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Tomographie Tribeam (MEB-FIB-Laser) : un outil pour la reconstruction 3D de microstructures

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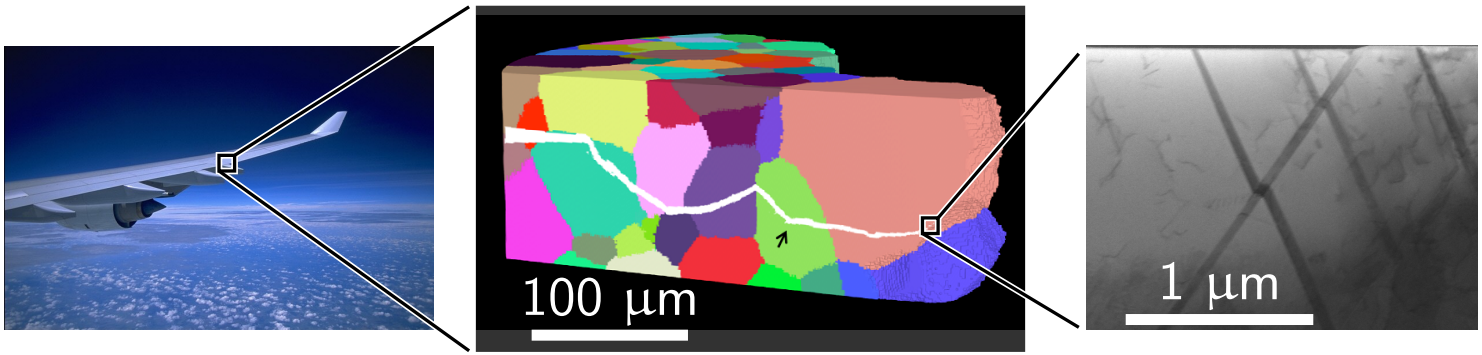
Impact of 3D material science for structral materials



structure microstructure nanostructure

- Fatigue : 80% of the world failures in service
- >50% of the fatigue life corresponds to growing short cracks
- 3D microstructure controls the propagation
- Double challenge : observe inside opaque materials, predict (mul)

Solution : 3D microstructure characterisation coupled to damage/deformation observations.

→ ultimately this research aims to improve design methods and microstructure optimisation

Contents

1. 3D grain mapping by TriBeam serial sectioning
2. In situ testing with micro-DIC and TriBeam
   - In situ testing with micro-DIC
   - Site-specific TEM lift-out
   - Tribeam experiment on specimen G8
3. Combined DCT + TriBeam analysis
   - DCT experiment
   - Locating the DCT region within the gage length
   - Specimen preparation for the TriBeam experiment
   - Tribeam acquisition and reconstruction
4. Summary and outlook

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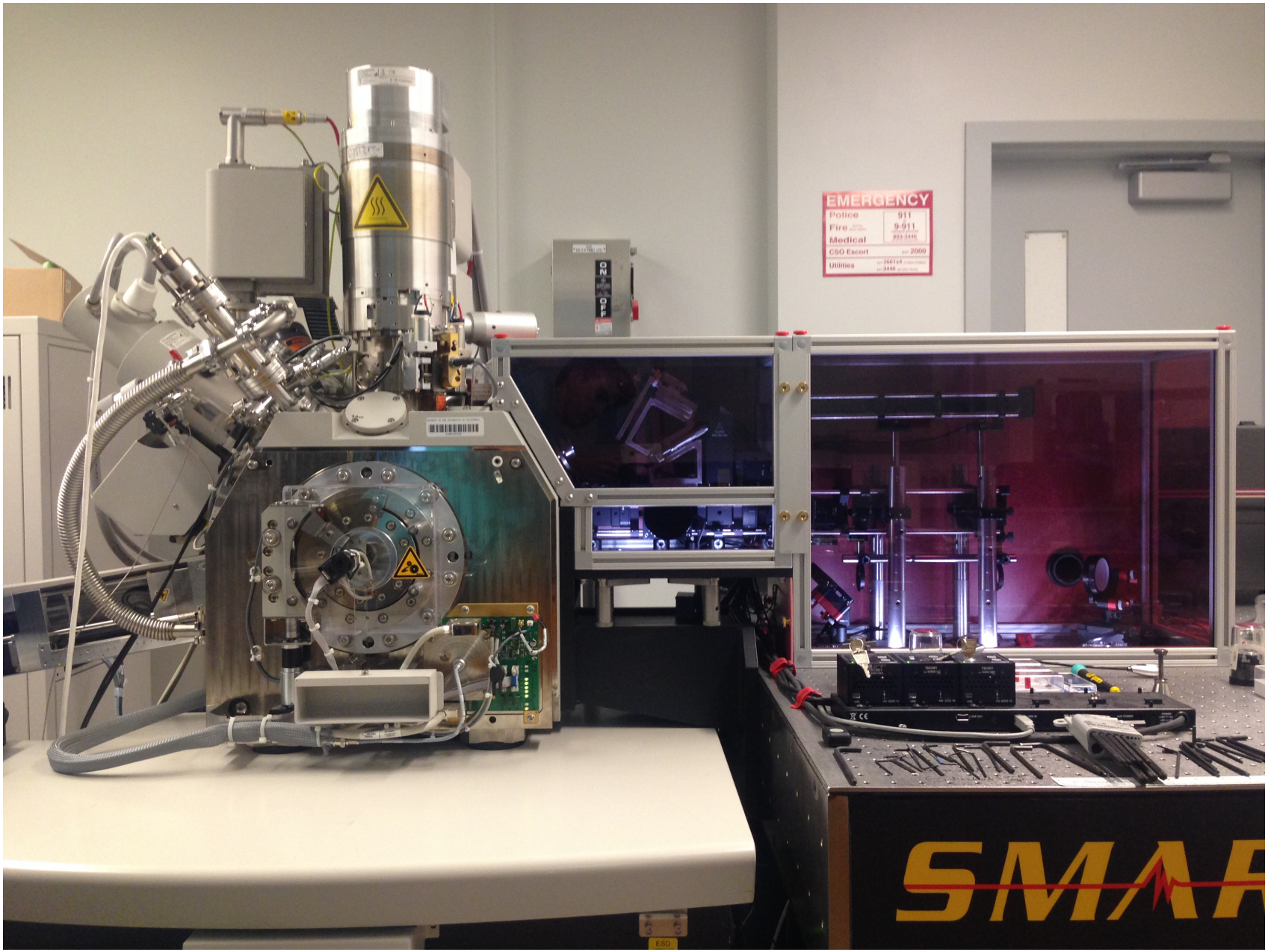
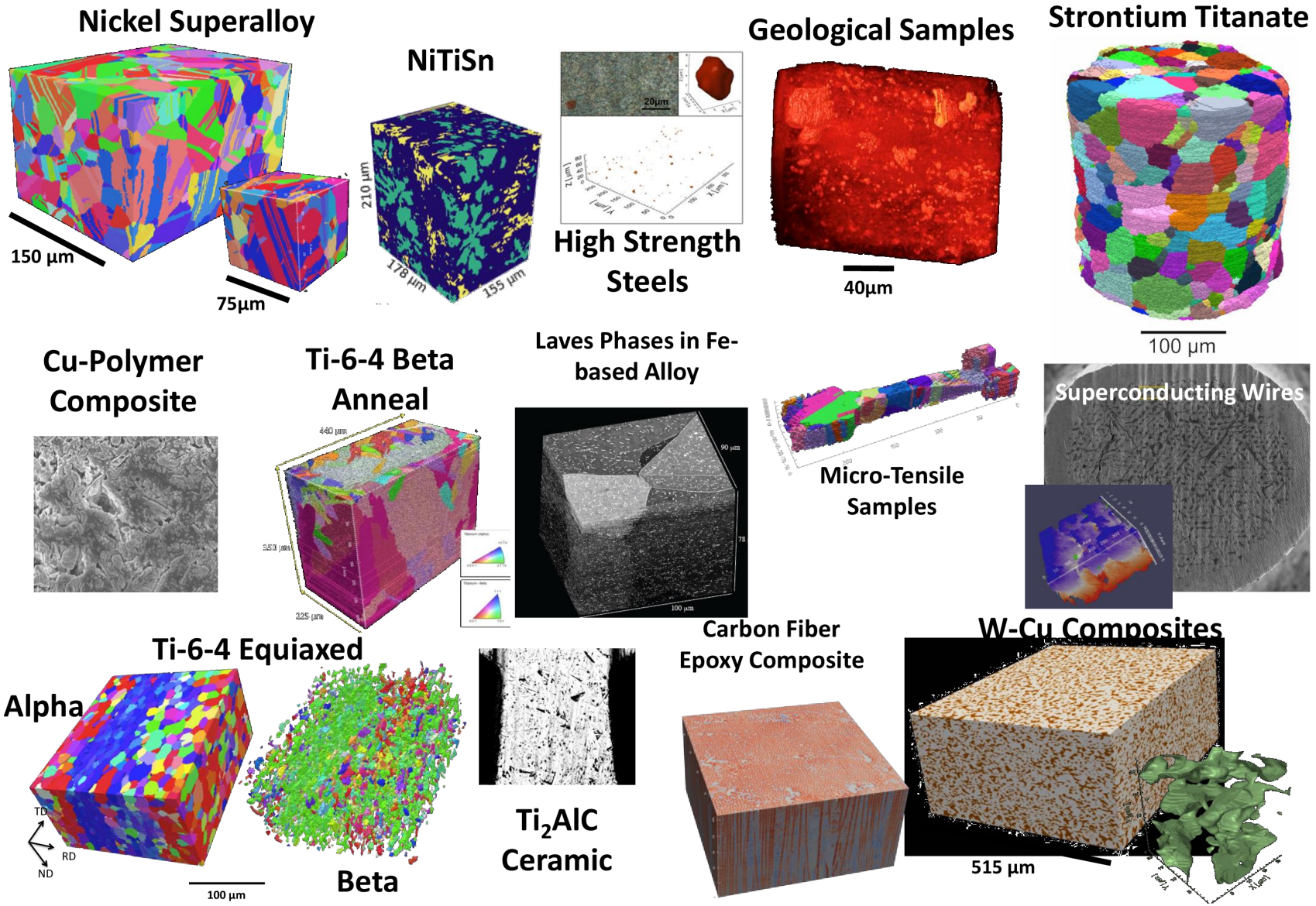
4 Summary and outlook

The tribeam system at UCSB



Tribeam tomography of structural materials



[Echlin et al., 2012, Echlin et al., 2015]

