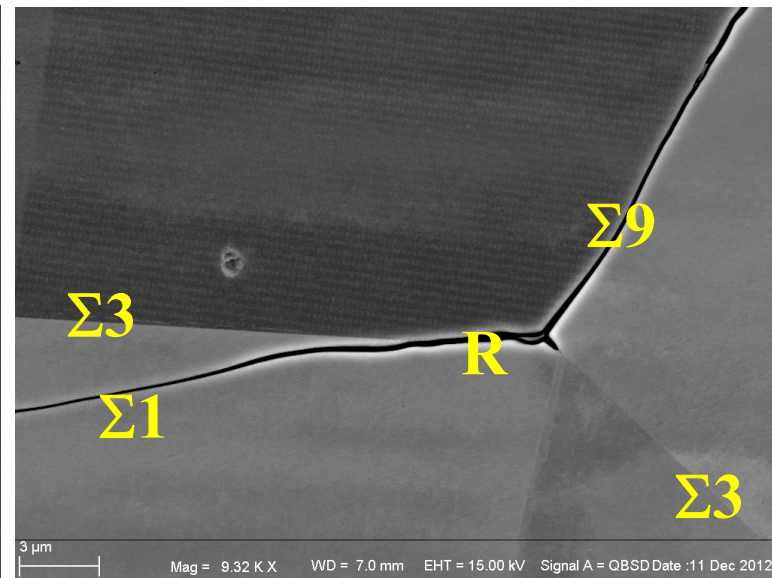
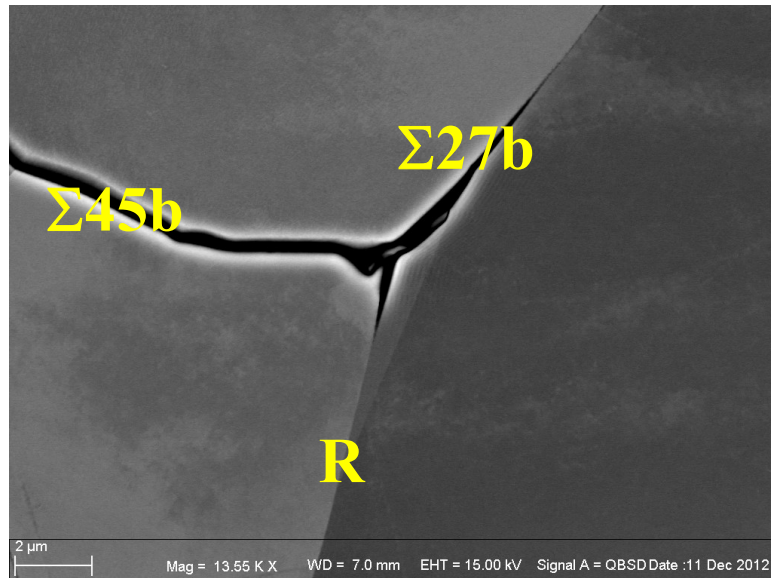
Mean misorientation:
 $53^\circ \langle 323 \rangle$



5 rotational parameters: ω (1), $\langle uvw \rangle$ (2), $(hkl)_{gb}$ (2)

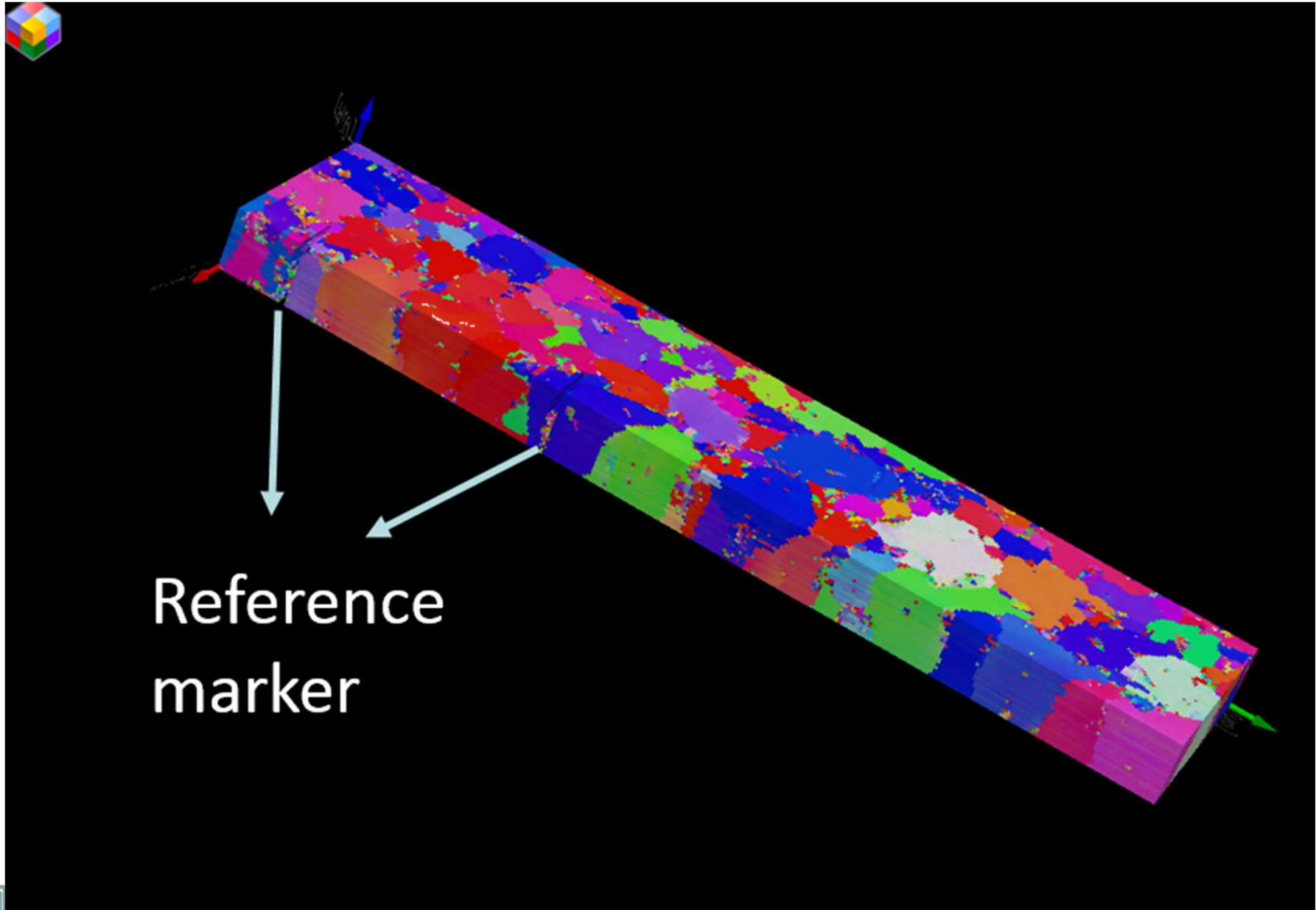
Measurement of grain boundary character requires 3D observation

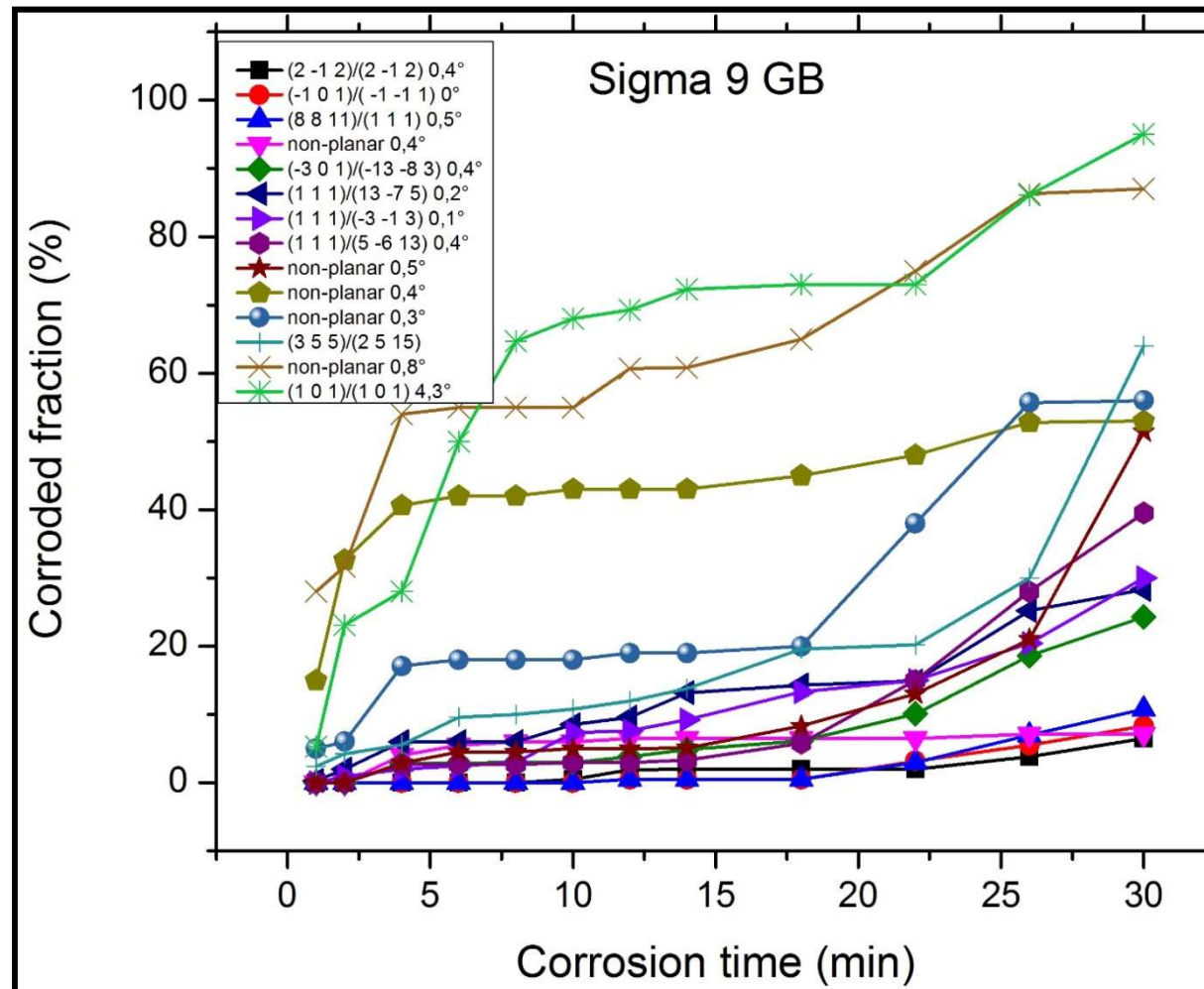
Chemical properties: corrosion of boundaries



◀ Corrosion behaviour is not only depending on misorientation ▶

Large-size 3D EBSD map (mechanical sectioning)





- $\Sigma 9$ with coherent or low Miller index GB plane have higher corrosion-resistance than those with high Miller index GB plane or non-planar GBs.
- $\Sigma 9$ with large deviation angle are easily corroded.



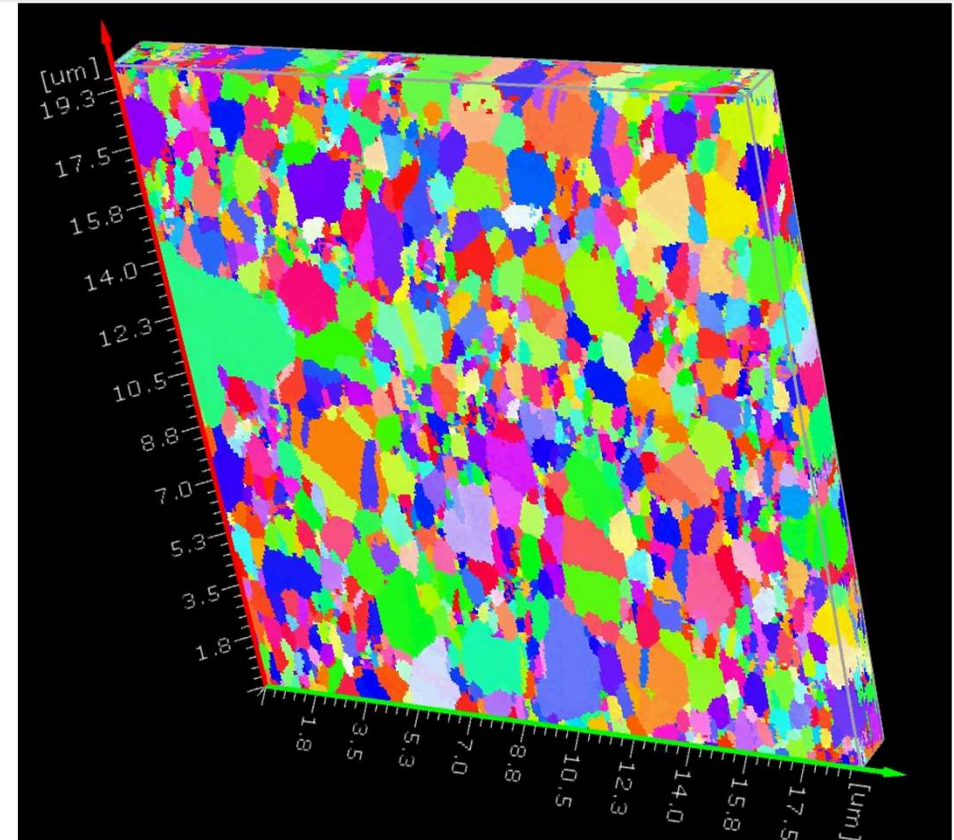


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Sources of beam drift:

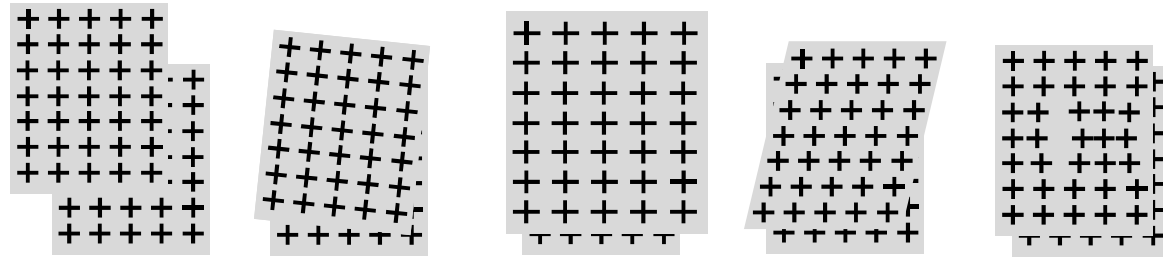
- Electro static (de)charging
- Thermal effects (e.g. lens cooling water)
- Mechanical relaxation (stage)
- Magnetic fields (lens)
- Surface roughness (e.g. curtaining)
- Incorrect sample repositioning



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)





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


Parametric

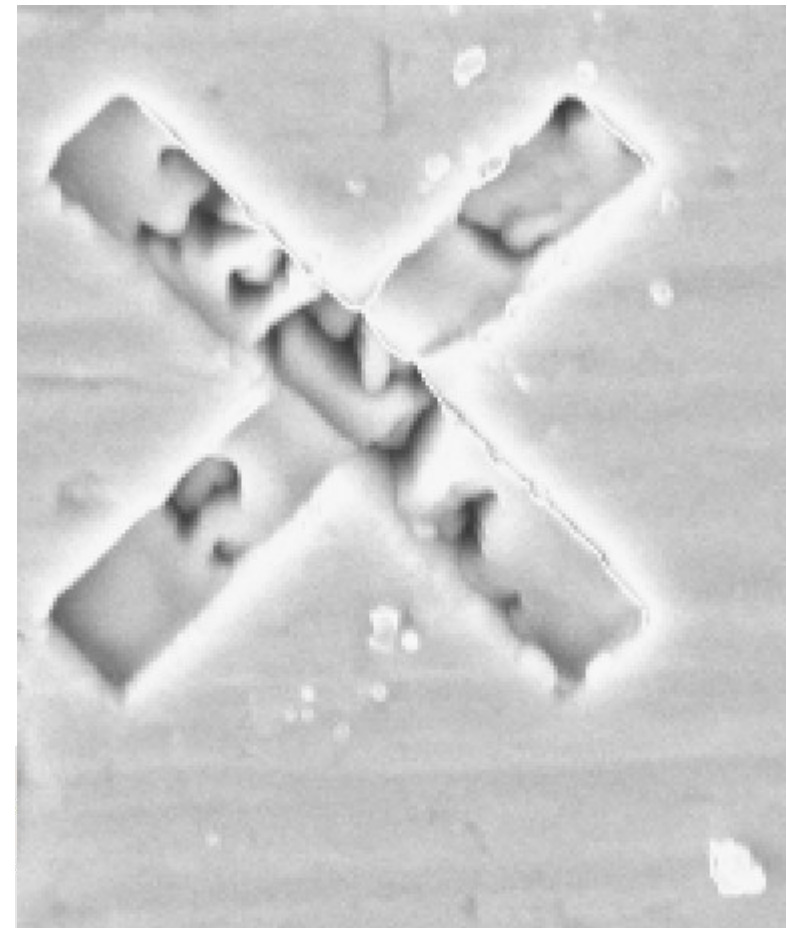
Non-rigid alignment of fundamental importance for the quality of all subsequent analysis:

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



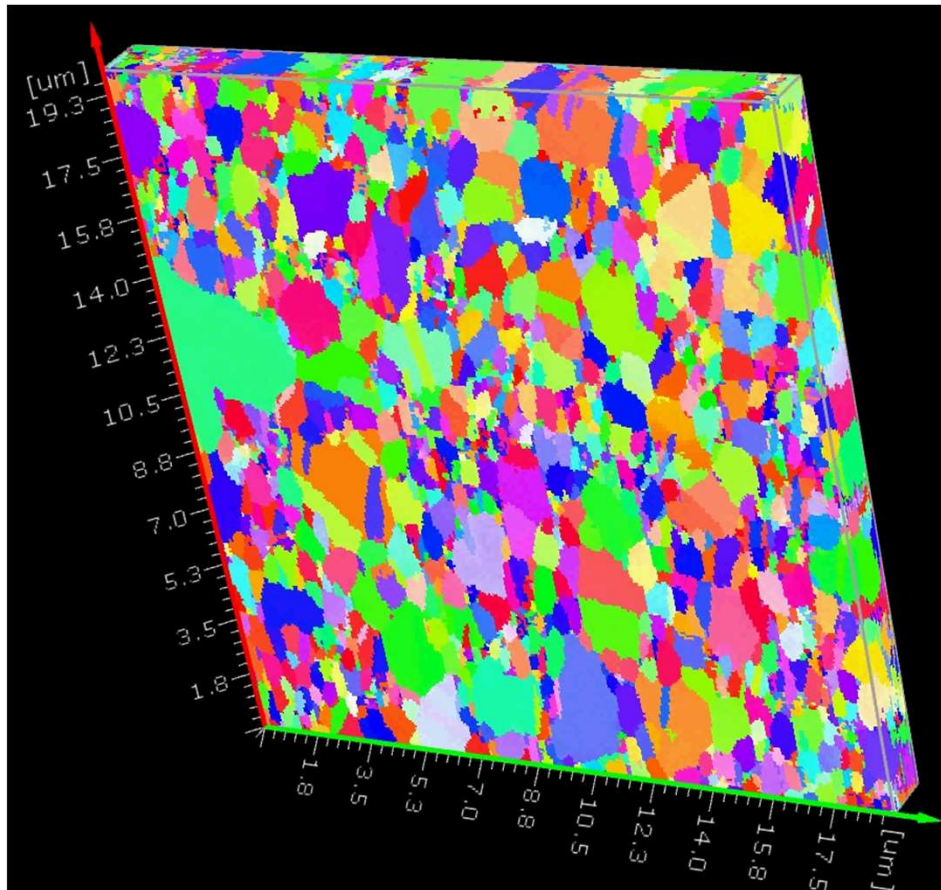


Generic row-wise drift (X and Y) 

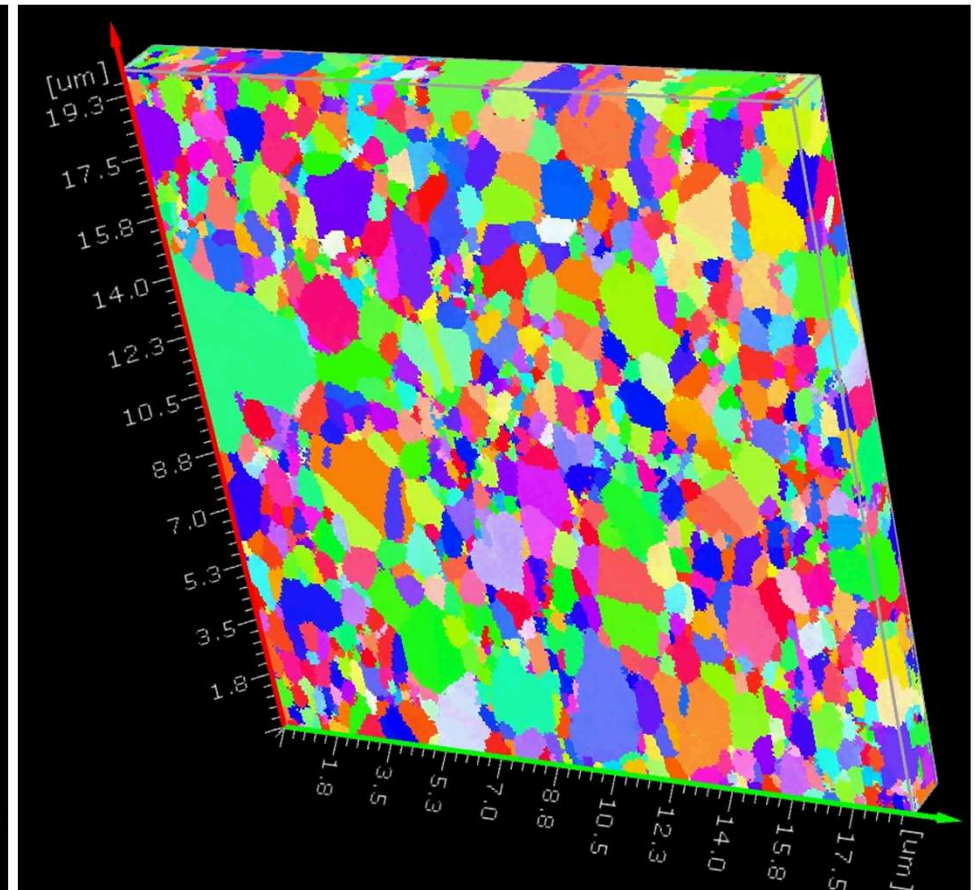


Original

Example: non-rigid alignment



before alignment



after alignment





- 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 microstructures observable

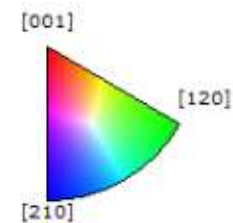
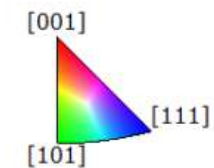
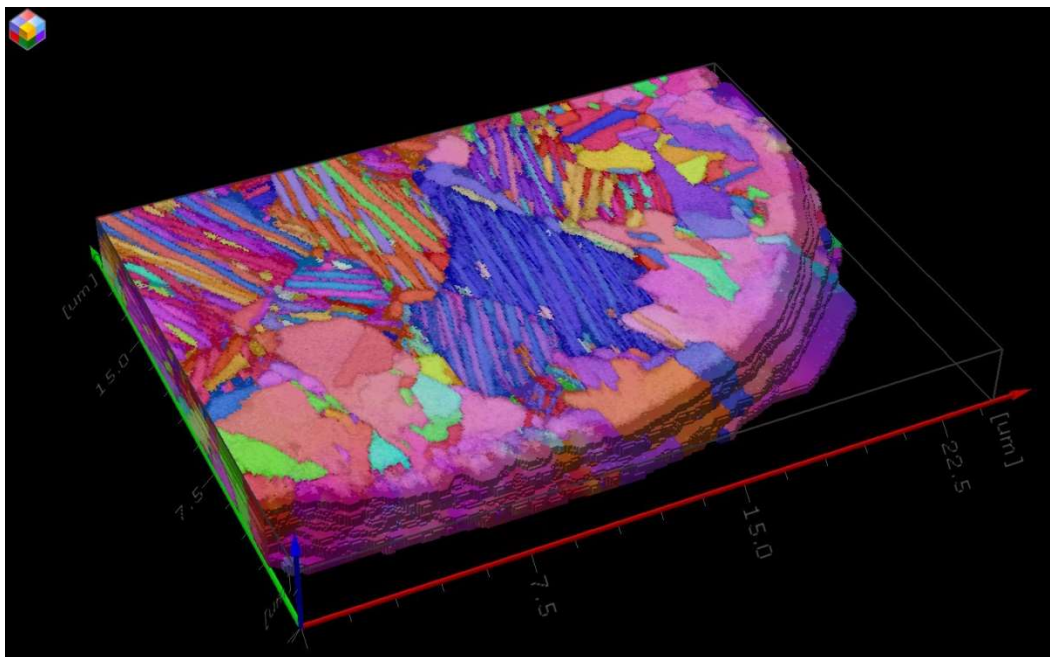


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Data postprocessing – Subsetting & visualization



- 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

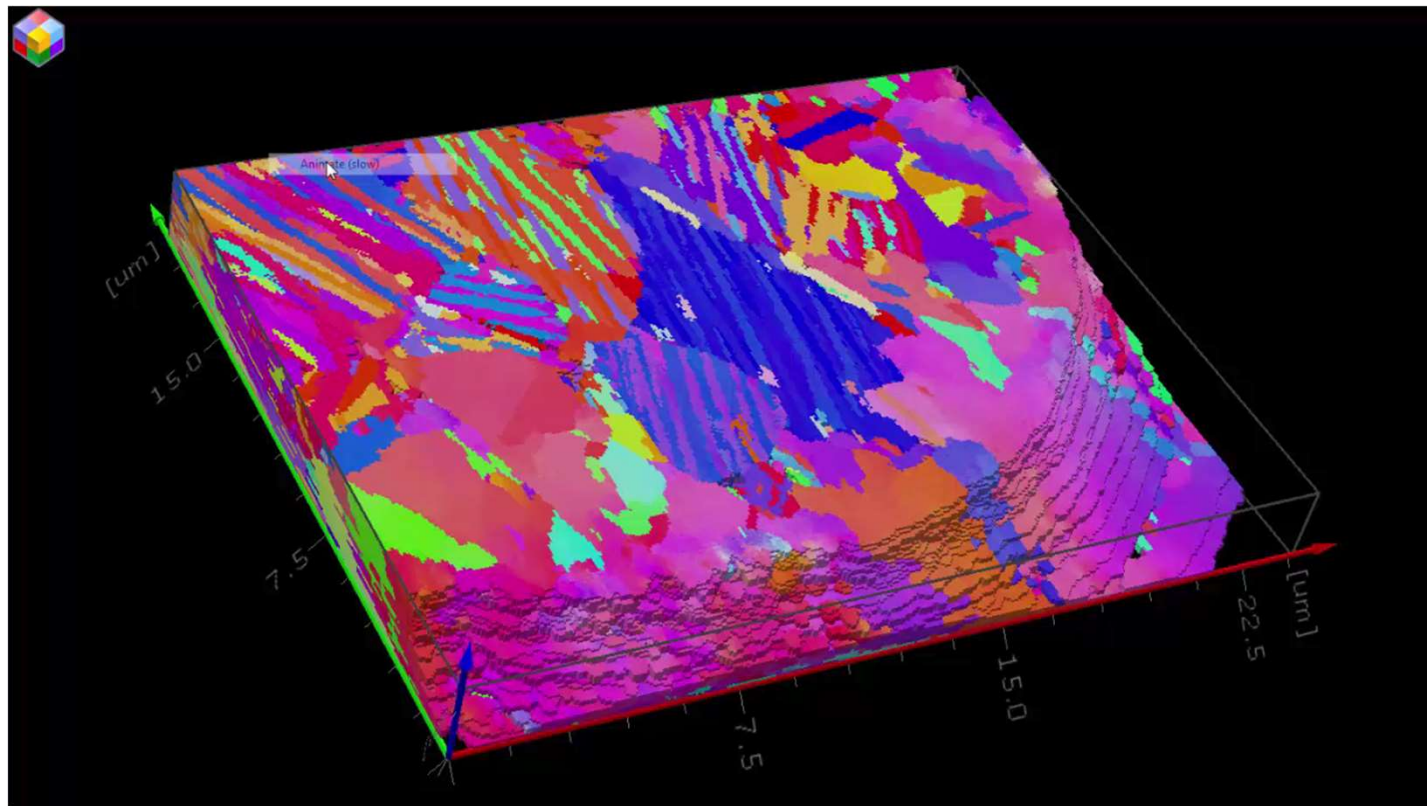


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Data postprocessing – slice realignment