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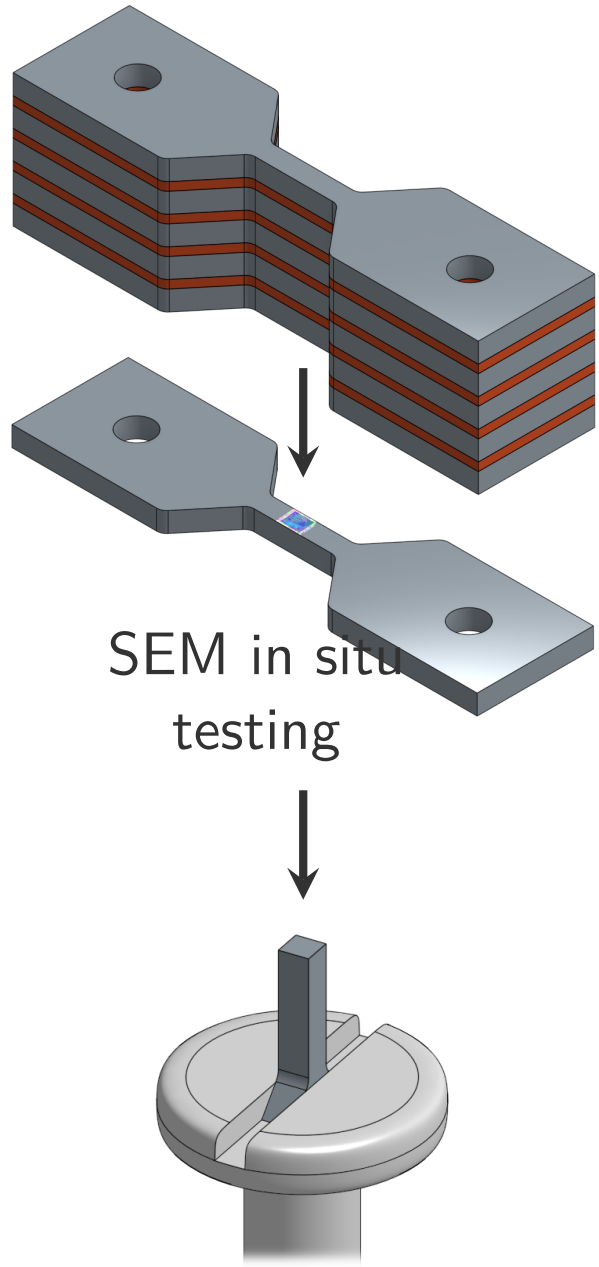
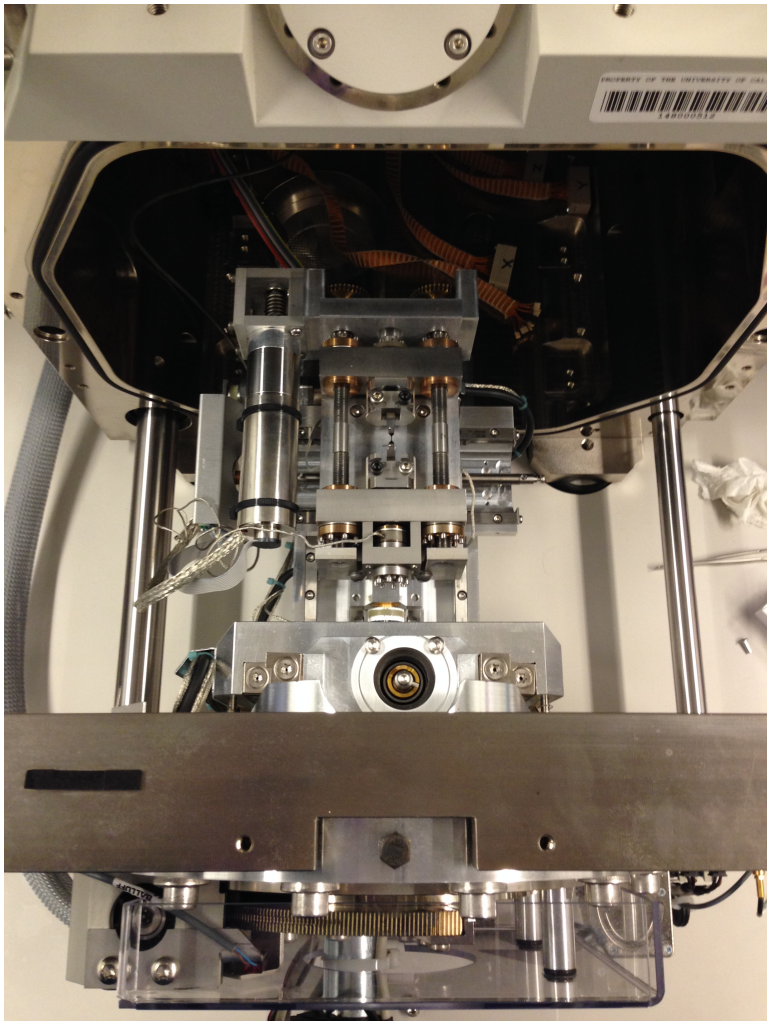
Specimen preparation

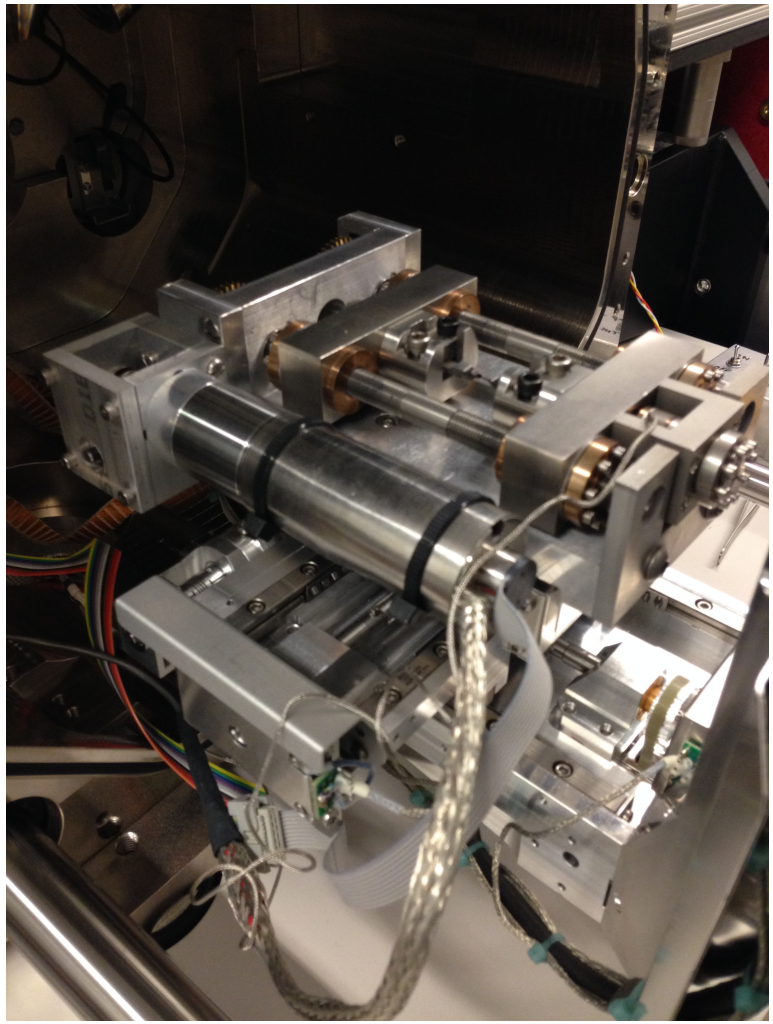
- γ-TiAl (Ti-48Al-2Cr-2Nb) tensile specimen blanks are first EDM cut and polished on the edges to remove EDM damage.
- Specimens are then sliced and both faces are polished down to 1 µm diamond paste.
- Vibromet polishing of top face + surface EBSD.
- Fiducial FIB marking on both edges of the gage length.
- **In situ SEM testing (0.3% strain)**.
- Saw cut of the gage length region and mounting on stub for Tribeam experiment.



In situ tensile stage (Ch. Torbet)

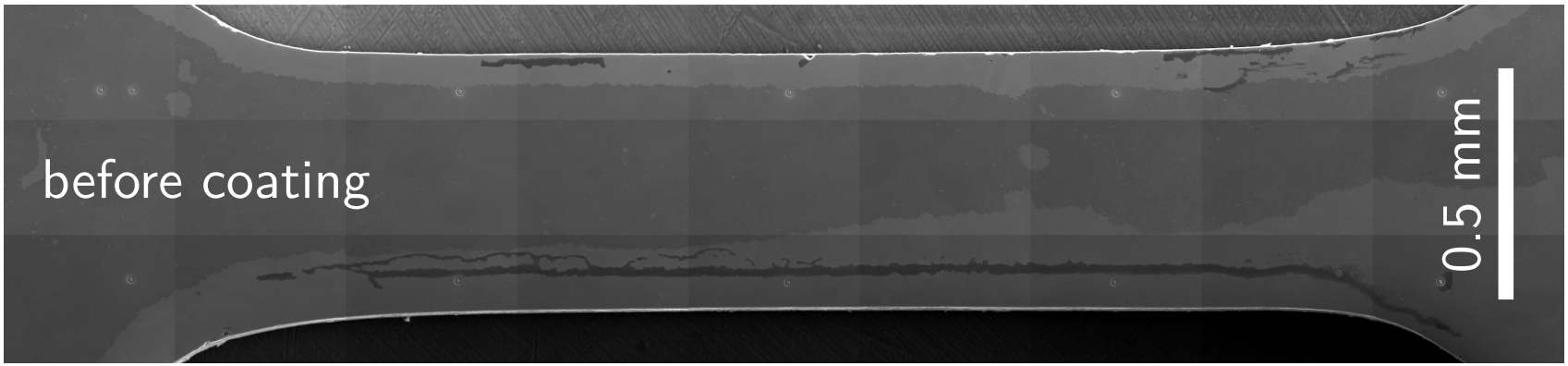
G8 in situ tensile test

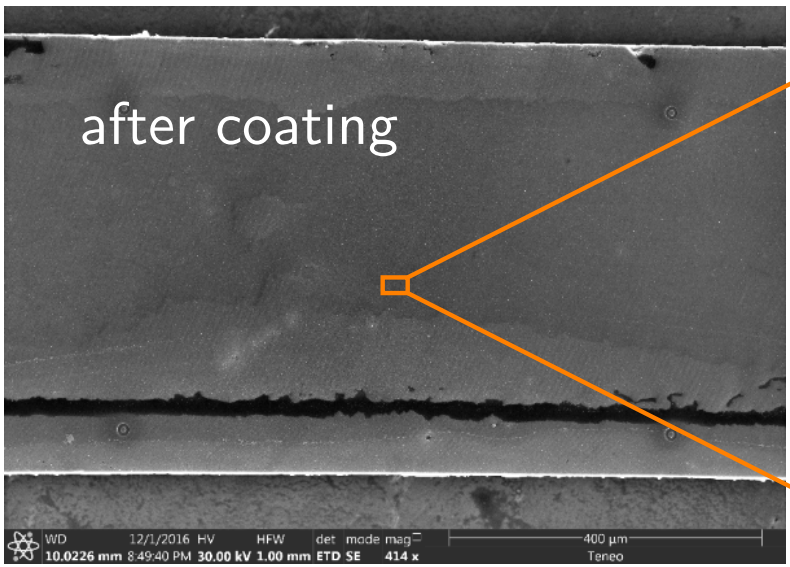


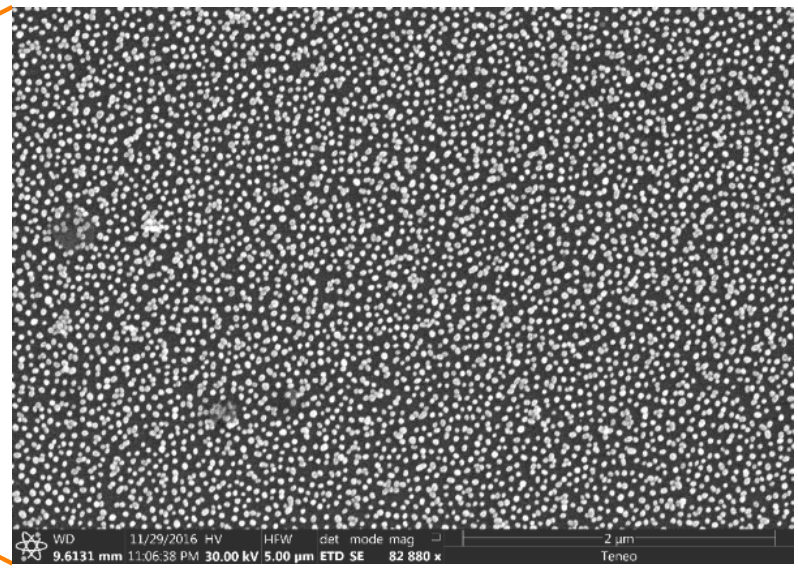


G8 SEM surface observation

G8 in situ tensile test, initial state, coated with 40 nm gold nanoparticles (Zhe Chen, Sam Daly) [Kammers and Daly, 2013b, Kammers and Daly, 2013a, Chen and Daly, 2016]