3D EBSD – 27 slices – no realignment applied



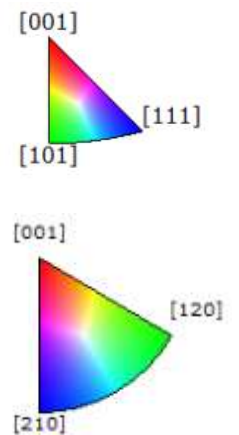
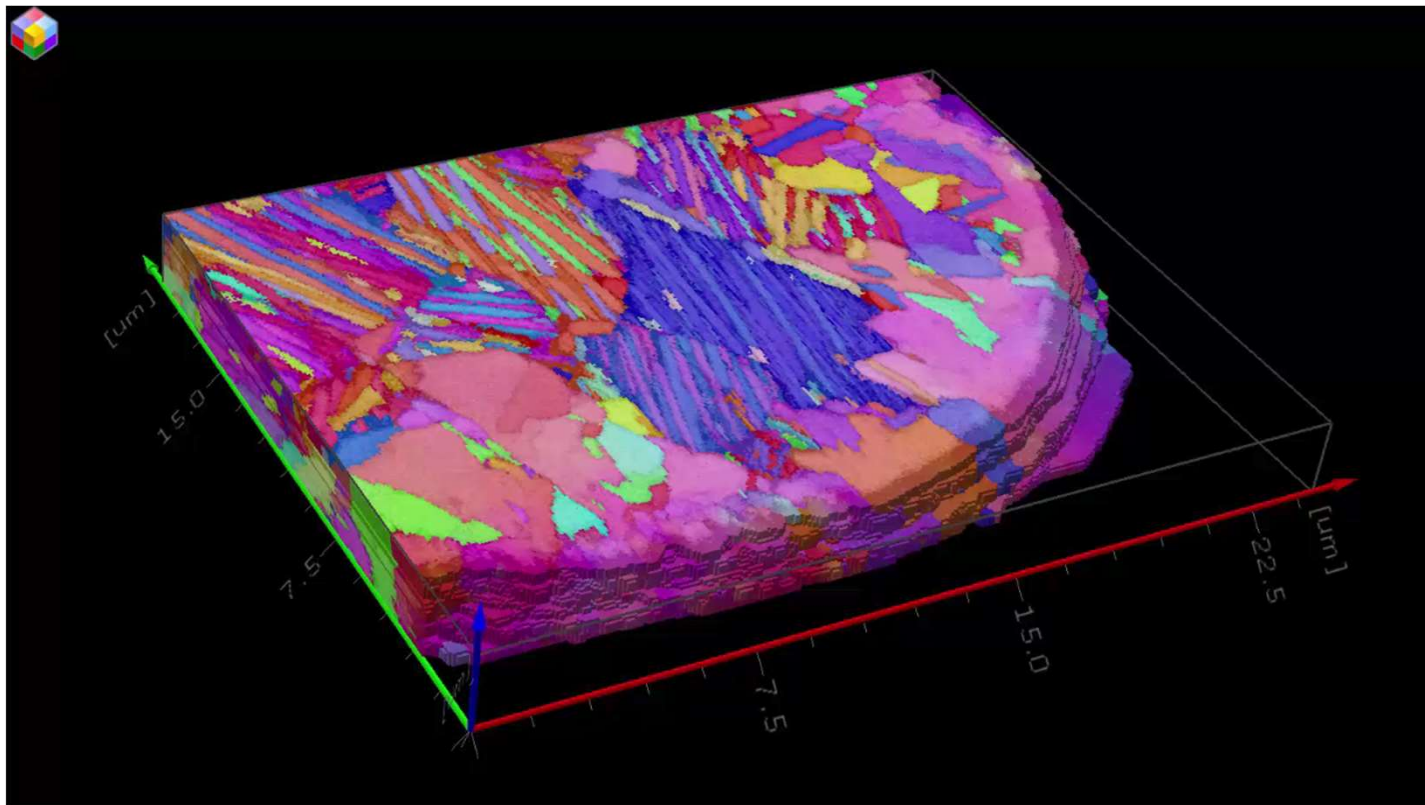
- Strong initial misalignment due to particle instability

ESPRIT QUBE

Data postprocessing – slice realignment



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



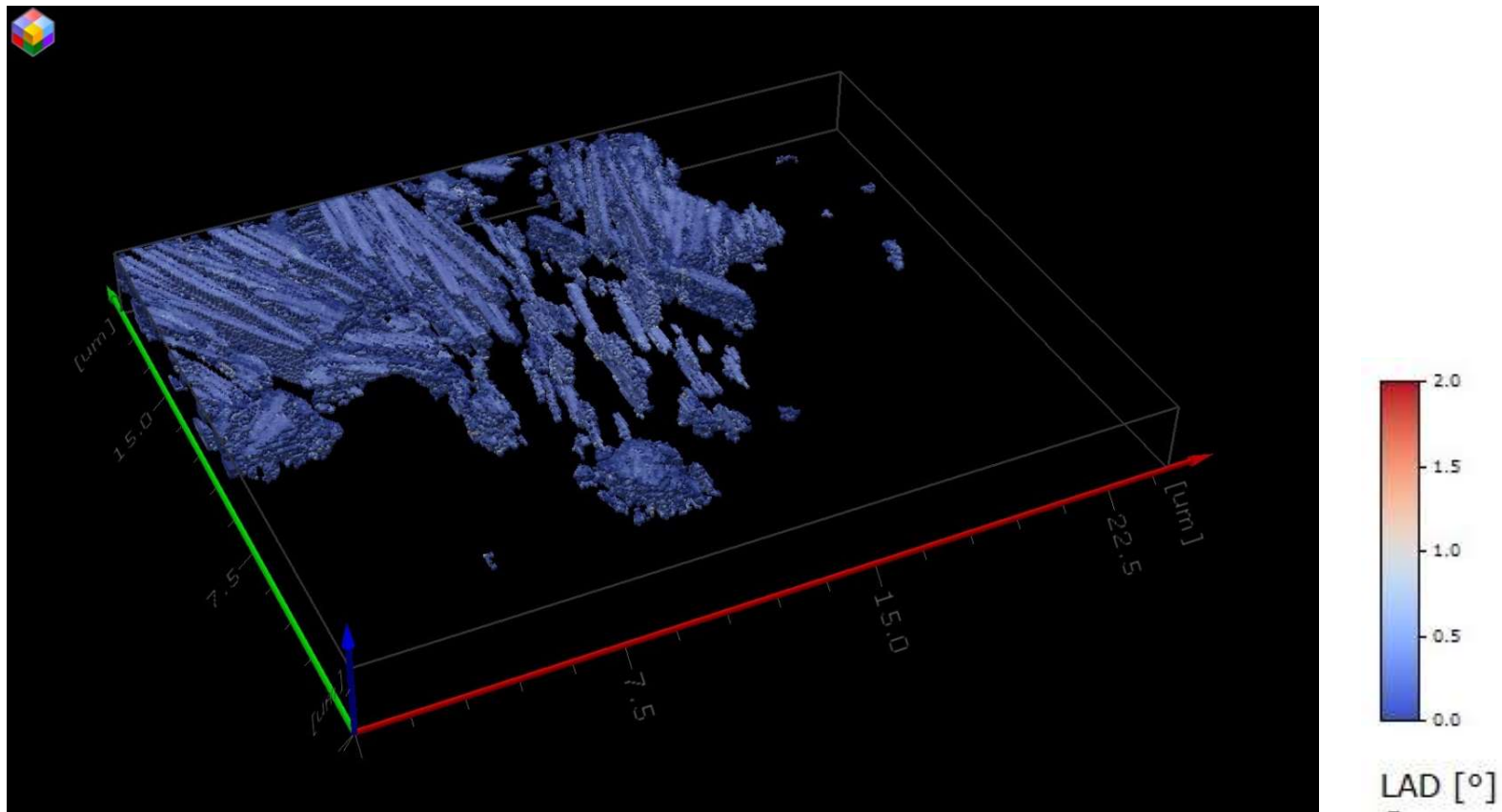
- Data cube after realignment and cropping (on two sides only)

ESPRIT QUBE

Data postprocessing – LAD



Local Average Disorientation (LAD) inside the intermetallic phase



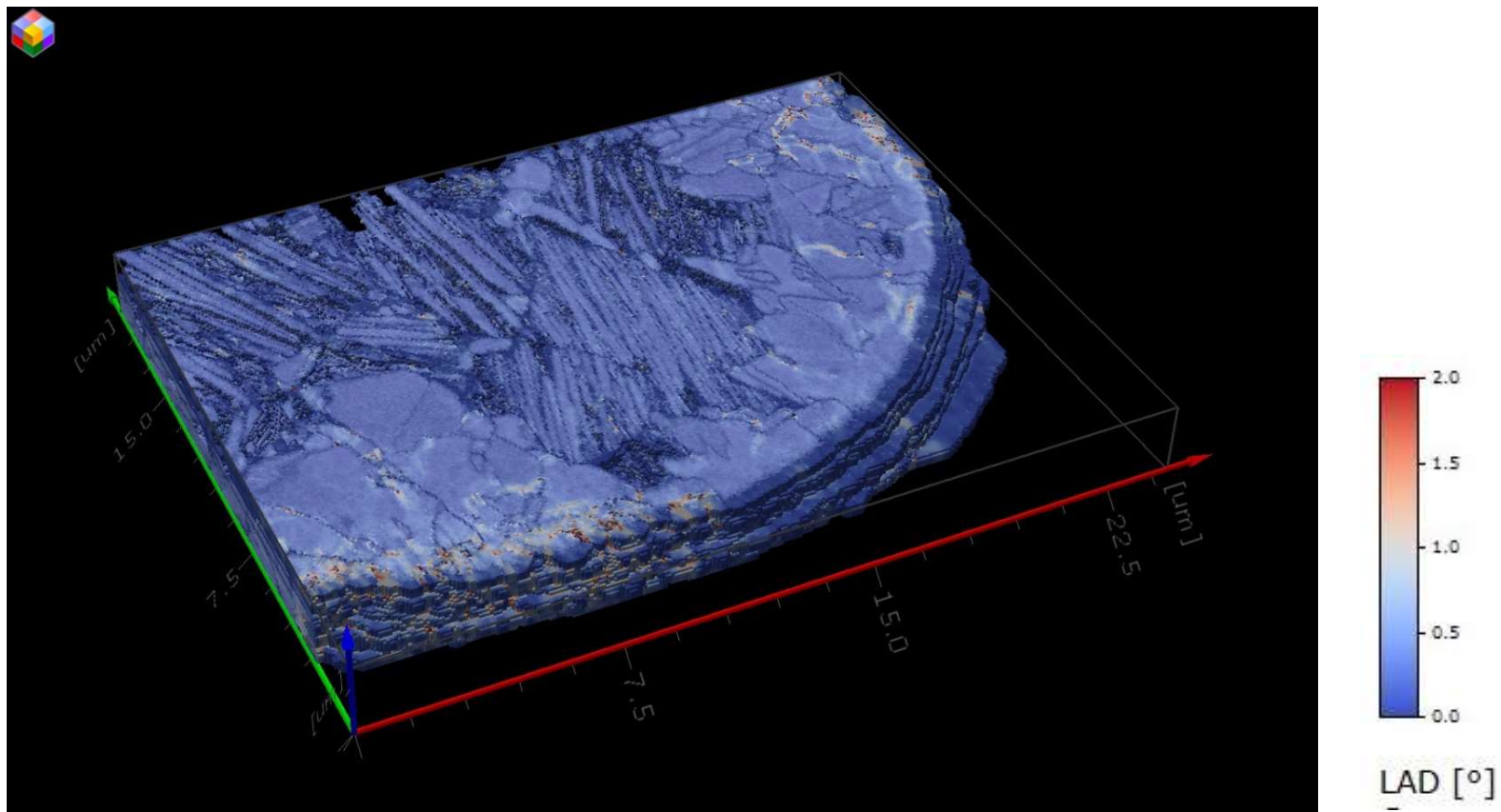
- Very low LAD values inside the hard intermetallic phase

ESPRIT QUBE

Data postprocessing – LAD



Local Average Disorientation (LAD) inside the CuZn alloy



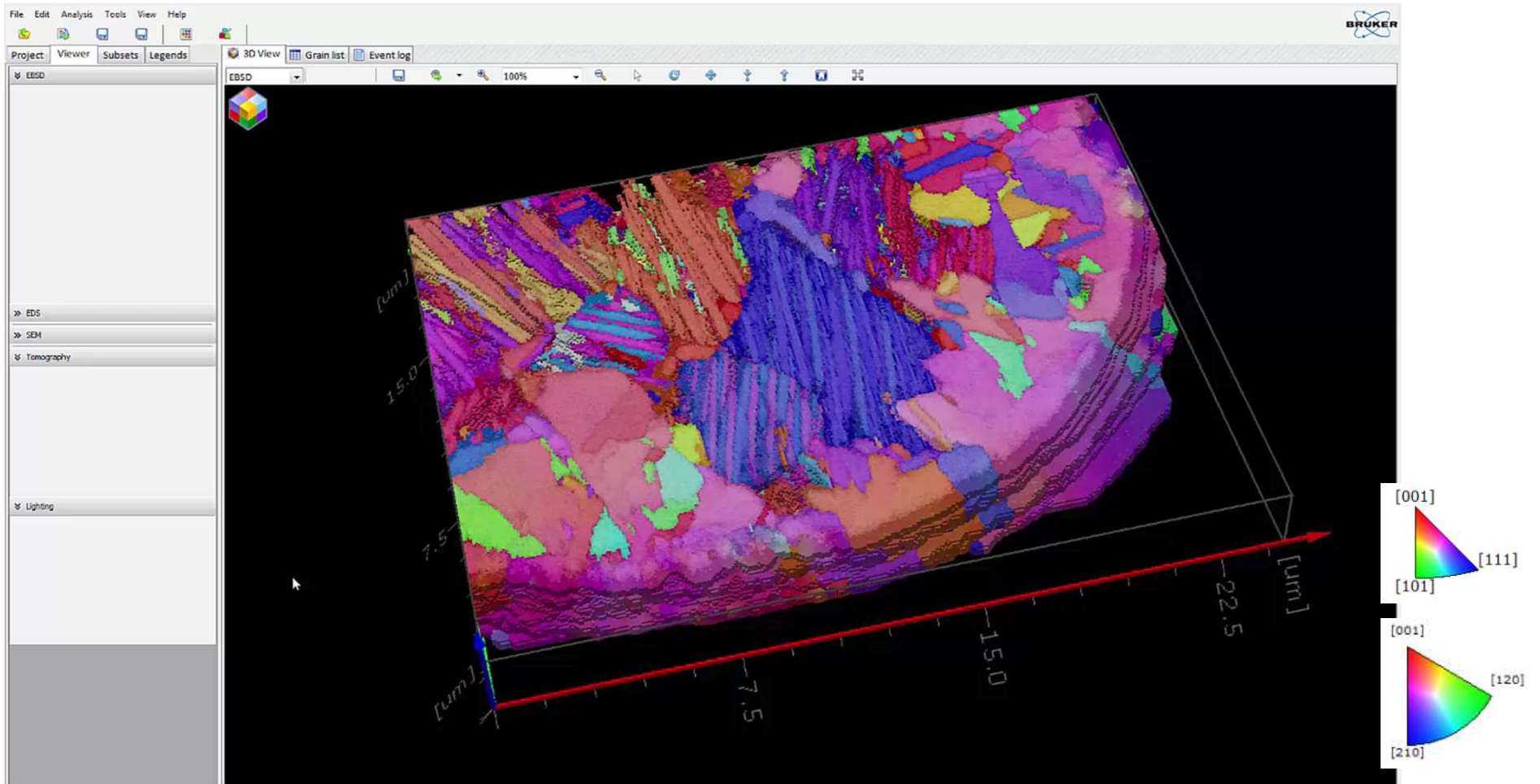
- Significantly higher low LAD values inside the soft CuZn alloy

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Data postprocessing – Subsetting & visualization



Advanced subsetting and interactive 3D data visualization

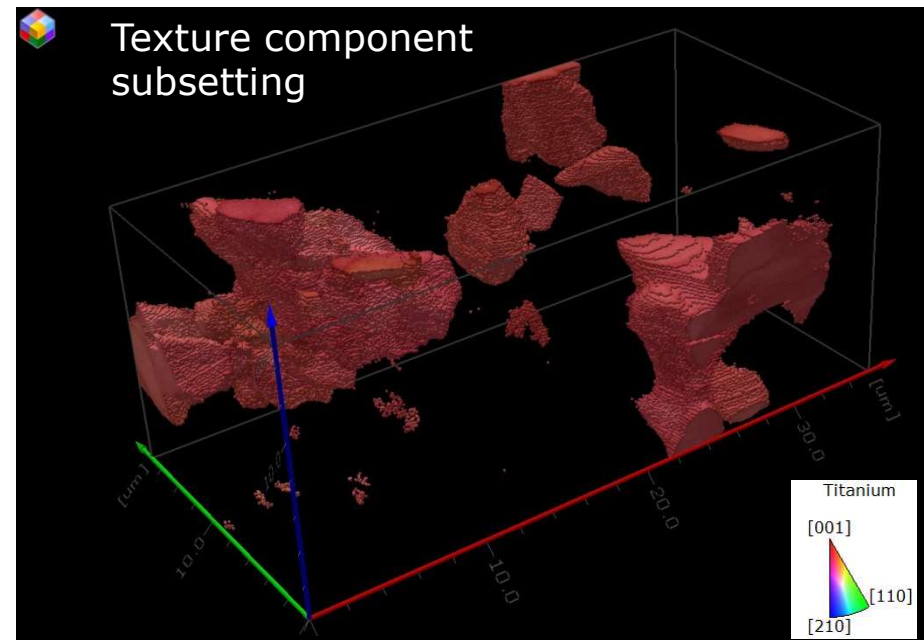
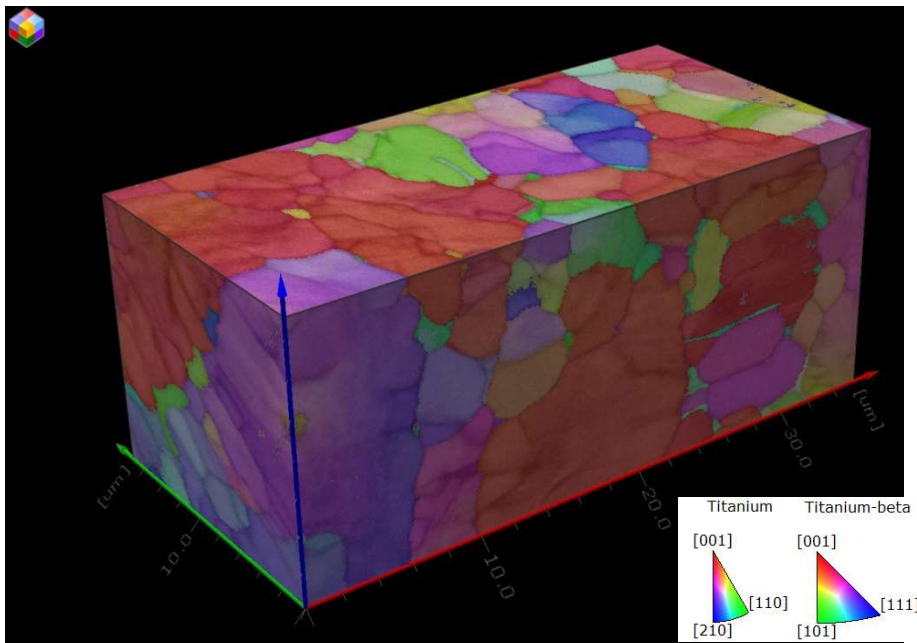


ESPRIT QUBE

Data postprocessing – Subsetting & visualization



- Deformed Ti alloy (alpha and beta phases)
- Large data cube – 245 slices ($35 \times 20 \times 20 \mu\text{m}^3$)
- Serial sectioning using a PFIB-SEM with the standard “rotation” method
- Slice preparation time: $\sim 2 \text{min/slice}$
- 3D EBSD data acquisition time: $\sim 7 \text{min/slice}$

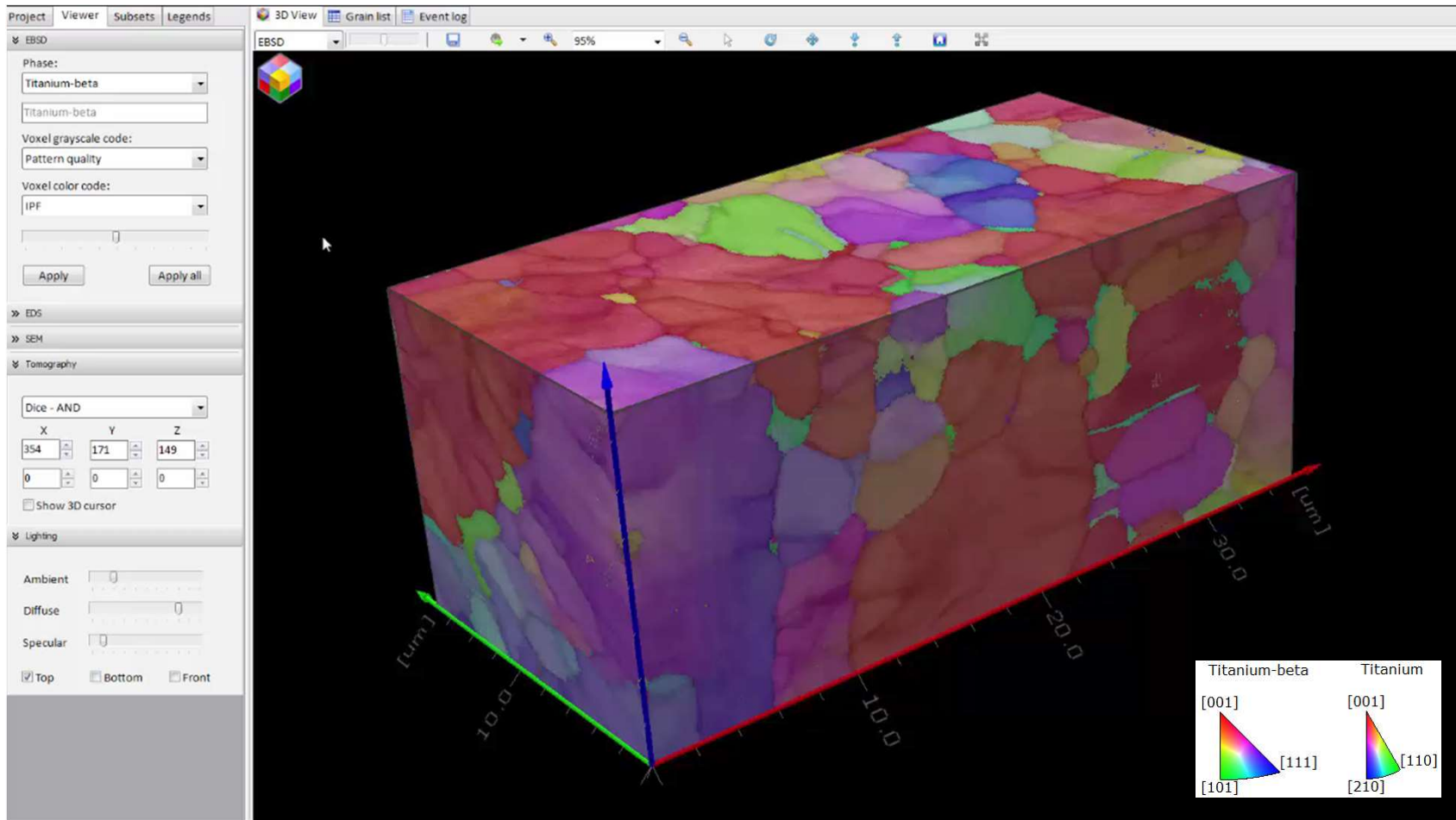


ESPRIT QUBE

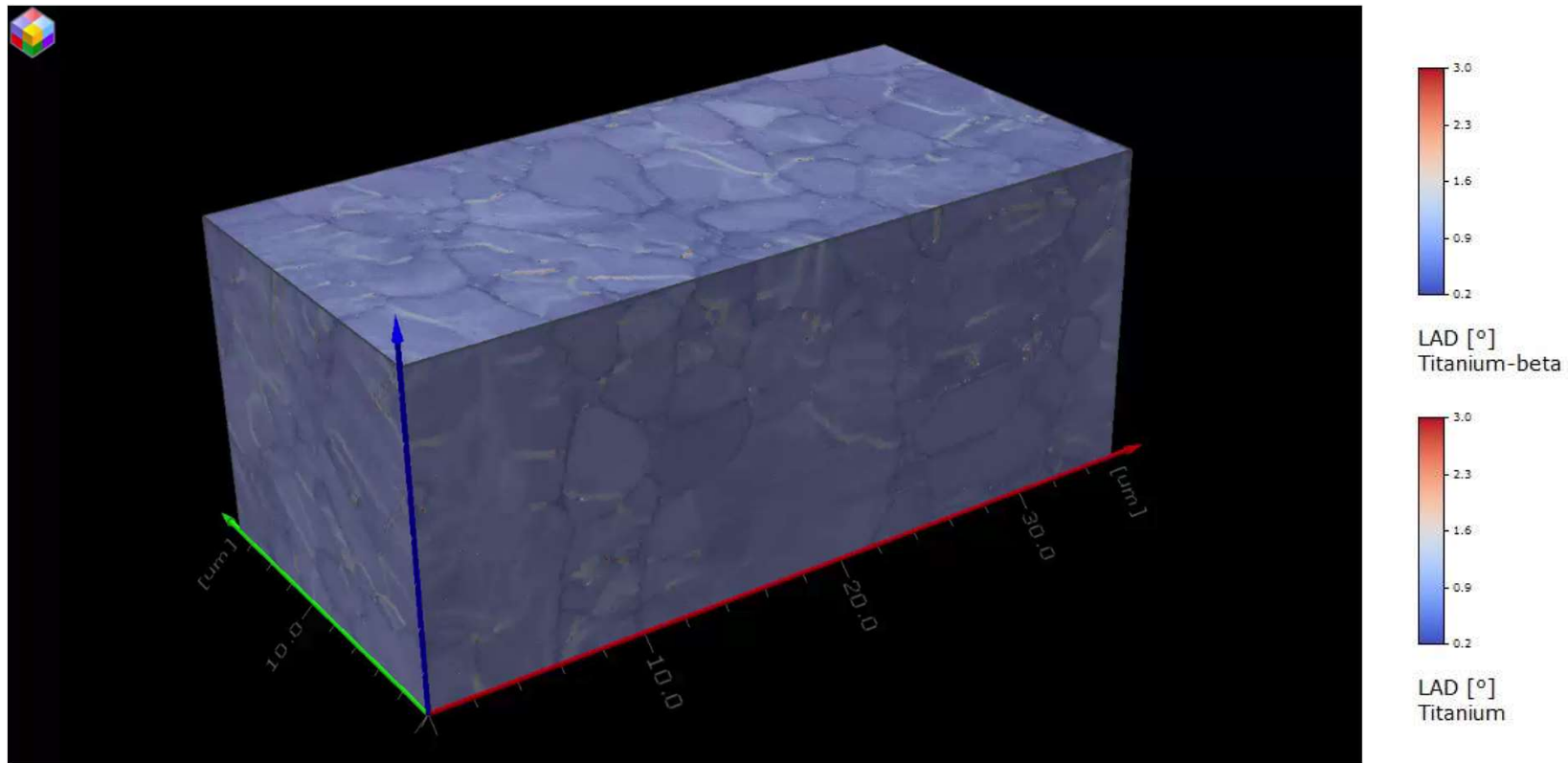
Data postprocessing – Subsetting & visualization