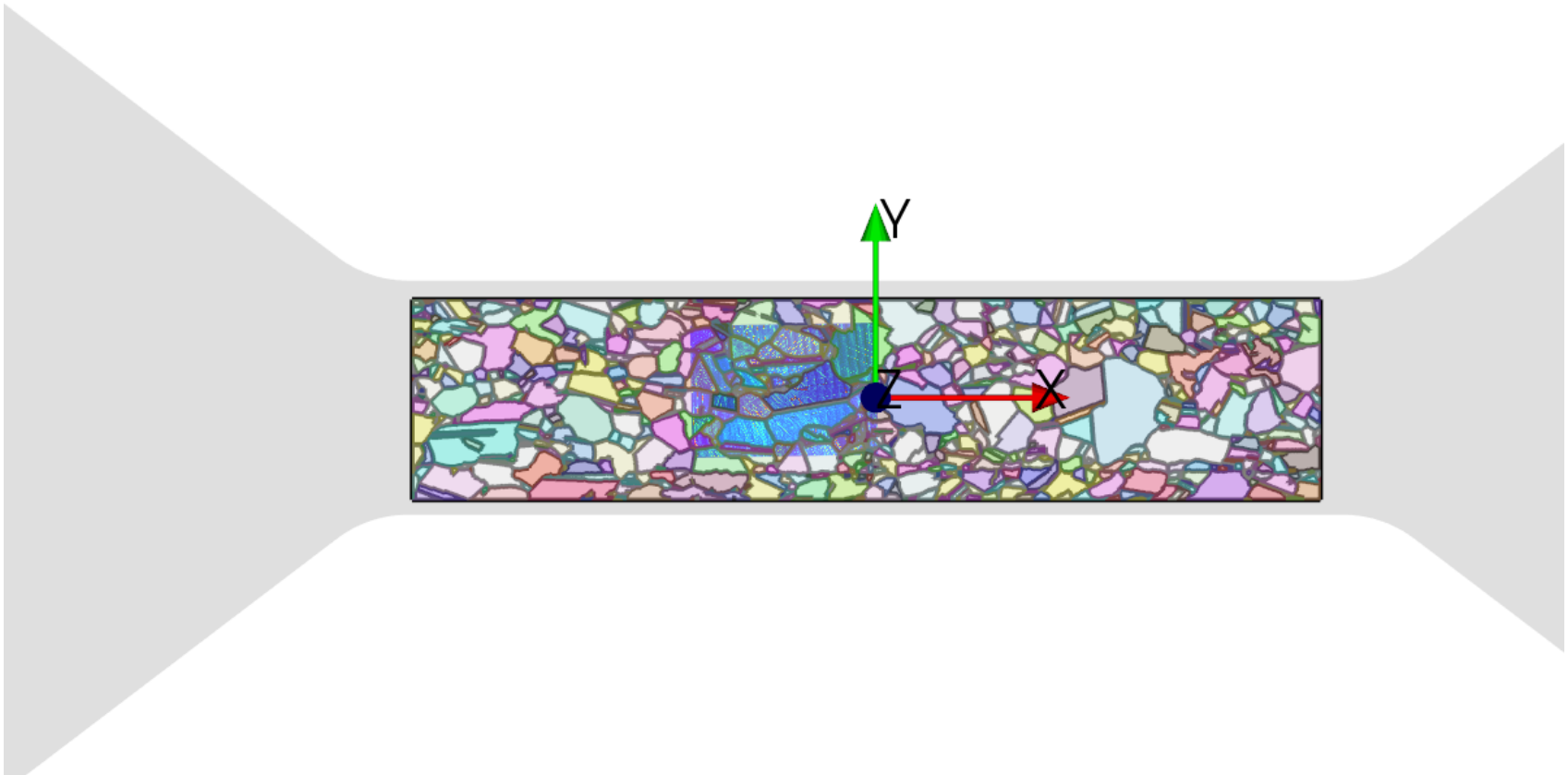






G8 SEM in situ testing : results

- Registration of surface EBSD and DIC data in the laboratory frame
- Spatial resolution 20 nm, 3 loading steps + unloading
- Correlation between localization of the deformation and microstructural features



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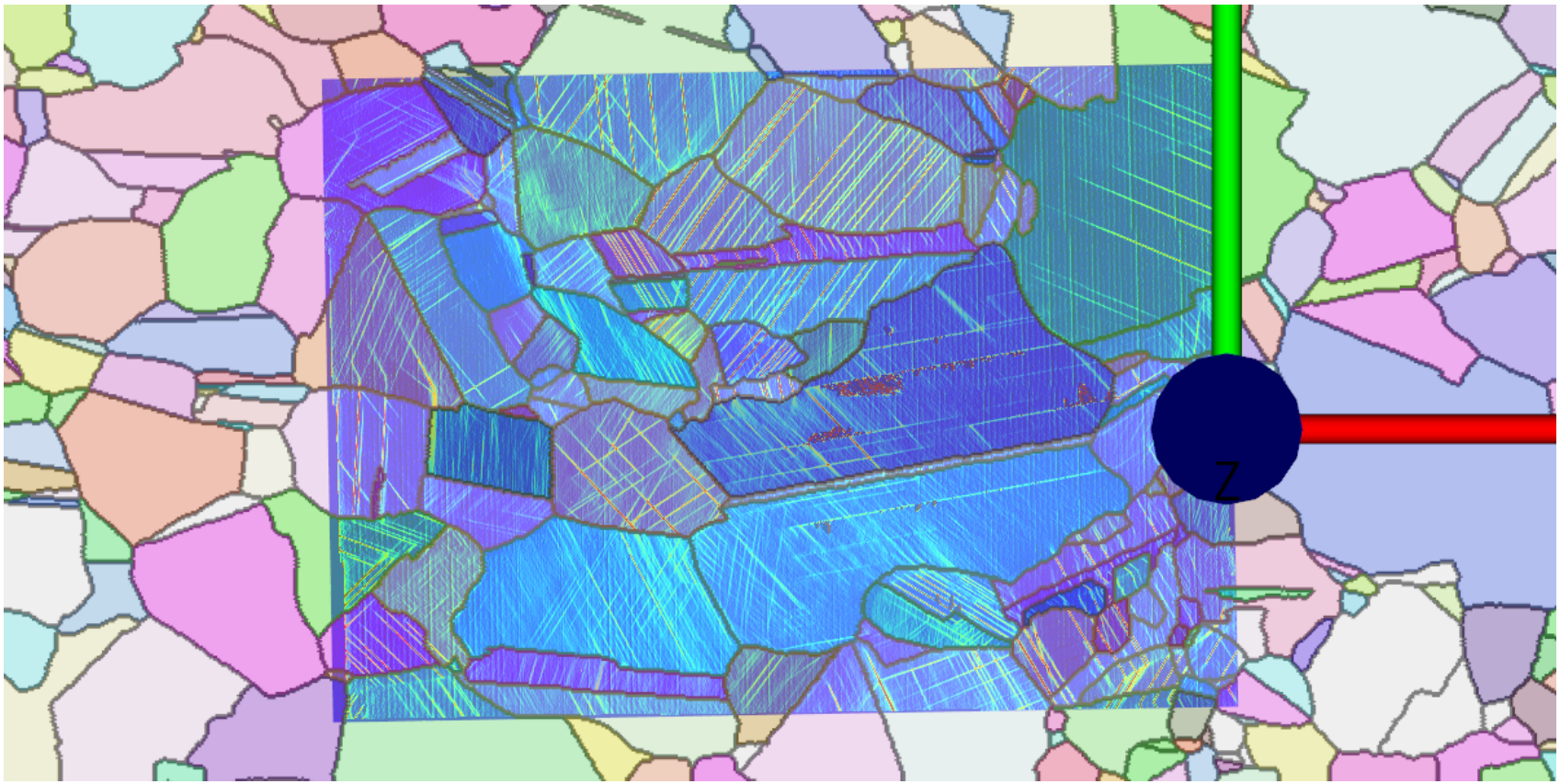

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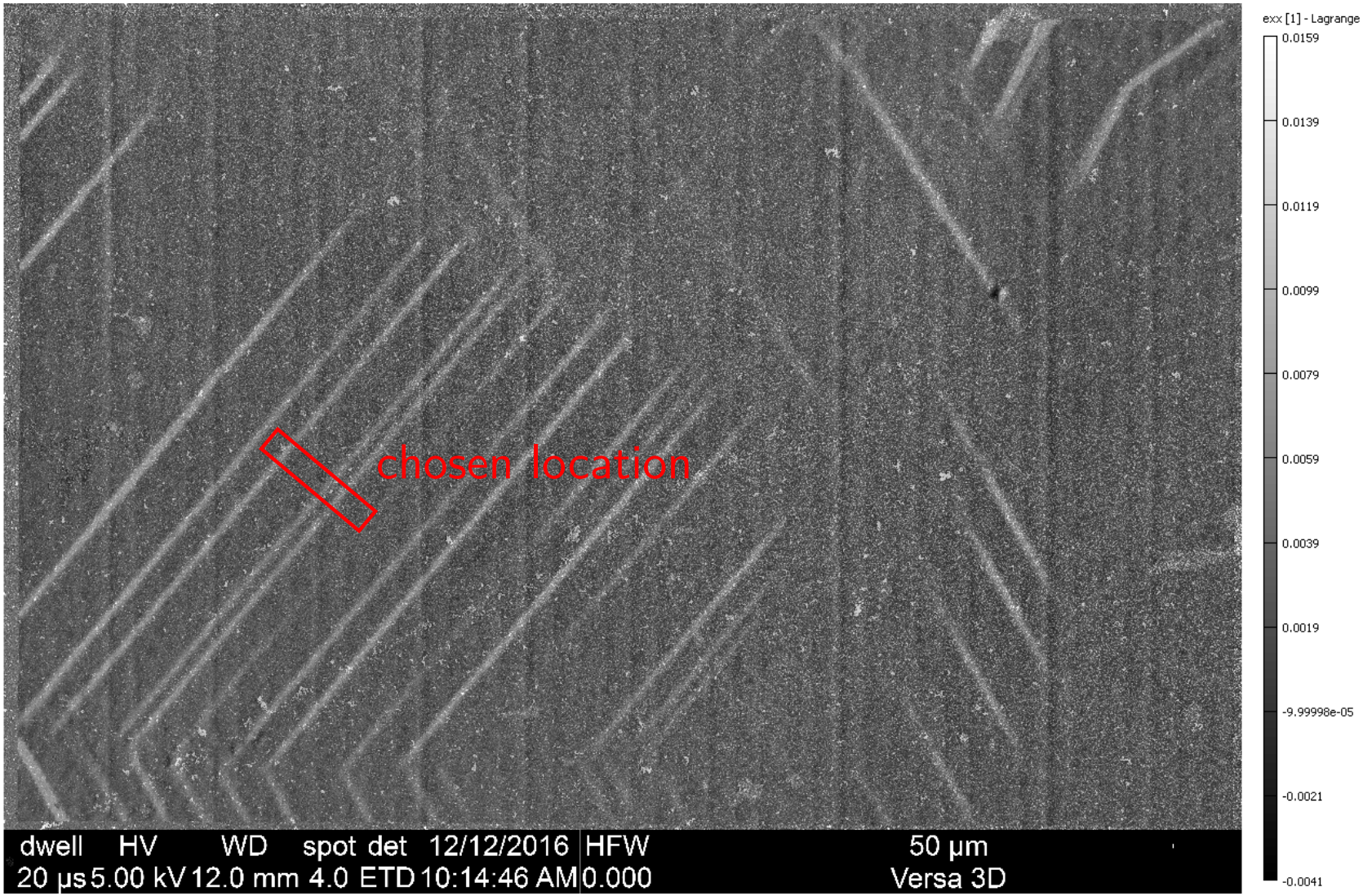
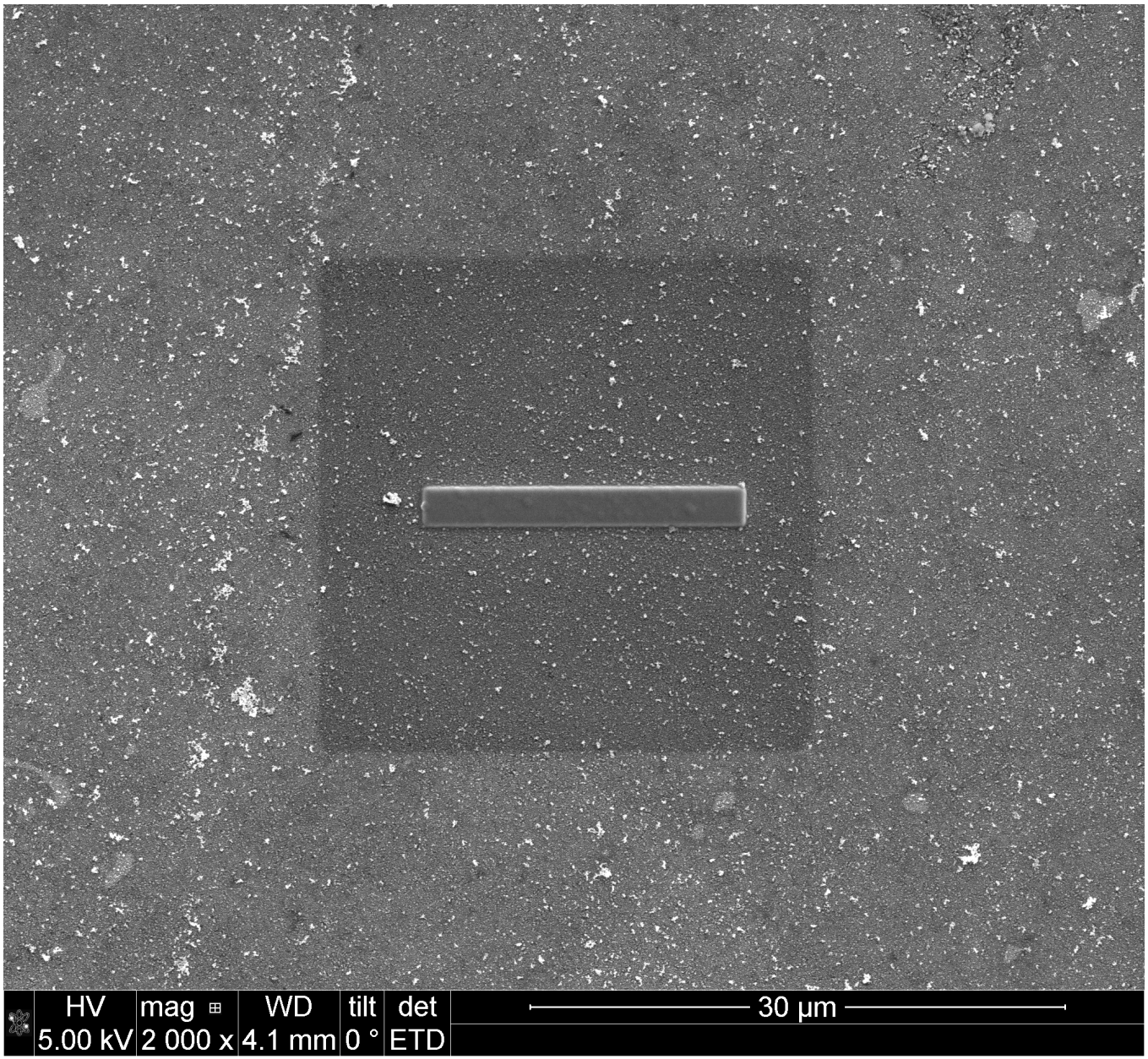
Chose the site using the μ-DIC information