3D EBSD on deformed Ti alloy – phase subsetting



3D EBSD on deformed Ti alloy – LAD visualization



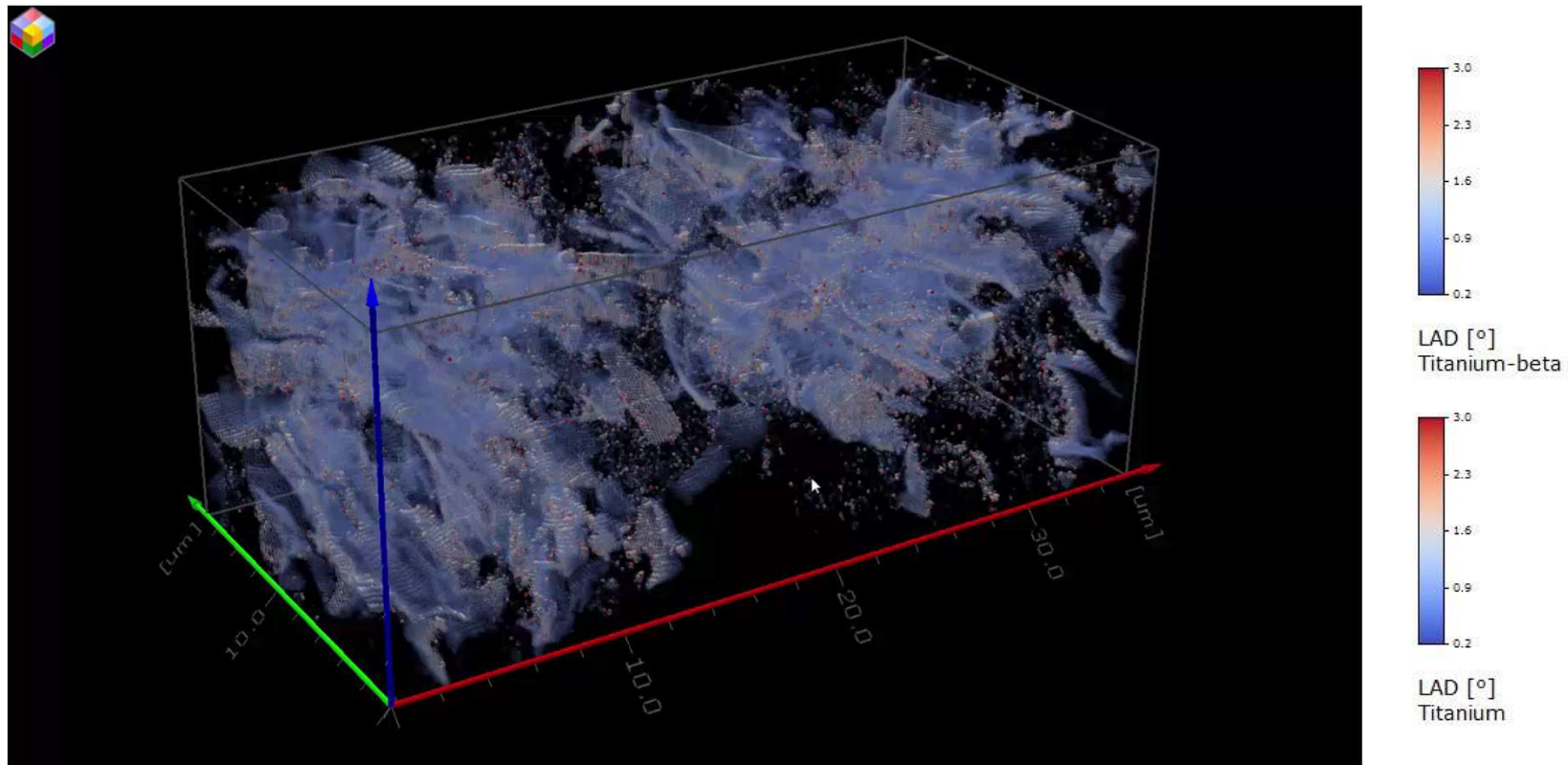
- LAD after slice realignment & cropping + data filtering
- Slice & dice visualization

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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

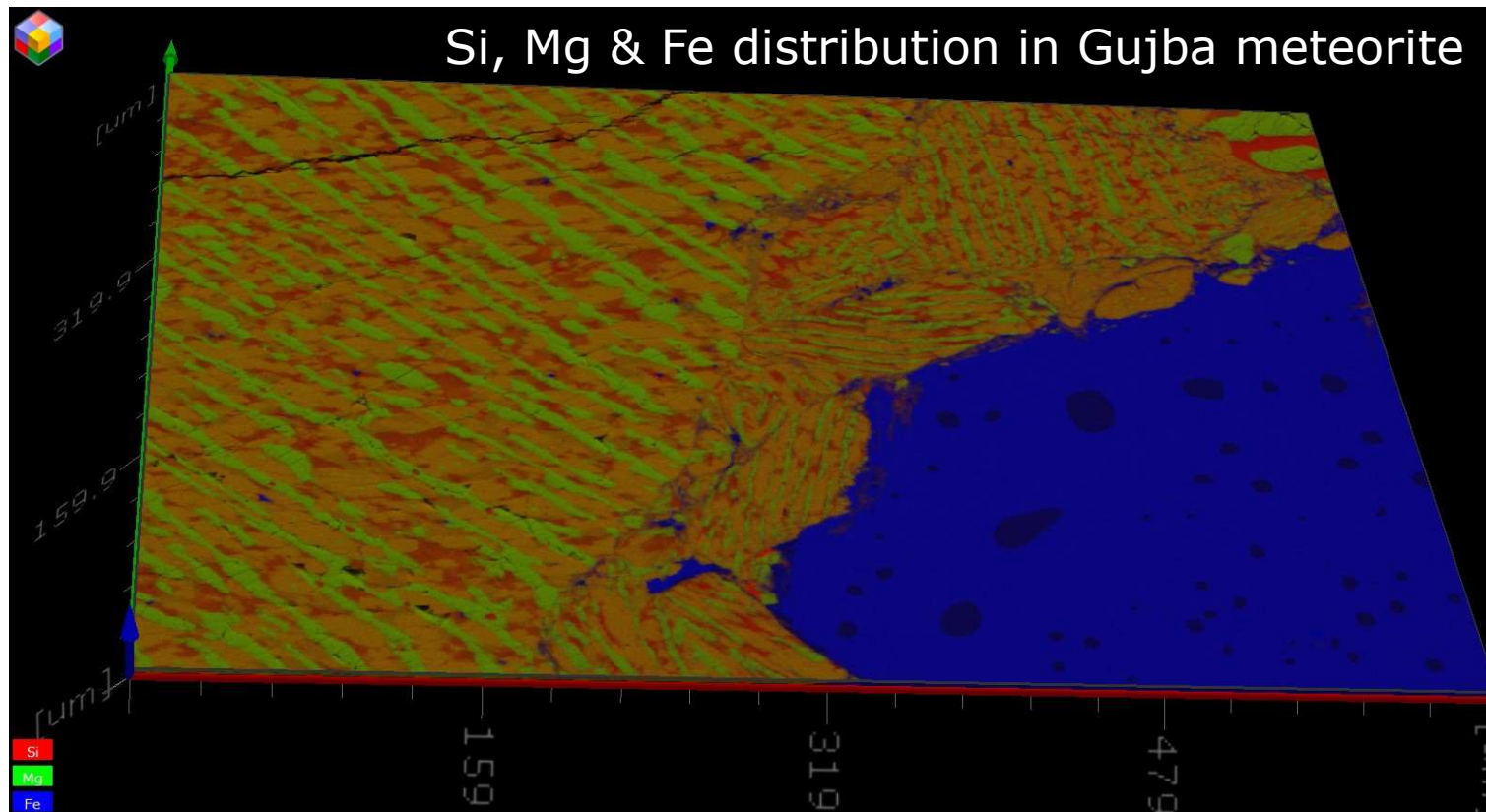
ESPRIT QUBE

3D EDS – visualization



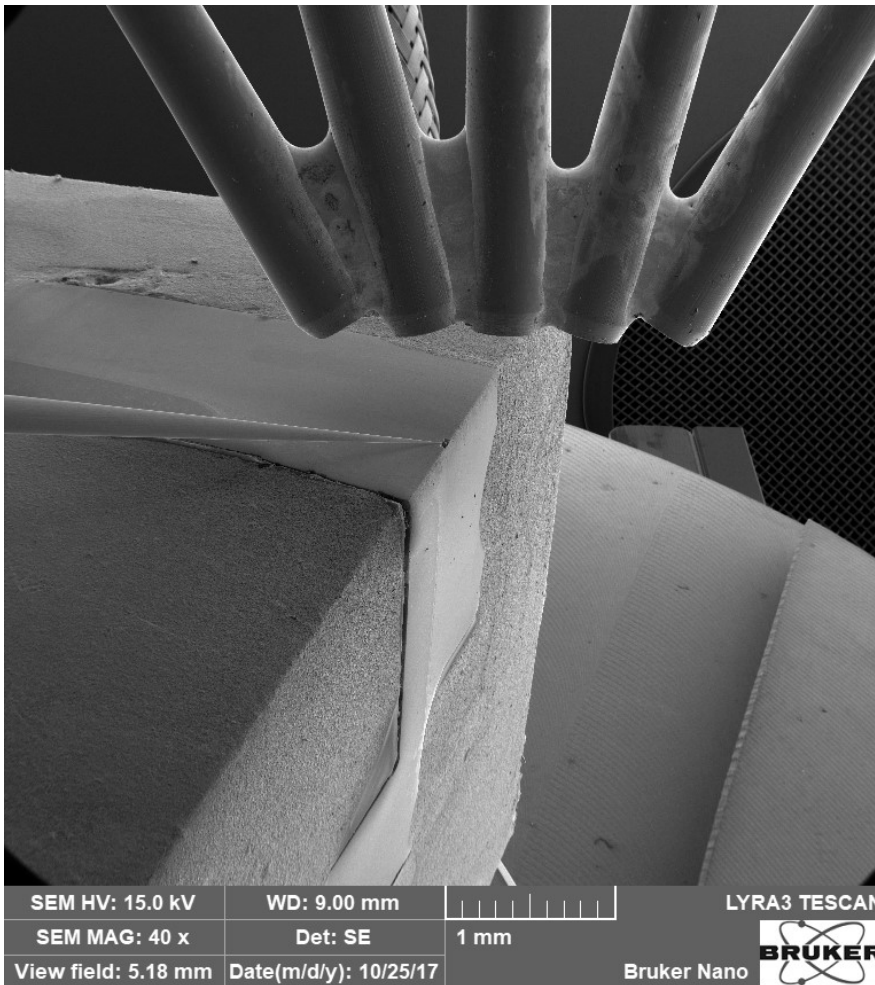
Extensive data subsetting with visual, interactive data exploration:

- EDS based subsetting (counts & quantified data)



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3D EBSD data processing: application example on stainless steel



stainless steel

Phases:
Ferrite, Austenite, Sigma

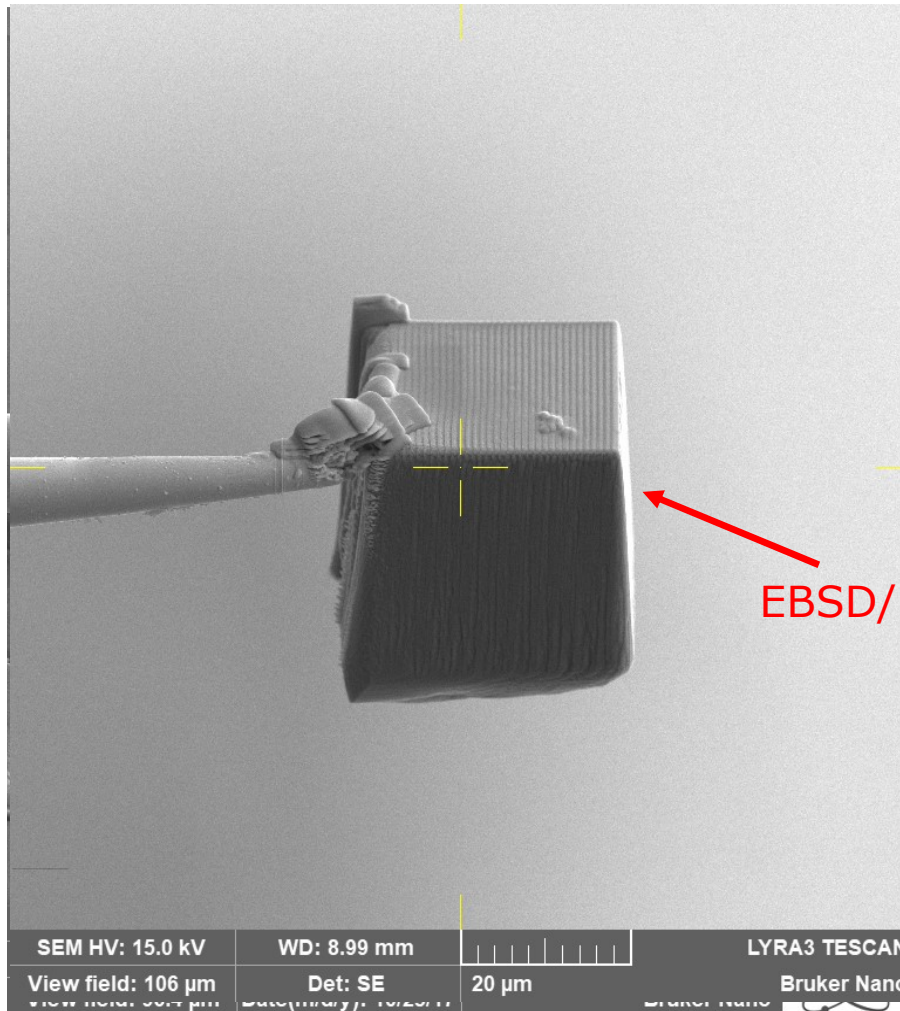
Parameters:

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

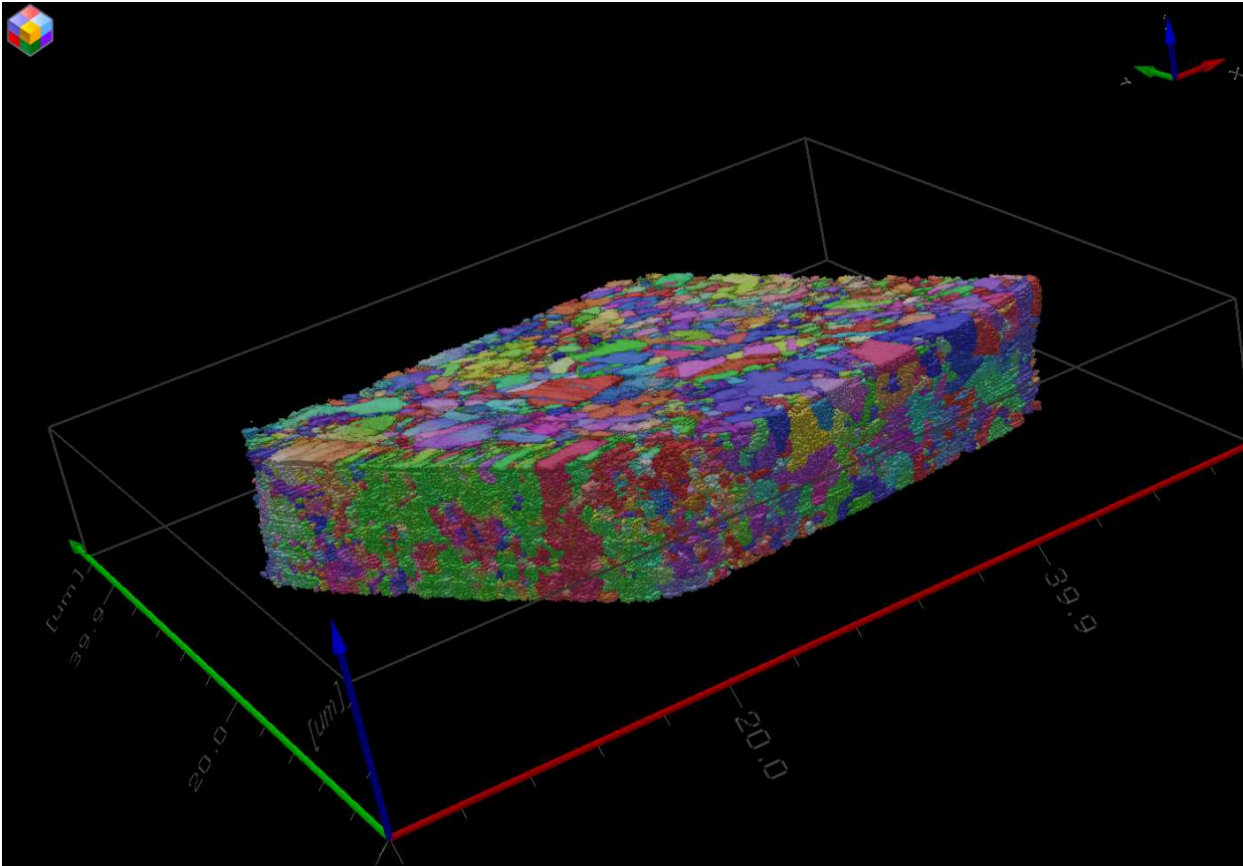


FIB setup

1. Trenches
2. undercut
3. Cube lift out

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3D EBSD processing: data realignment

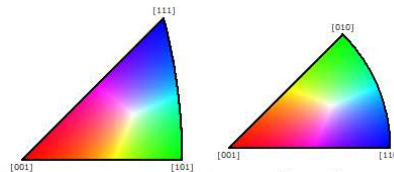


stainless steel
90 slices

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

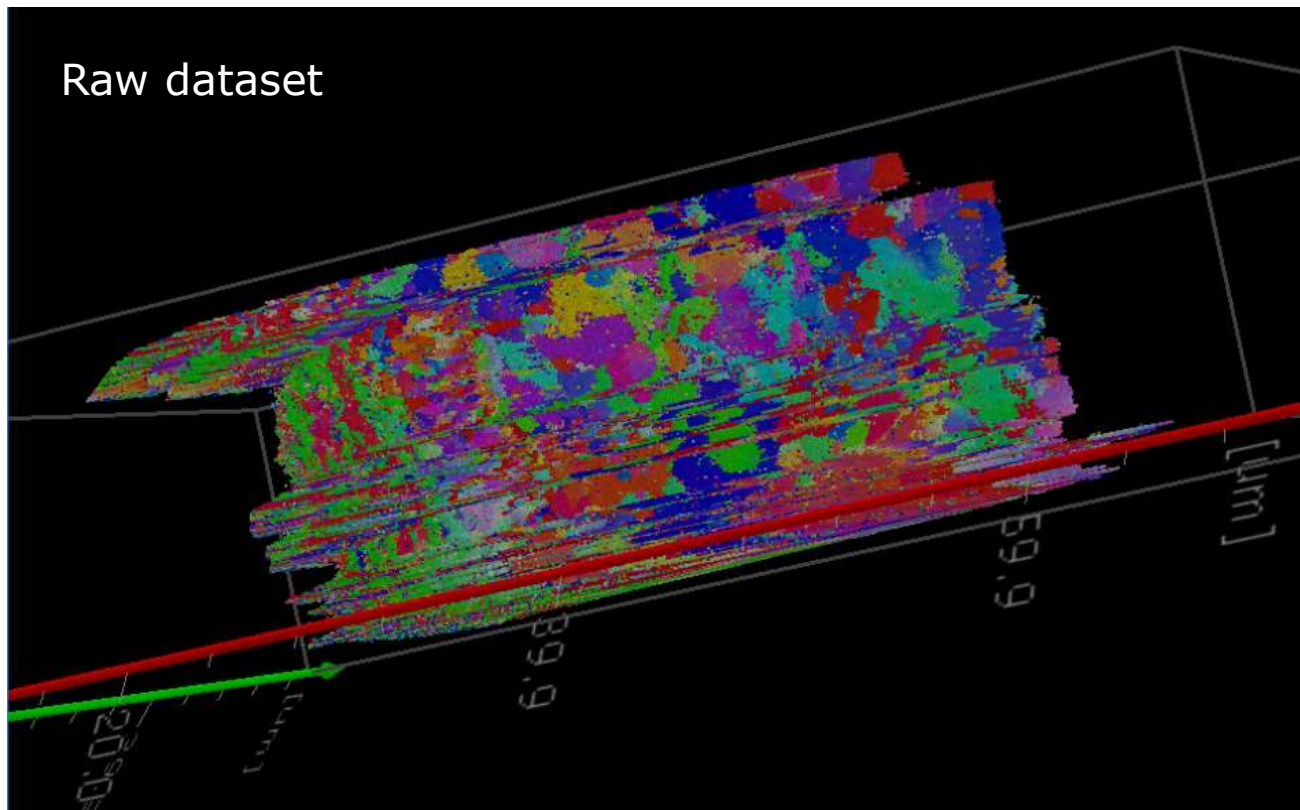


ESPRIT QUBE

3D EBSD processing: data realignment



- Dataset editing: rigid realignment



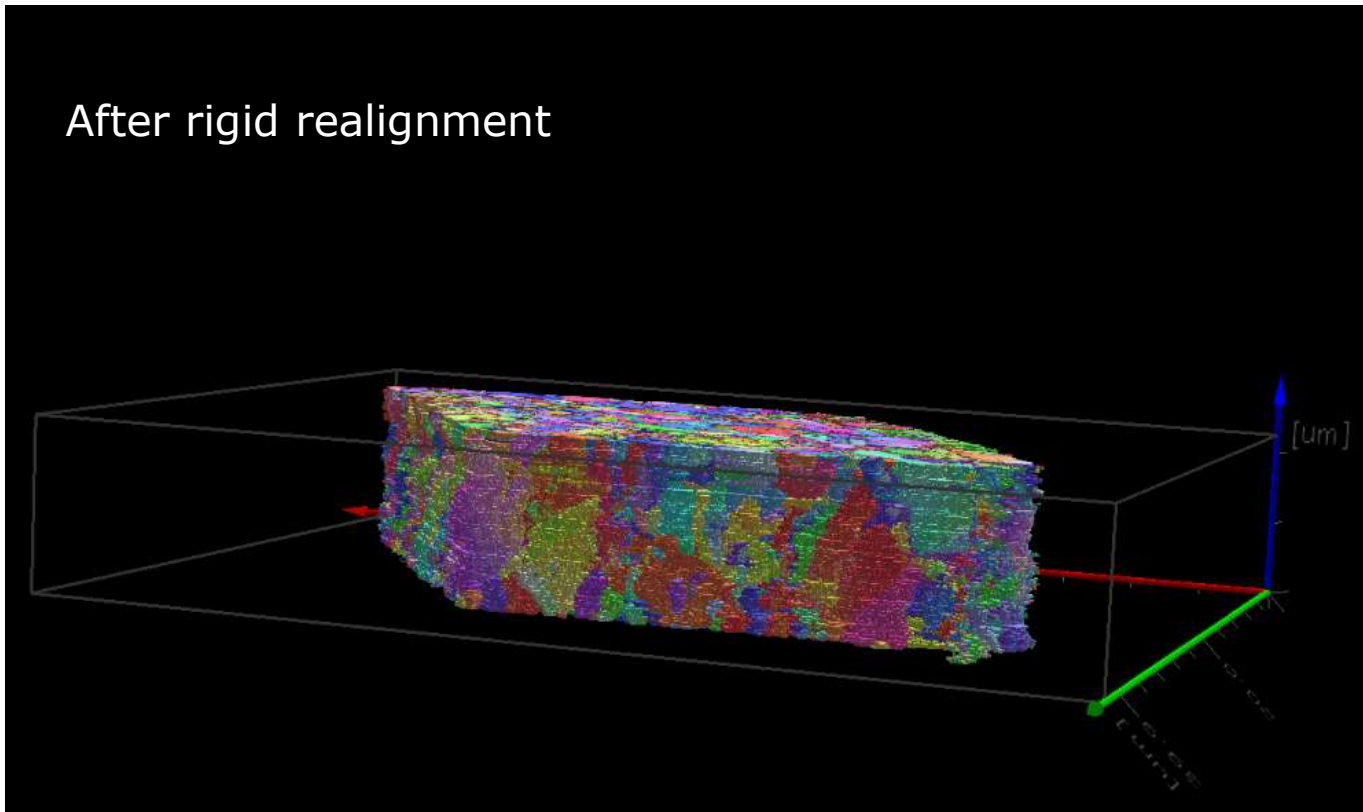
ESPRIT QUBE

3D EBSD processing: data realignment



- Dataset editing: rigid realignment

After rigid realignment



ESPRIT QUBE

3D EBSD processing: data realignment



RIGID REALIGNMENT

Preview Shifts 14

Preview

Data: EBSD
Channel: Red
Magn: 1
Transparency: [slider]

Alignment

ΔX [vox]: 0.000
 ΔY [vox]: 0.000
X-corr (ROI) [/]:
ROI [vox]: 15 557 24 56

Auto Store

44 voxels displacement in X direction

0%

ESPRIT QUBE

3D EBSD processing: data realignment



RIGID REALIGNMENT

Preview Shifts 14

Preview

Data: EBSD
Channel: Red
Magn: 1
Transparency: [slider]