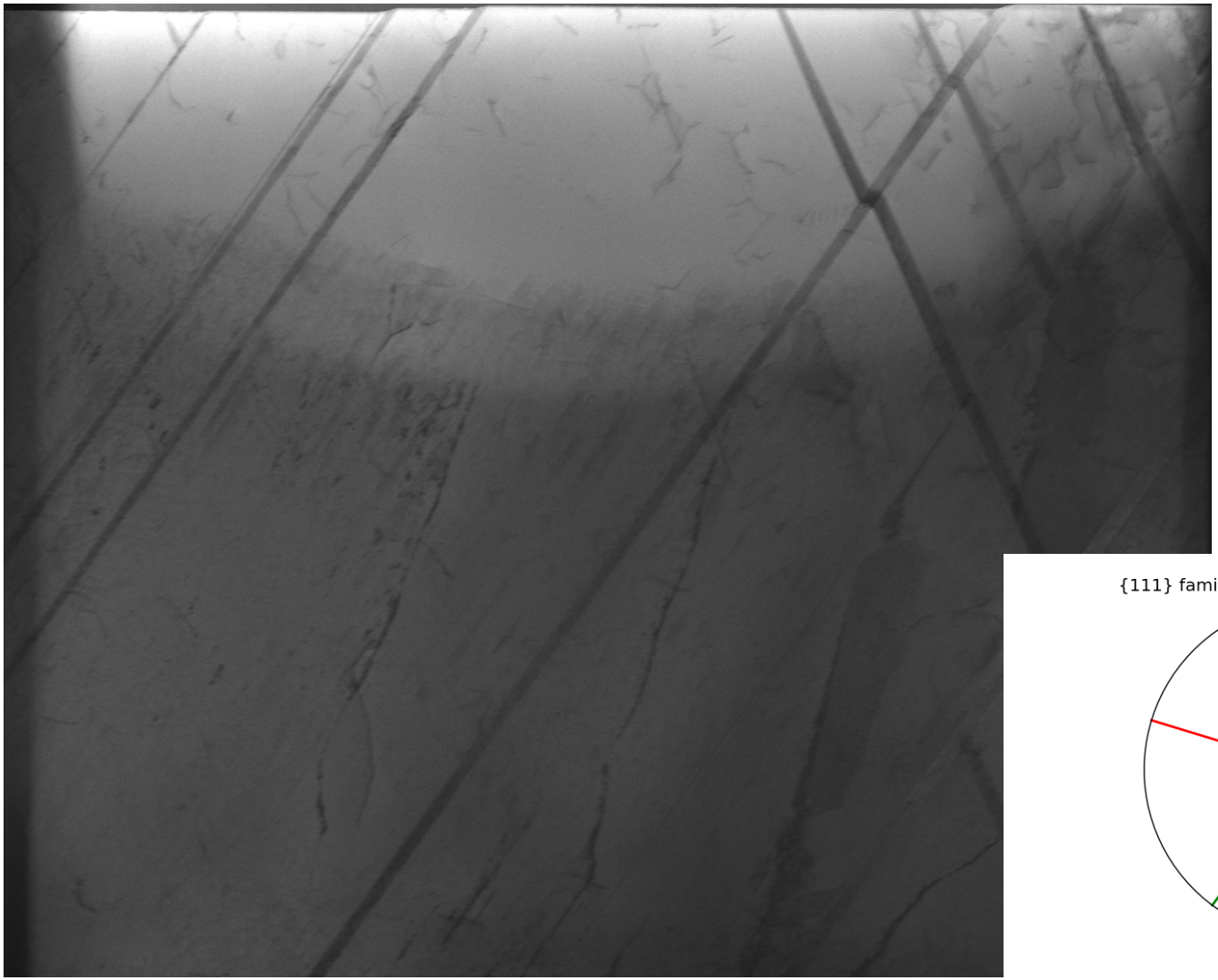


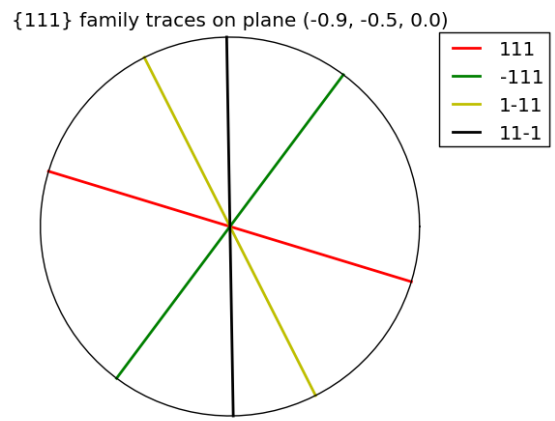
composite image of the DIC results and the SEM speckle

TEM foil preparation using the FIB



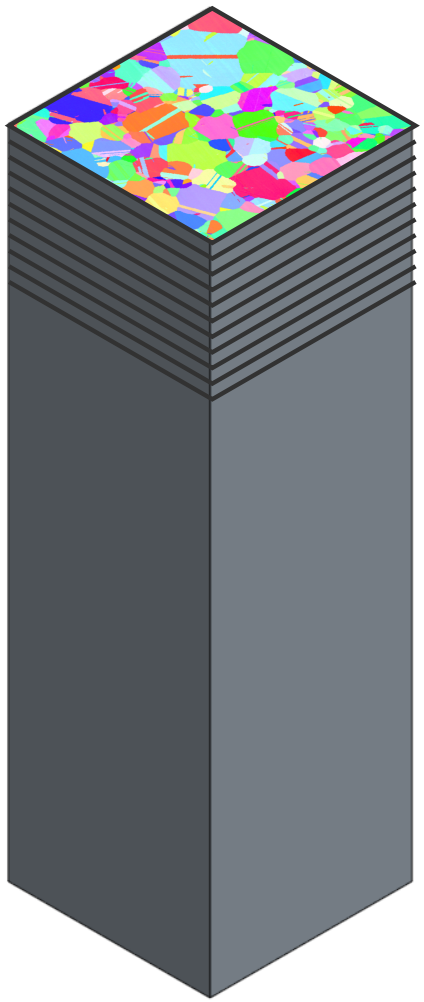
STEM image of the thin foil





STEM imaging reveal a combination of ordinary dislocation / twinning deformation mechanisms.

Tribeam acquisition geometry



Laser miling + data acquisition

Tribeam acquisition sequence :

1. Move sample up 1.5 µm
2. Laser milling (300 mW, 200 passes, 20 sec)
3. Acquire SEM image
4. FIB the surface (30 kV 65 nA, 8 minutes)
5. Get EBSD data (5-10 minutes)
6. Go back to step 1

20-30 minutes/slices, 370 slices have been acquired successfully during several days.

Final reconstructed volume : 560 µm × 400 µm × 555 µm with $\sim$ 2000 grains.

Tribeam data postprocessing



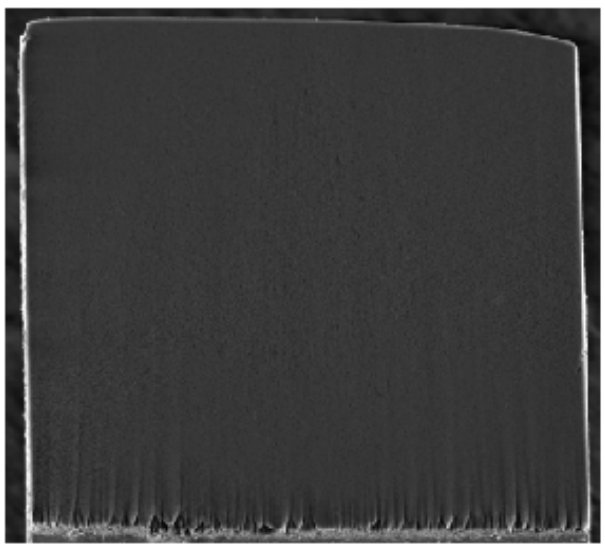
Done via DREAM.3D [Groeber and Jackson, 2014]
`http://dream3d.bluequartz.net`

1. Convert EBSD data into a stack and store it as `hdf5`
2. Correct for in plane distortions
3. Align the images (in plane alignment)
4. Threshold the image quality ($\geq$ 6000 here) to get a mask of the sample
5. Convert orientation data to quaternions
6. Cleaning via neighbor orientation correlation
7. Segment grains using a misorientation criterion (2°) here
8. Remove grains smaller than 5 µm in diameter
9. Export data to paraview or pymicro for visualisation

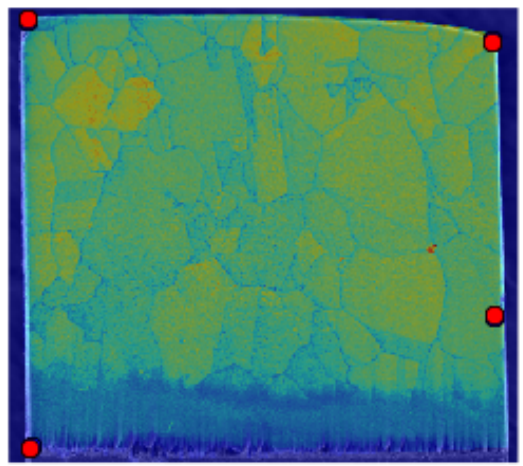
Compute the distortions by point set registration

We use the SE images (or BSE) after each cut to compute the affine transform of the distortions in the EBSD data collection.

$$\underset{\sim}{\mathbf{A}} = \begin{pmatrix} A_{xx} & A_{xy} \\ A_{yx} & A_{yy} \end{pmatrix}$$



SE image after laser cut



registered IQ onto SE image

Compute the distortions by point set registration

We use the SE images (or BSE) after each cut to compute the affine transform of the distortions in the EBSD data collection.

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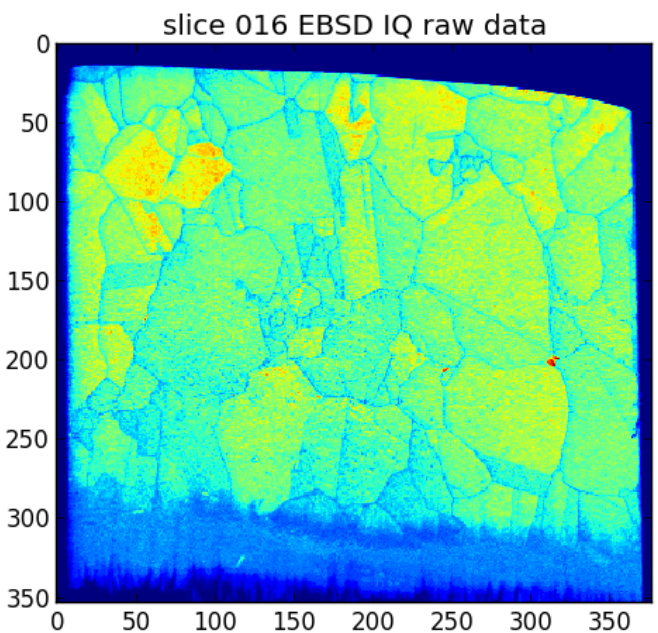
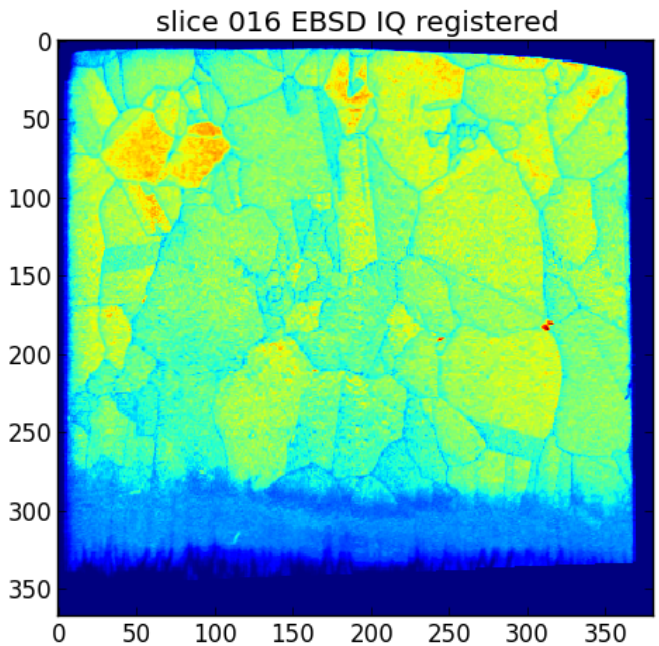


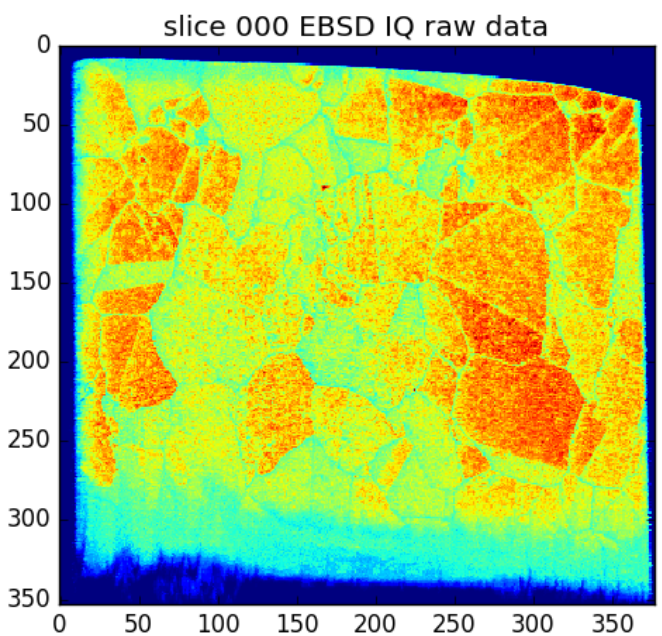


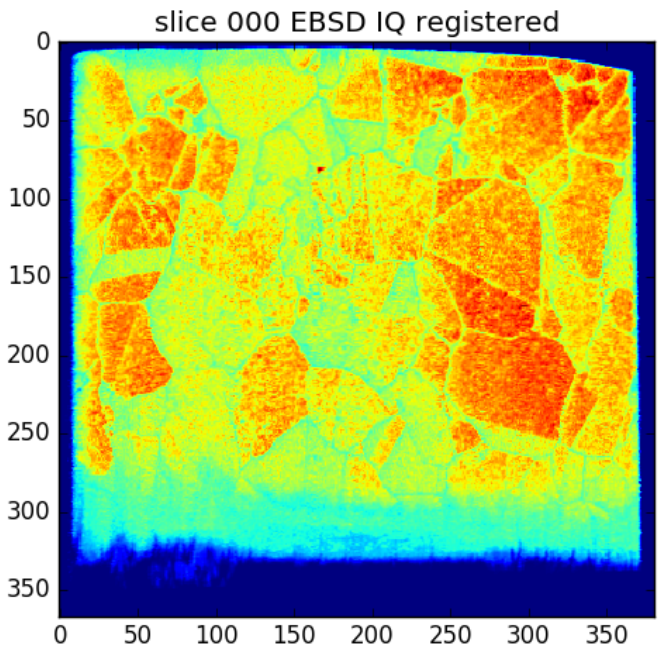
Also correct for magnification errors

The experiment was done in two parts, the EBSD camera was serviced in between $\rightarrow$ magnification calibration is a little different.

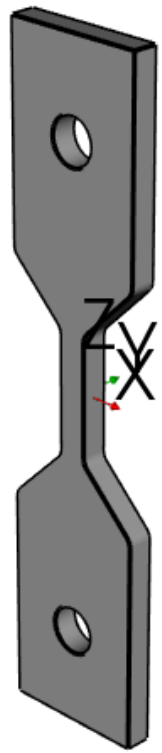
$$\underset{\sim}{\mathbf{A}}^{-1} = \begin{pmatrix} M & A_{xy} \\ A_{yx} & M \end{pmatrix}$$

with $M = 1.025$ for slice ≥ 140 (after the restart).