Alignment

ΔX [vox]: 44.000
 ΔY [vox]: 4.000
X-corr (ROI) [/]:
ROI [vox]: 15 557 24 56

Auto Store

44 voxels displacement in X direction

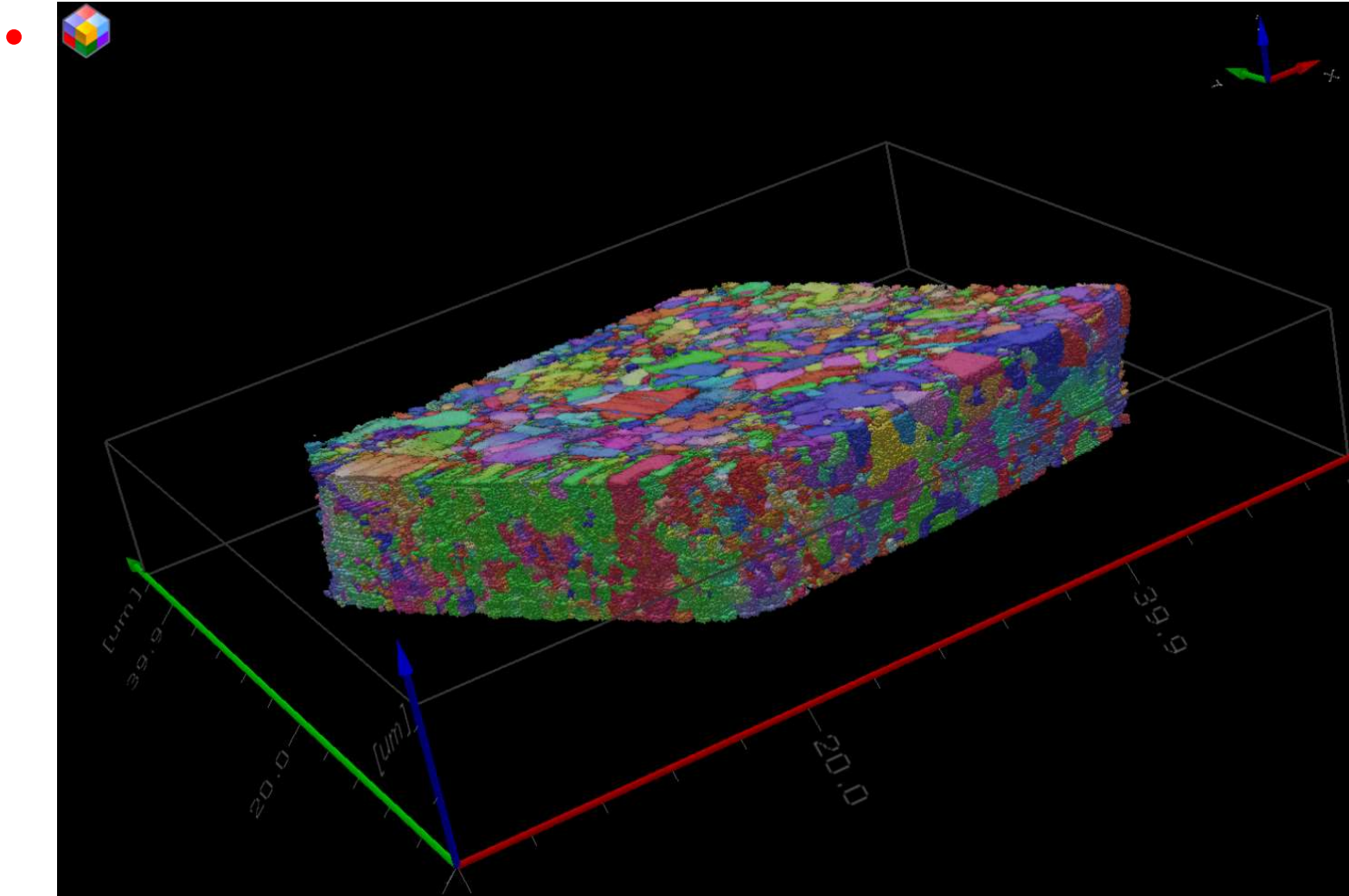
Y
x

0%

Ok
Cancel
Reset all

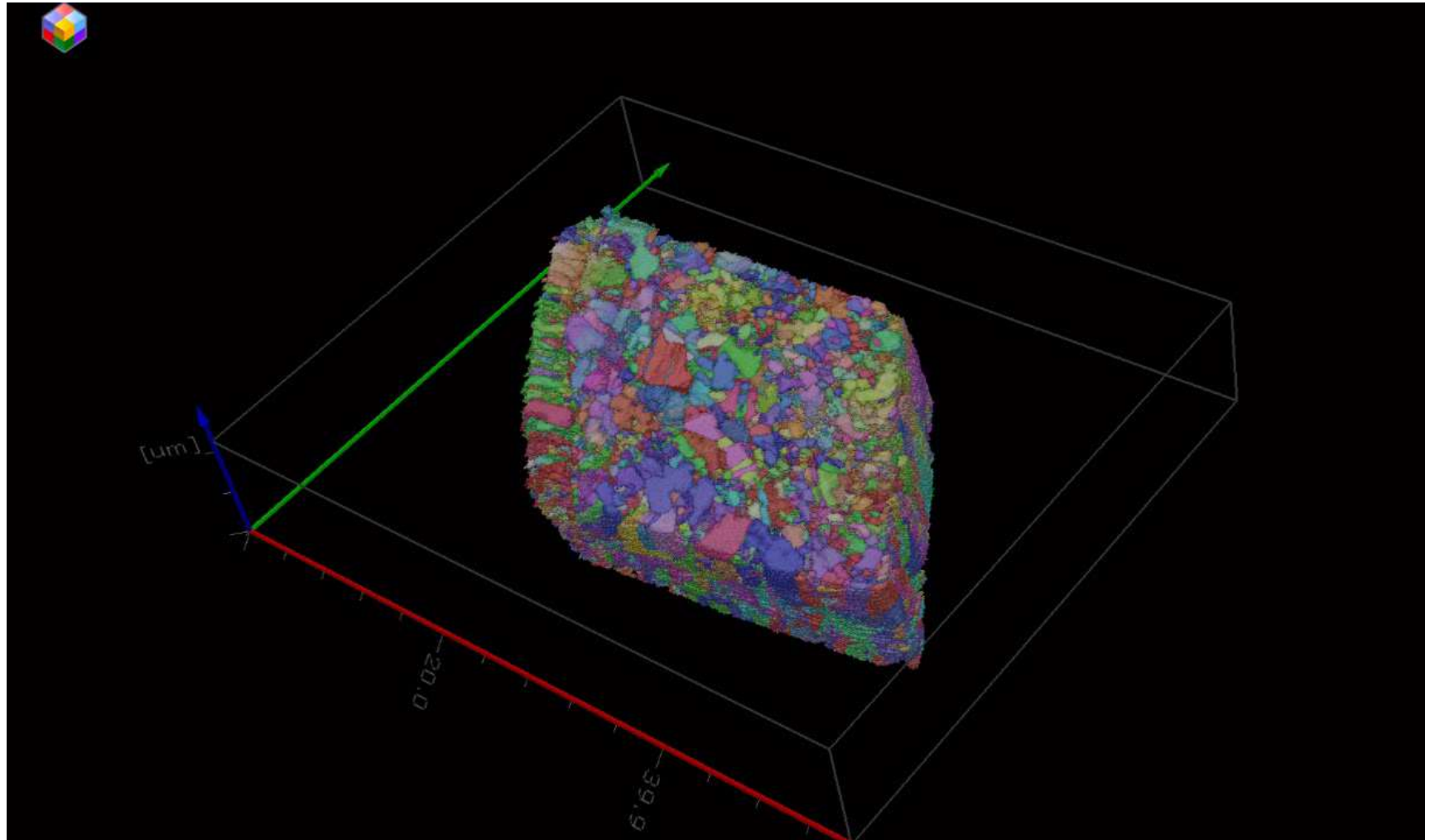
ESPRIT QUBE

3D EBSD processing: data realignment



ESPRIT QUBE

3D EBSD processing: data filtering



ESPRIT QUBE

3D EBSD processing: grain list



UNTITLED - ESPRIT QUBE 2.0.10. - BUILD 3

File Edit Analysis Tools View Help

Project Viewer Subsets Legends 3D View Grain list Event log

Phase: Iron-alpha
Voxel grayscale code:
Indexed bands:
Voxel color code:
IPF

Apply Apply all

EDS
SEM
Tomography

Dice - AND
X: 551 Y: 449 Z: 89
0 0 0
Show 3D cursor

Lighting
Ambient
Diffuse
Specular
 Top Bottom Front

ID	Phase	Ori. mean [Re lm]	Ori. variance	Volume [vox]	Surface [um ²]	CMS [um]	Nei...	Bor...	Ellipsoid [um]	Ellipsoid vol...	Ellipsoid surface [um ²]
8058	1	0.93 0.25 -0.28 -0....	0.0129	228176	2442.52	23.81 27.67 1.99	1119	1	7.67 4.93 2.32	367.82	301.54
9446	0	0.91 0.12 0.35 -0.18	0.0107	115891	686.88	44.84 15.77 3.92	312	1	5.60 4.01 2.21	208.09	191.52
12565	0	0.89 0.25 0.32 0.21	0.0207	107610	1041.96	27.00 16.75 3.79	416	1	8.99 5.08 3.36	643.02	406.25
11717	0	0.93 0.26 0.26 0.07	0.0080	103783	604.92	35.43 32.92 3.63	181	1	4.72 3.66 1.89	136.73	145.42
11802	0	0.89 0.22 0.36 -0.17	0.0116	77366	590.70	22.59 22.39 4.19	348	1	5.45 4.76 2.35	254.53	218.35
12557	0	0.92 0.22 0.13 -0.31	0.0028	76997	621.80	43.76 14.38 5.06	400	1	6.75 5.05 2.33	332.61	274.64
11771	0	0.86 0.08 0.35 -0.35	0.0194	75876	671.48	37.45 18.54 4.29	403	1	5.36 3.86 2.55	221.01	190.27
12703	0	0.94 0.17 0.28 -0.06	0.0164	71585	613.07	39.96 24.66 5.50	297	1	7.11 3.22 2.36	225.78	207.38
12514	0	0.90 0.33 0.27 0.14	0.0238	70690	595.33	13.05 30.57 4.63	200	1	4.48 3.87 1.96	142.32	147.36
9886	0	0.95 0.08 0.30 0.09	0.0268	70233	478.35	9.41 29.91 3.28	215	1	4.38 3.20 1.88	110.35	122.96
7191	1	0.90 -0.17 -0.08 -0....	0.0094	65031	248.14	17.66 20.01 2.60	167	1	4.23 3.14 1.31	73.19	103.96
6046	0	0.94 0.31 0.15 -0.04	0.0183	62944	866.86	19.65 28.48 2.50	292	1	8.56 3.55 2.45	311.61	266.66
8872	1	0.91 -0.23 -0.19 -0....	0.0144	61339	283.48	22.69 30.87 4.06	69	0	4.69 2.72 1.58	84.72	108.22
8373	1	0.97 0.14 -0.02 -0....	0.0133	58881	250.10	21.84 18.79 3.49	103	0	3.46 3.00 1.55	67.24	88.79
9748	0	0.87 0.24 -0.42 -0....	0.0165	56131	466.86	33.65 26.20 3.97	174	1	4.82 3.64 1.67	123.13	141.57
12707	1	0.98 0.20 0.01 -0.03	0.0074	54422	253.01	23.71 22.47 7.40	121	1	4.31 2.12 1.77	67.66	88.83
7743	0	0.90 0.20 0.29 -0.24	0.0096	53077	395.89	39.73 26.58 2.57	128	1	4.24 3.45 2.11	129.19	132.75
9995	0	0.81 0.29 -0.45 -0....	0.0888	51816	590.09	13.33 26.04 3.26	253	1	6.69 3.57 2.04	203.89	199.25
12796	0	0.91 -0.40 0.06 -0....	0.0064	51491	723.77	32.36 32.12 5.10	263	1	5.68 2.85 1.56	105.92	132.47
12291	0	0.95 0.29 0.03 0.10	0.0170	51107	562.35	20.93 14.10 3.69	156	1	5.38 3.00 1.47	99.68	128.71
7844	0	0.89 0.11 0.28 -0.35	0.0028	46805	291.08	28.06 16.17 4.16	154	0	5.75 2.62 1.43	90.52	122.39
5883	1	0.95 -0.13 -0.04 -0....	0.0109	45960	186.17	28.11 24.35 2.73	94	1	3.43 2.70 1.39	53.80	77.99
12799	0	0.91 0.18 -0.22 -0....	0.0213	44797	508.10	24.33 32.05 5.47	291	1	4.12 3.16 2.45	133.66	130.98
10532	0	0.95 0.04 -0.13 -0....	0.0021	44308	326.03	38.43 23.32 3.13	177	1	5.52 3.53 1.21	98.70	143.21
7491	1	0.93 -0.16 0.00 -0....	0.0149	43348	208.74	15.47 24.63 3.71	78	0	2.98 2.37 1.81	53.33	70.86
10546	0	0.91 0.34 0.24 -0.07	0.0138	43040	450.76	21.73 31.23 4.93	205	1	6.01 3.23 2.80	227.26	194.45
12708	0	0.94 0.12 0.23 -0.24	0.0159	42585	345.00	34.21 24.99 7.20	224	1	5.64 3.25 1.92	147.80	156.38
12621	0	0.93 0.05 0.36 -0.05	0.0258	42215	537.55	16.45 17.69 6.80	214	1	6.24 3.03 1.68	132.77	154.86
12329	0	0.89 0.08 0.23 -0.39	0.0044	41789	330.07	33.09 12.31 7.54	226	1	3.78 2.39 2.15	81.62	95.03
12728	0	0.93 0.31 -0.20 0.06	0.0769	40122	465.74	11.32 26.26 7.13	189	1	4.02 2.51 2.29	96.86	106.57
9207	0	0.93 -0.12 -0.34 0....	0.0119	38842	626.14	25.74 30.09 1.96	428	1	6.52 3.91 2.29	244.63	217.58
9121	0	0.90 0.15 -0.36 0.20	0.0088	38614	318.65	38.27 12.80 3.42	171	0	3.88 2.62 2.06	87.70	100.52
10297	1	0.99 0.09 -0.03 0.09	0.0081	36690	285.66	15.62 33.23 3.89	98	1	3.07 2.82 1.58	57.14	77.36
12793	1	0.90 -0.27 -0.02 -0....	0.0037	36277	335.95	13.19 31.05 7.17	78	1	5.31 2.48 1.45	80.08	109.62
7907	1	0.95 0.31 0.04 0.07	0.0143	35898	488.54	33.17 21.95 2.74	179	0	3.37 2.70 1.78	67.93	85.27
12362	0	0.88 0.28 0.37 -0.08	0.0141	35613	365.62	31.60 16.32 5.31	207	1	4.28 2.73 1.73	84.63	103.64
9917	0	0.91 -0.02 0.29 -0....	0.0123	34201	333.62	26.66 14.91 2.93	111	1	4.38 2.91 2.40	128.67	129.18
12374	0	0.90 0.38 0.01 0.20	0.0212	30267	332.78	22.27 20.40 4.67	180	1	6.46 4.03 1.02	111.73	181.40
10227	0	0.96 -0.28 0.04 0.04	0.0115	28454	295.47	33.21 29.05 2.75	131	1	5.06 4.12 1.84	160.68	168.06
5093	1	0.94 -0.14 0.11 -0....	0.0038	27248	171.54	38.39 26.59 1.80	41	1	2.75 2.06 1.63	38.68	57.32
9079	0	0.93 0.23 0.24 -0.13	0.0073	27237	182.24	38.74 27.84 4.99	58	0	3.23 2.33 1.56	49.34	69.80
5800	1	0.84 -0.23 0.37 -0....	0.0051	27169	290.64	12.83 32.24 1.97	79	1	3.35 2.26 1.33	42.41	65.86
10550	0	0.97 -0.24 -0.09 0....	0.0143	26787	231.05	18.08 28.17 5.78	128	1	3.50 2.38 1.55	54.08	75.45
11454	0	0.91 0.06 0.29 -0.28	0.0170	25088	211.19	28.39 22.63 6.76	143	0	3.25 1.93 1.73	45.38	64.95