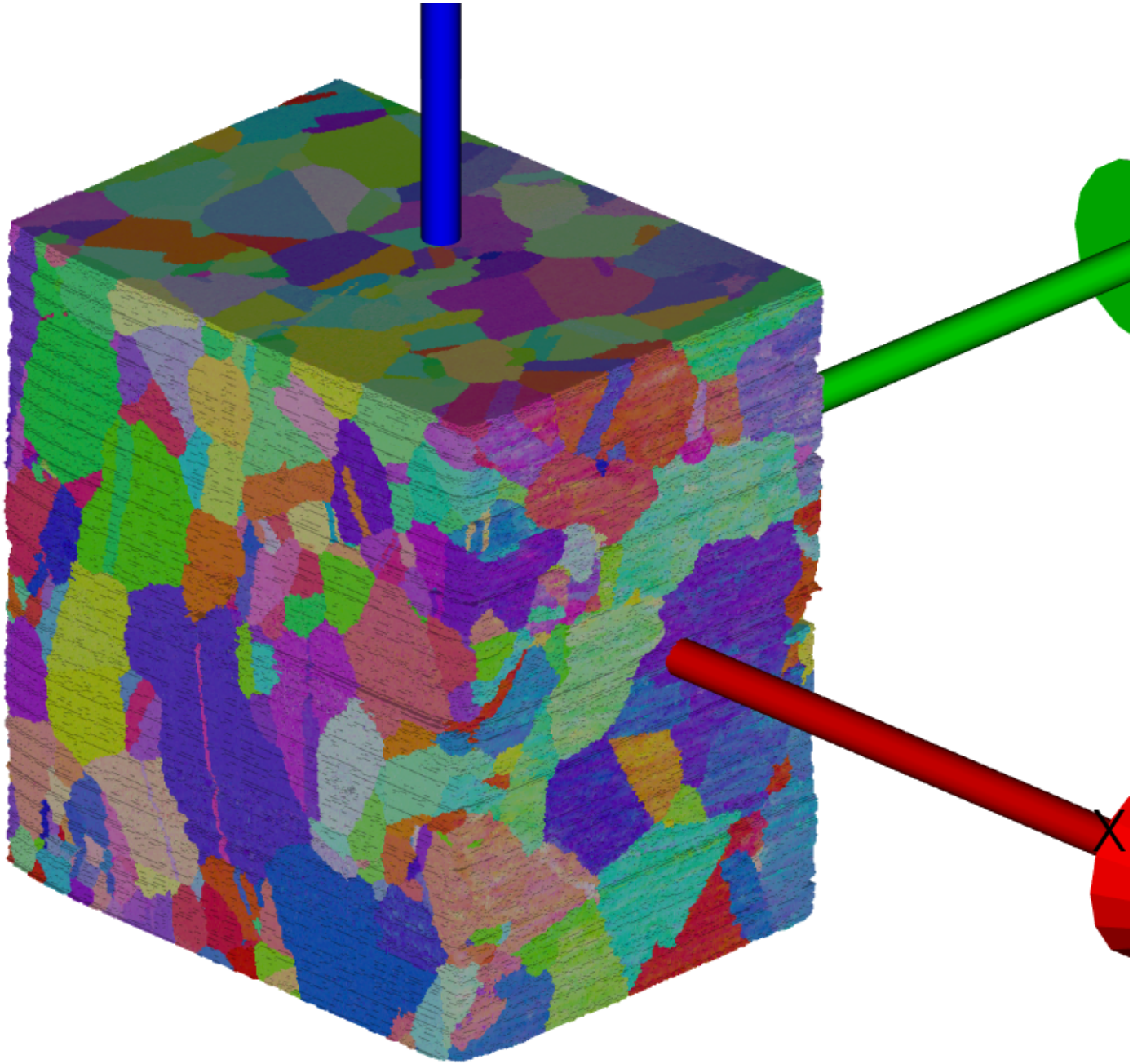




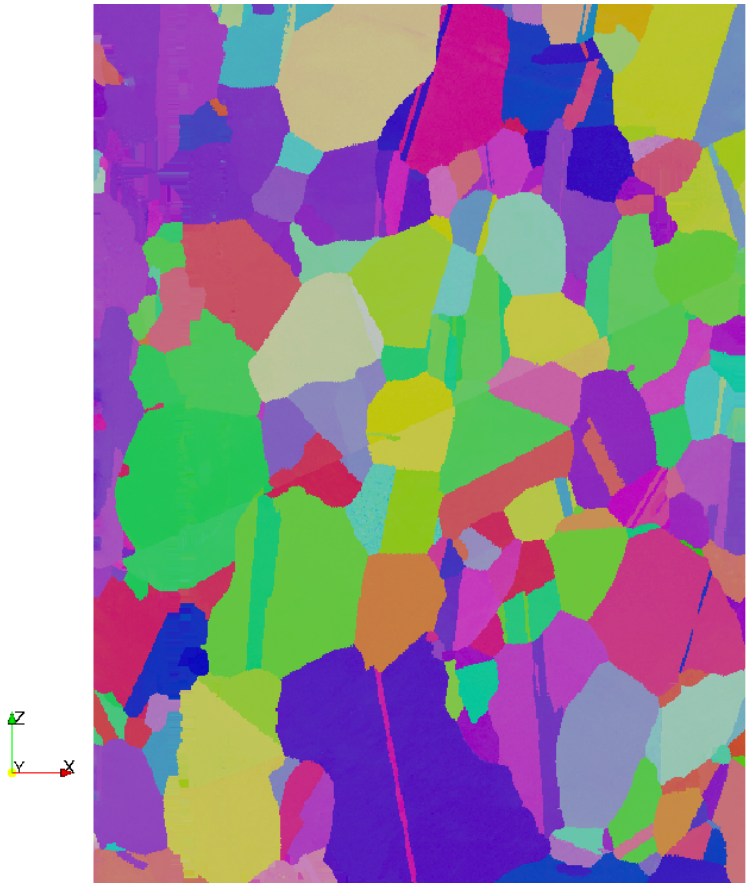
Final reconstruction



Final reconstruction



Comparison with surface EBSD





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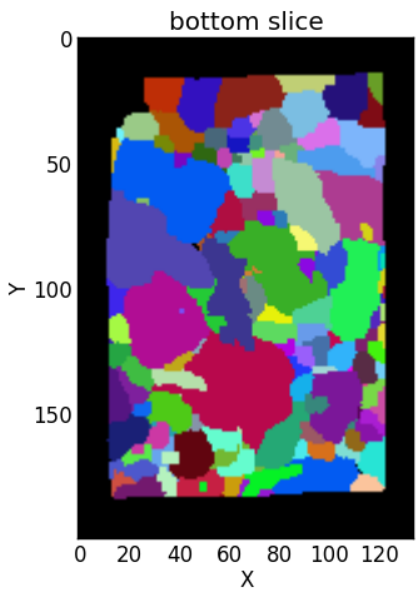
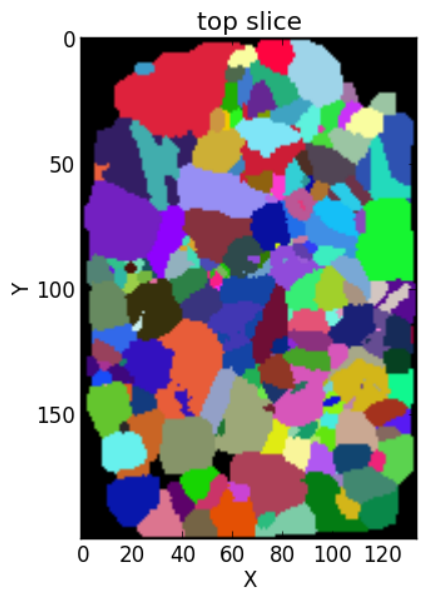
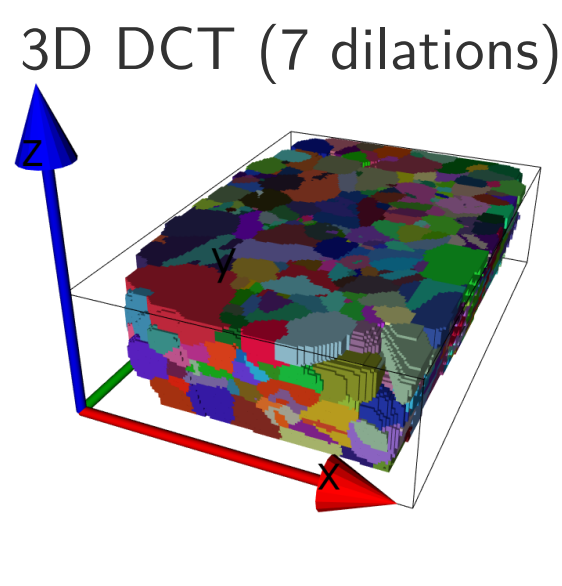
DCT Experiment for 3D grain mapping

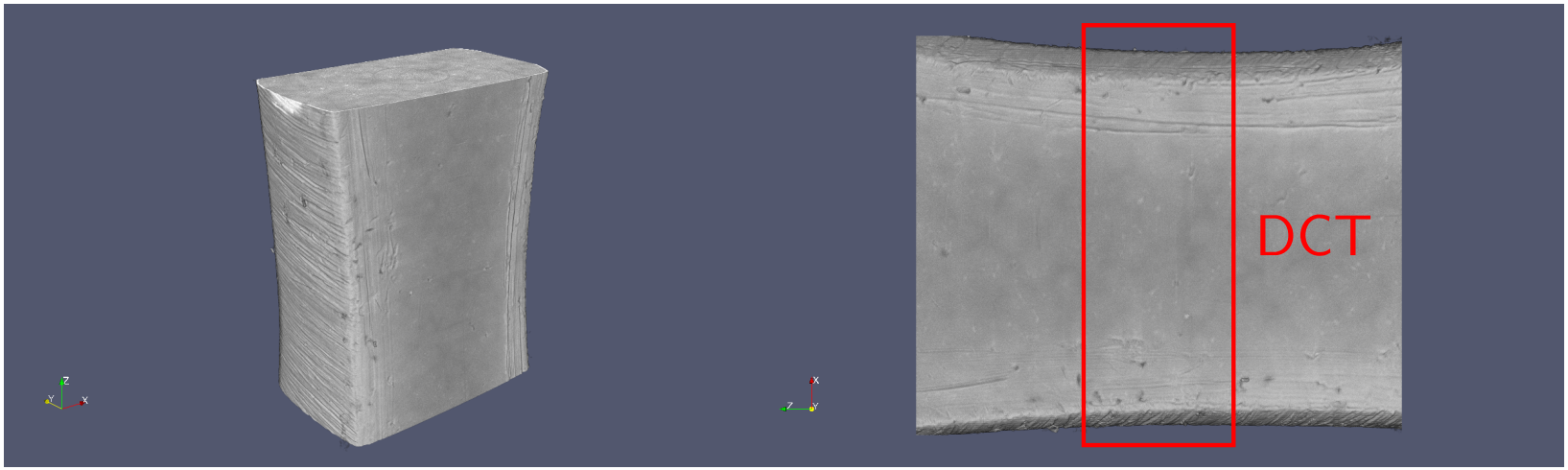
- Mar 2016 at ID11, ESRF (W. Ludwig)
- scan `g2_dct2_` undeformed state
- 37.22 keV, pixel size 1.5 µm
- 1013 grains
- cropped reconstructed volume size (pixels) : $253 \times 393 \times 104$



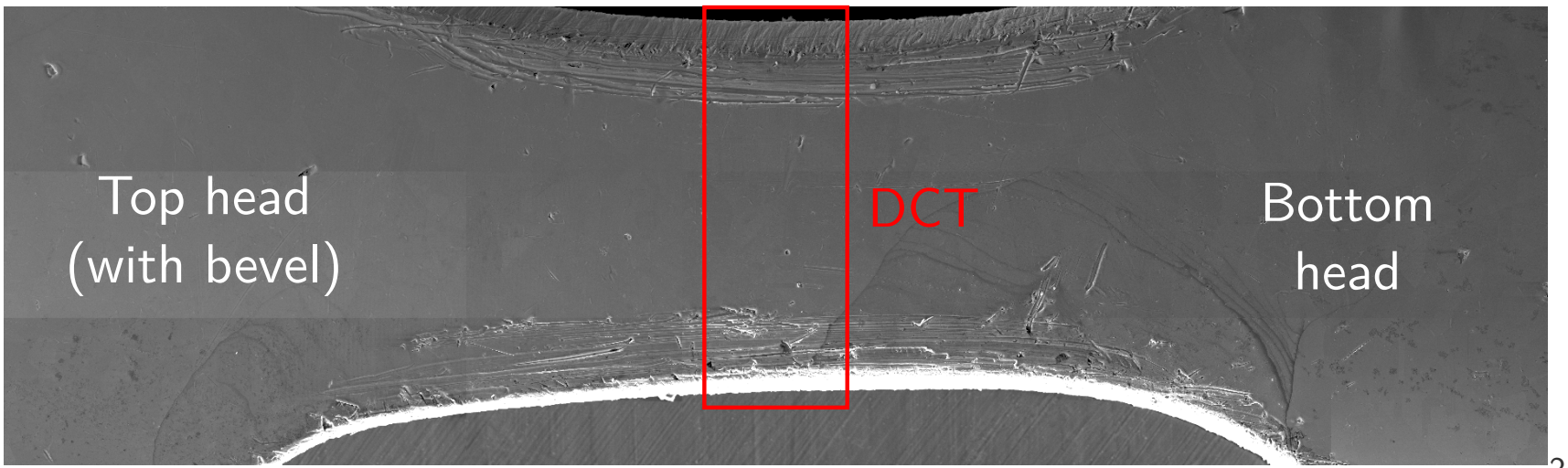




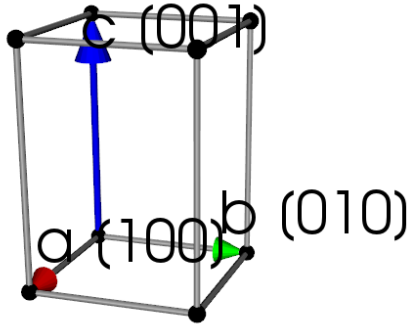
Registration with PCT volume

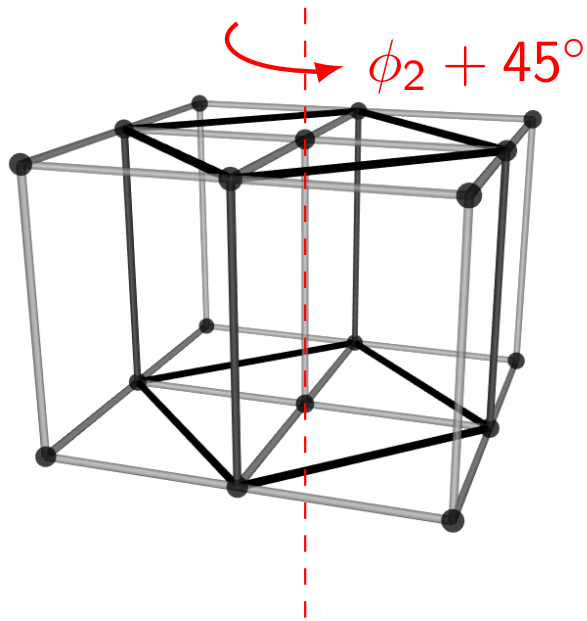


- Registration of absorption volume from the DCT acquisition onto a separate PCT volume.
- surface scratches in the PCT volume allo to locate the DCT region precisely ($\sim$ in the middle).



Note : from tetragonal to pseudo-fcc lattice



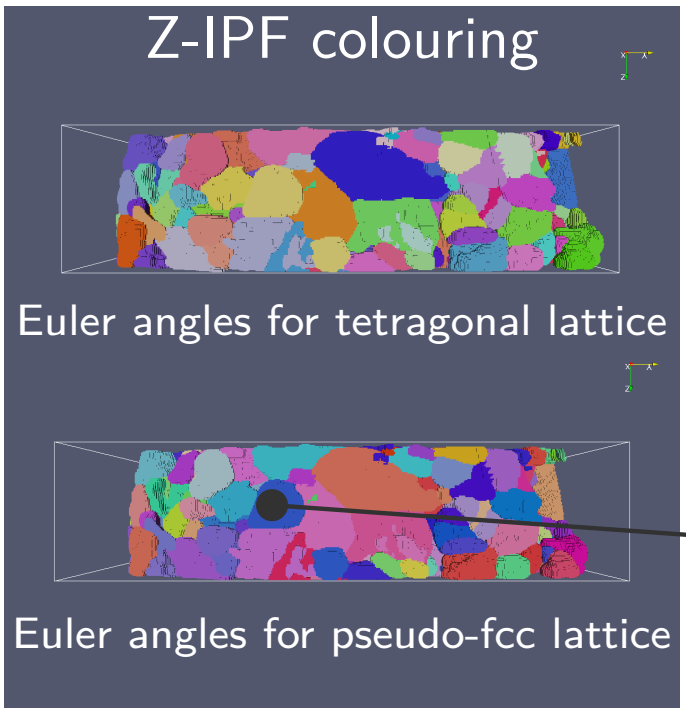


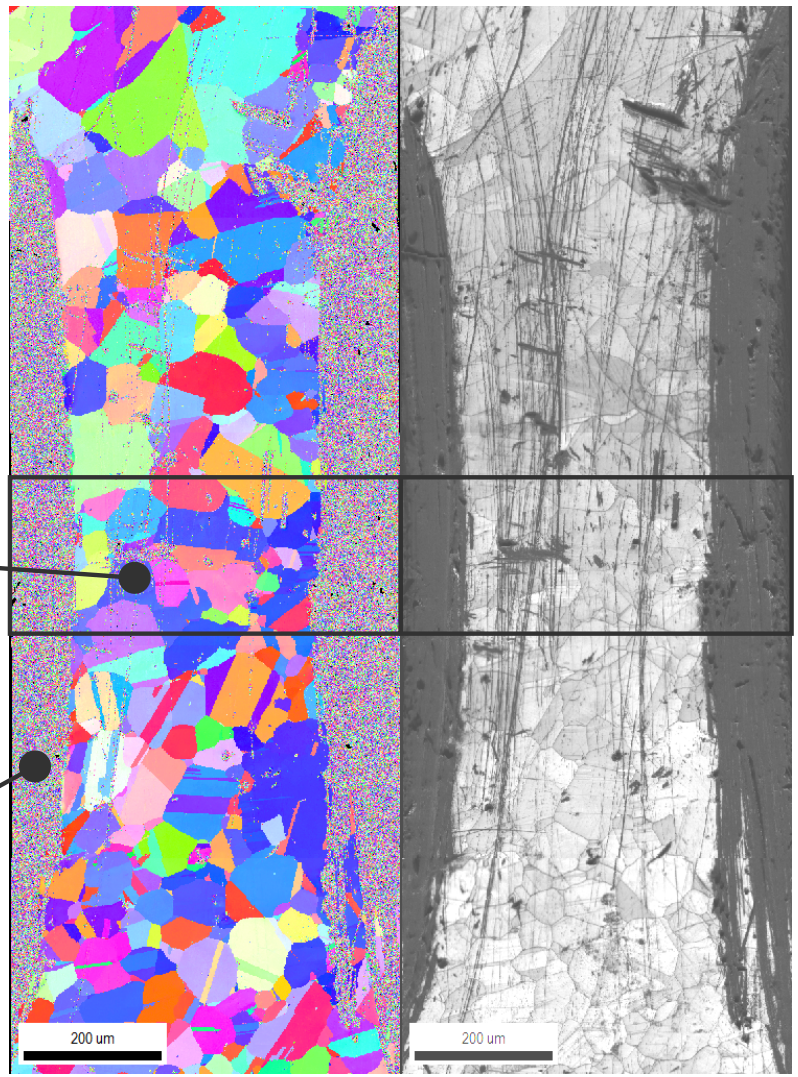
To account for the rotation around the c-axis, we need to add 45° to the ϕ_2 angle.
Ex for grain 9 (DCT numbering) :

- Euler angles for tetragonal lattice are $(\phi_1, \Phi, \phi_2) = (284.7, 59.4, 71.8)$
- Euler angles for pseudo-fcc lattice are $(\phi_1, \Phi, \phi_2) = (284.7, 59.4, 116.8)$

Identifying surface grains

Grain shape at the surface in the current DCT reconstruction is approximate, this made it hard to identify the region. Registration of the EBSD image quality allowed to find it.

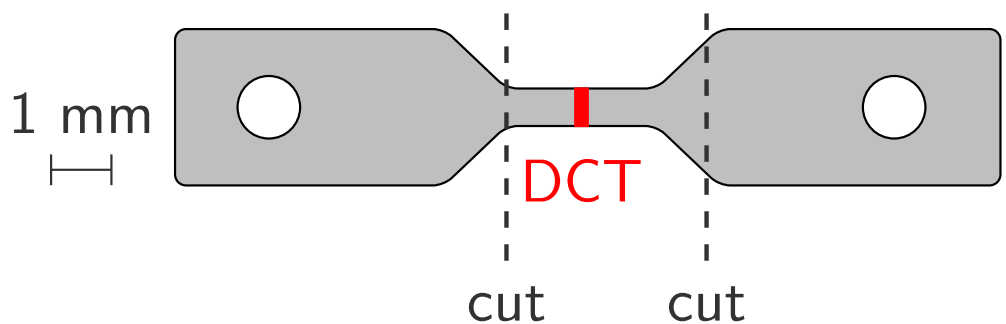




no indexing due to rounded shape

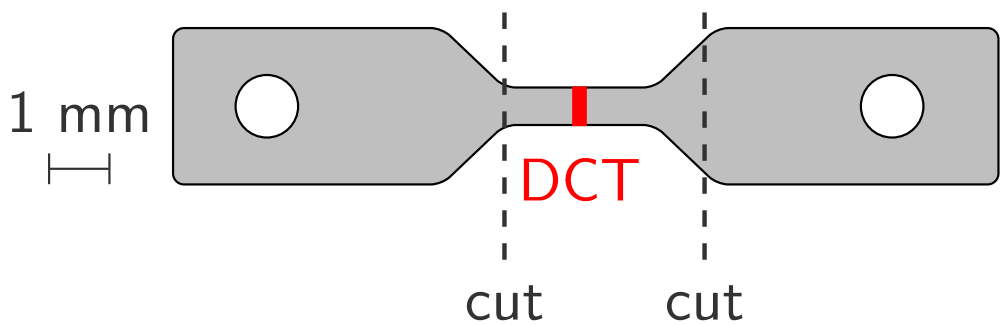
G2 pillar

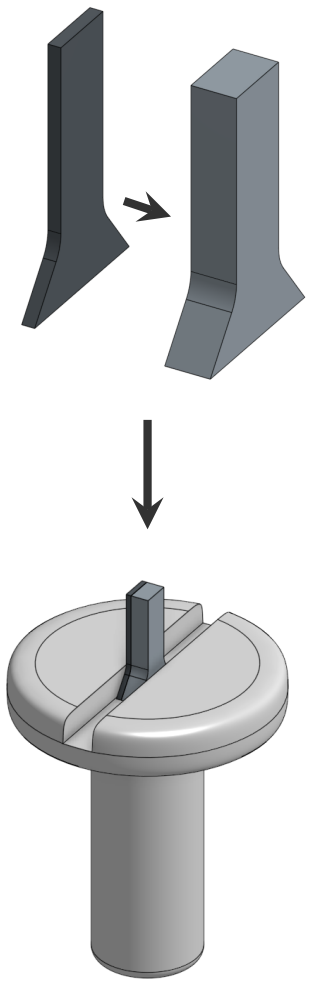
- γ-TiAl (Ti-48Al-2Cr-2Nb)
  G2 specimen characterized by DCT
- Saw cut of the gage length region
  to make a 560 µm $\times$ 350 µm pillar



G2 pillar

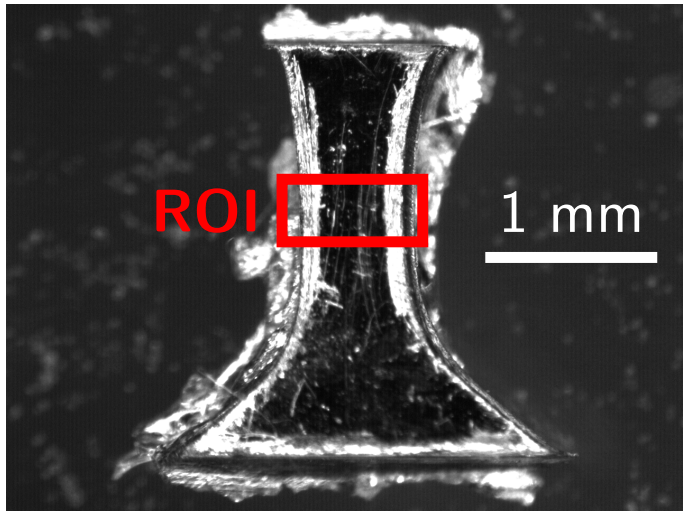
- γ-TiAl (Ti-48Al-2Cr-2Nb)
  G2 specimen characterized by DCT
- Saw cut of the gage length region
  to make a 560 µm × 350 µm pillar
- Back face protected by a 100 µm
  TiAl layer glued with silver paint
- Assembly mounted on a custom
  SEM stub





G2 pillar mounted on SEM stub

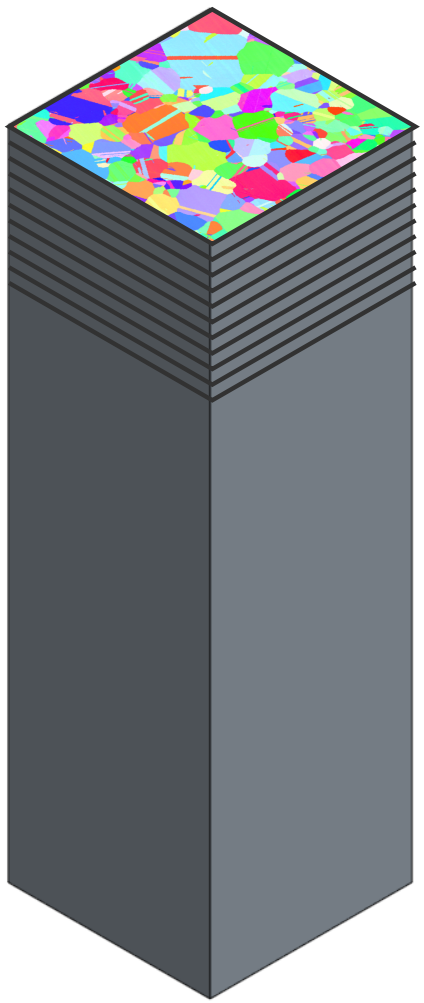
G2 pillar cut with TiAl layer glued on the back with silver paint







Tribeam acquisition geometry



Laser miling + data acquisition

Tribeam acquisition sequence :

1. Move sample up 1.5 µm
2. Laser milling (300 mW, 200 passes, 20 sec)
3. Acquire SEM images
4. FIB the surface (30 kV 65 nA, 10 minutes)
5. Get EBSD data (10 minutes)
6. Go back to step 1

30 minutes/slices, 230 slices have been acquired successfully in 4 days.

Final reconstructed volume : 600 µm × 400 µm × 345 µm with $\sim$ 2500 grains.