0%

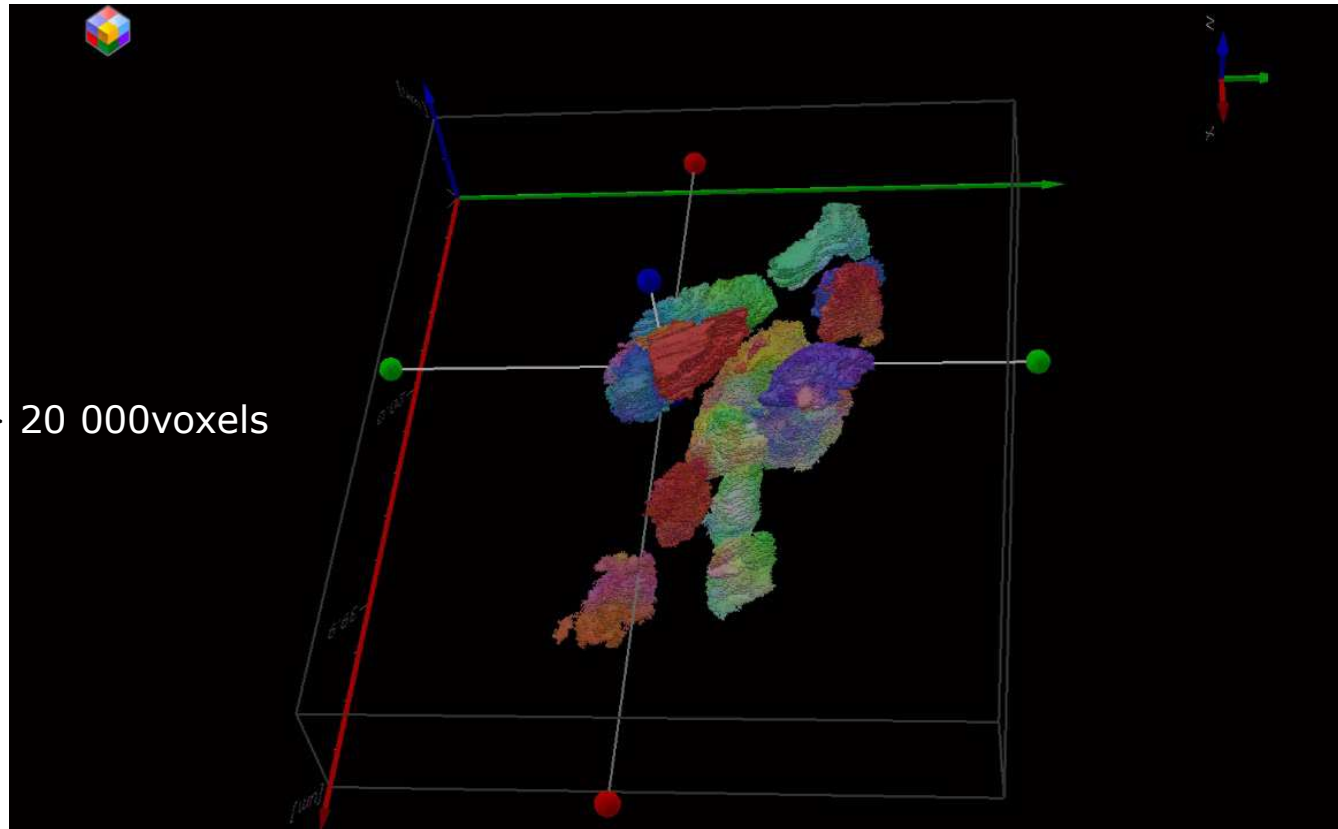
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3D EBSD processing



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

Grains containing > 20 000voxels



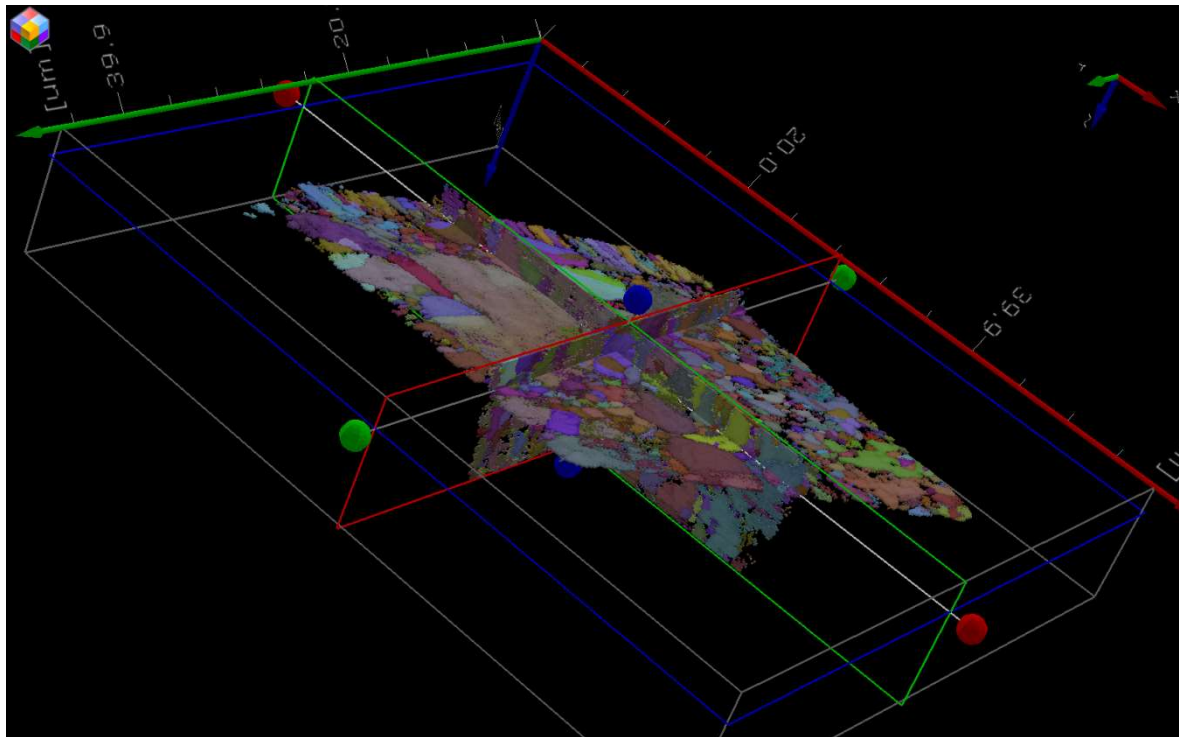
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3D EBSD processing



Main features:

- Visualization, slicing & dicing of EBSD data cubes

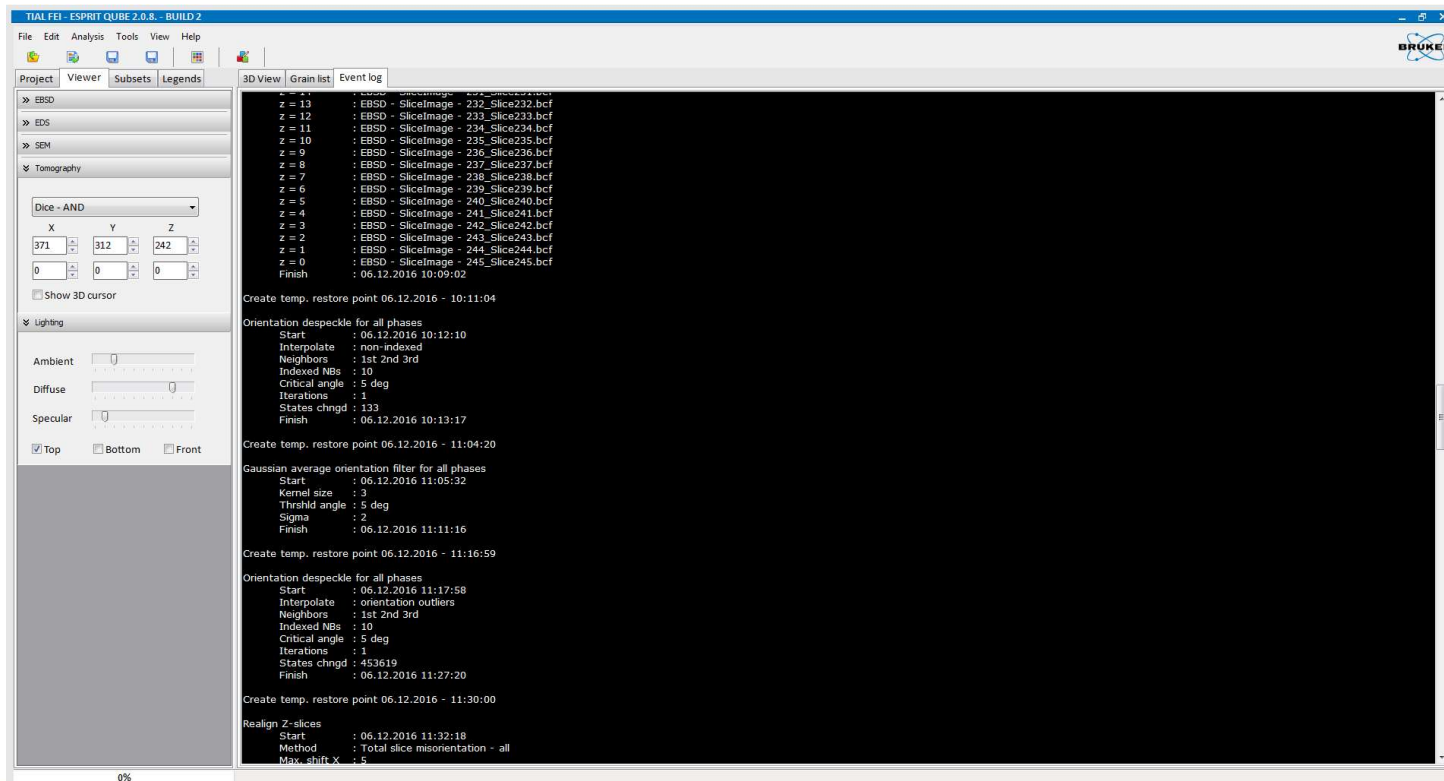


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3D EBSD processing



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



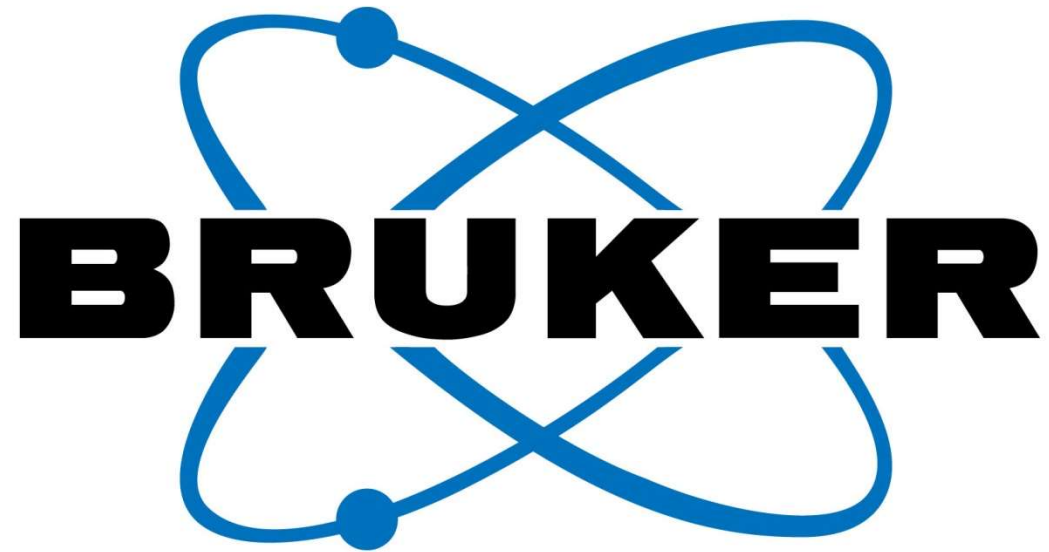
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Advanced 3D data postprocessing & visualization



Summary

- Unit **Quaternion** 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