Tribeam data postprocessing



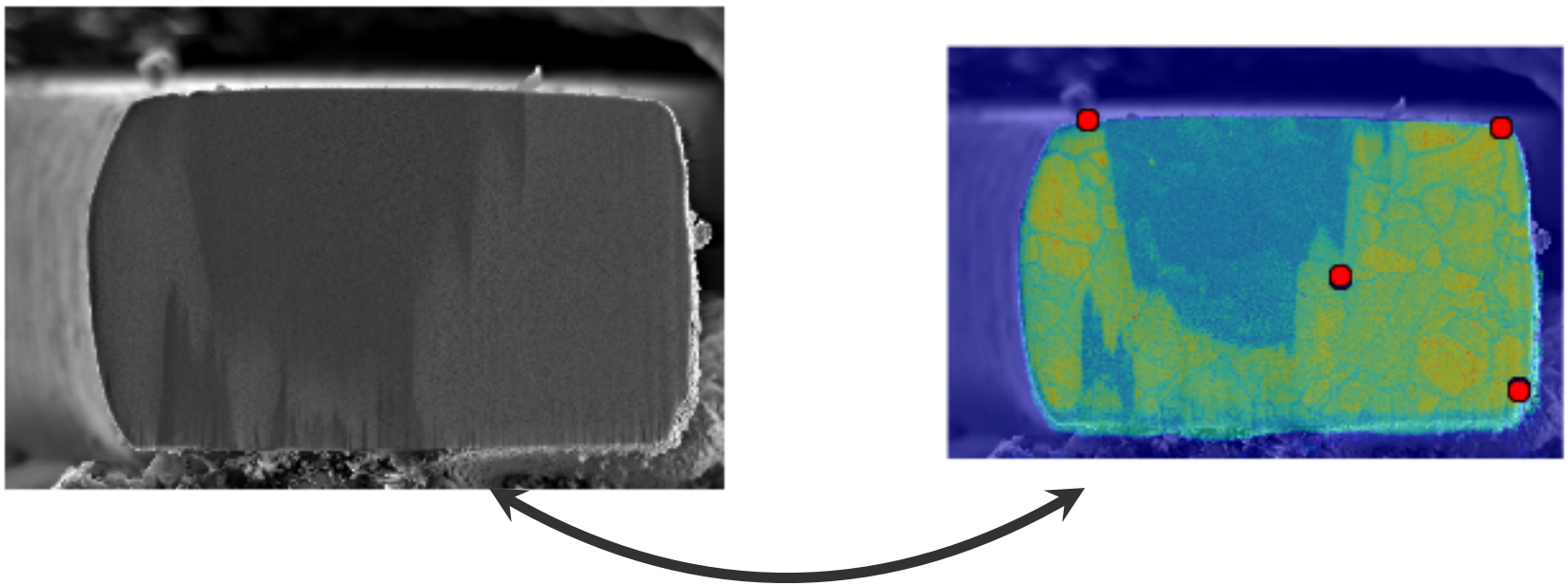
Done via Python and DREAM.3D
[Groeber and Jackson, 2014]
`http://dream3d.bluequartz.net`

1. Convert EBSD data into a stack and store it as `hdf5`
2. **Correct for in plane distortions**
3. Align the images **(IQ auto-correlation)**
4. Convert orientation data to quaternions
5. **Register with DCT/PCT 3D data sets**
6. **Use the PCT volume as a mask for the reconstruction**
7. Cleaning via neighbor orientation correlation
8. Segment grains using a misorientation criterion (2°) here
9. Remove grains smaller than 5 µm in diameter
10. Export data to paraview or pymicro for visualisation

In plane distortion corrections

Registration of the IQ signal onto the SE image taken after each cut $\rightarrow$ affine transform for the distortions during EBSD data collection.

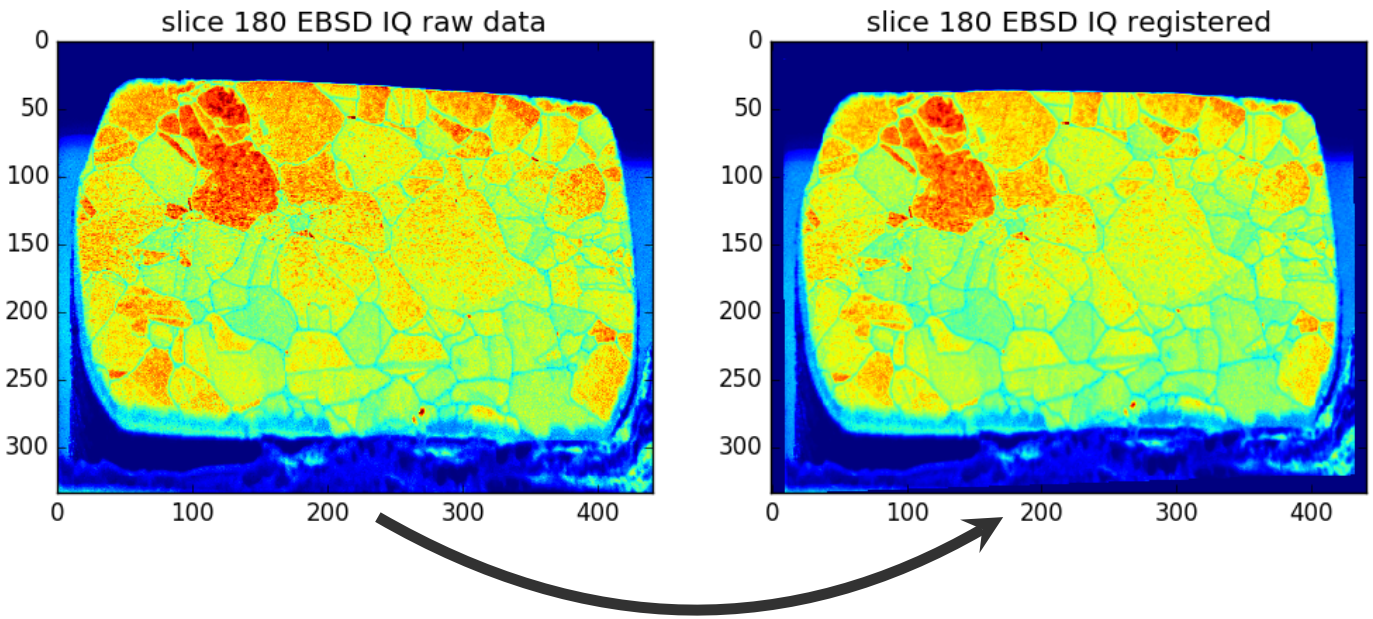
$$\underset{\sim}{\mathbf{A}} = \begin{pmatrix} M & A_{xy} \\ A_{yx} & M \end{pmatrix} \quad \text{here, } M = 1.04 \text{ and } A_{yx} = 0.011$$



In plane distortion corrections

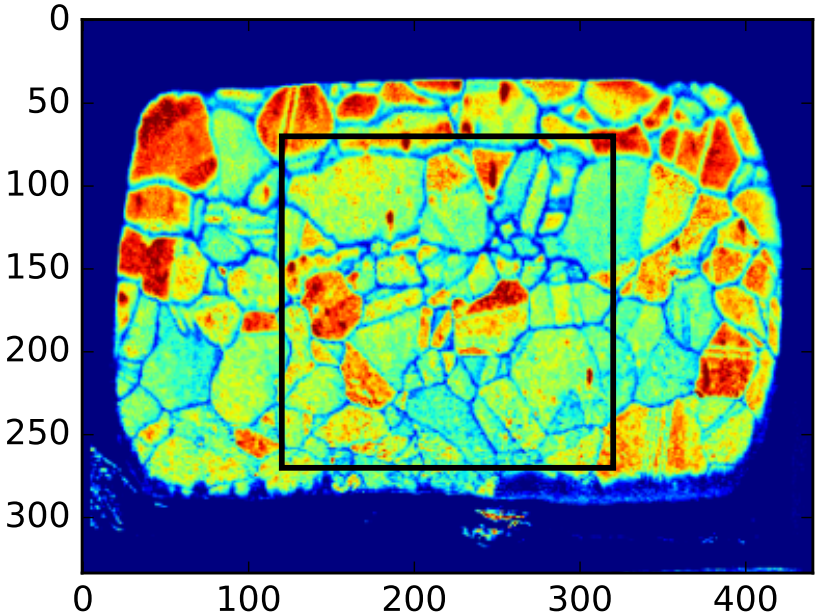
Registration of the IQ signal onto the SE image taken after each cut $\rightarrow$ affine transform for the distortions during EBSD data collection.

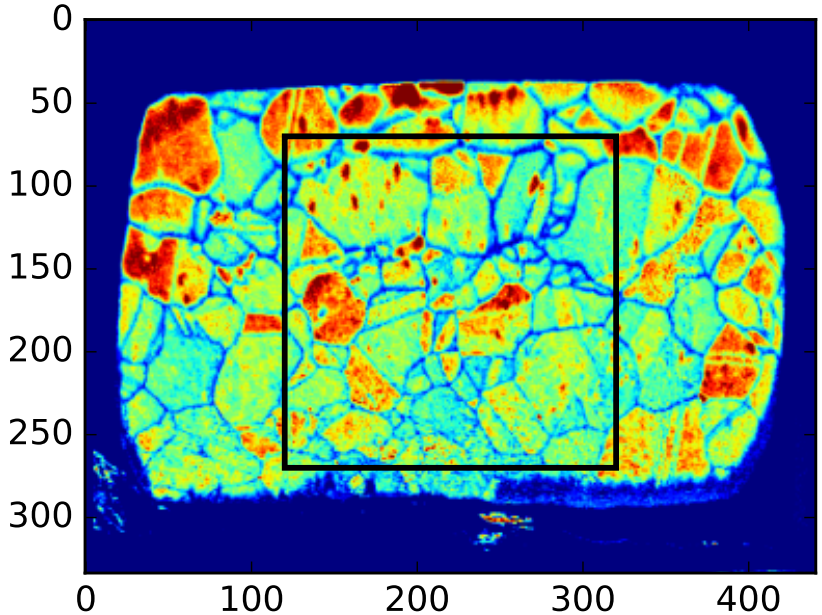
$$\underset{\sim}{\mathbf{A}} = \begin{pmatrix} M & A_{xy} \\ A_{yx} & M \end{pmatrix} \quad \text{here, } M = 1.04 \text{ and } A_{yx} = 0.011$$



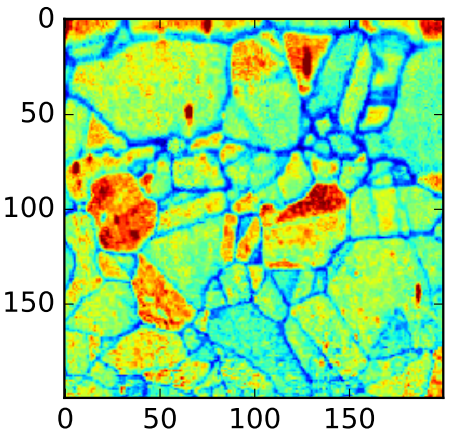
Stack alignment

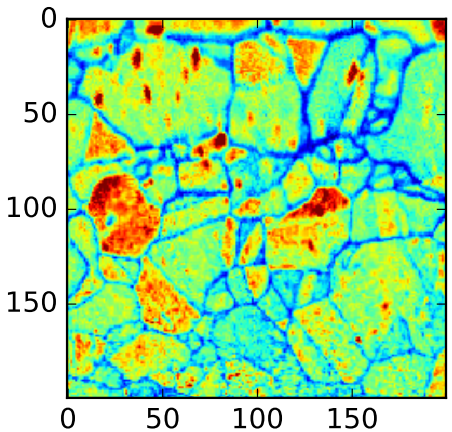
Leverage IQ contrast in the central region of the slices (for the example the shift has been increased 5 times) :

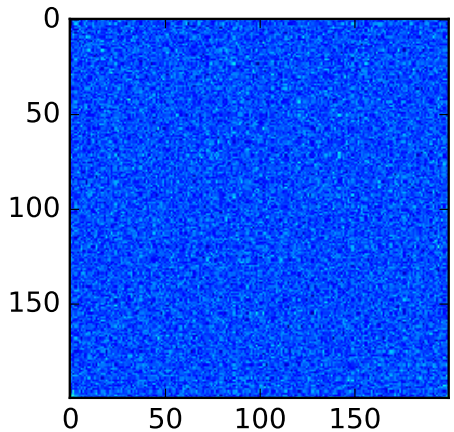




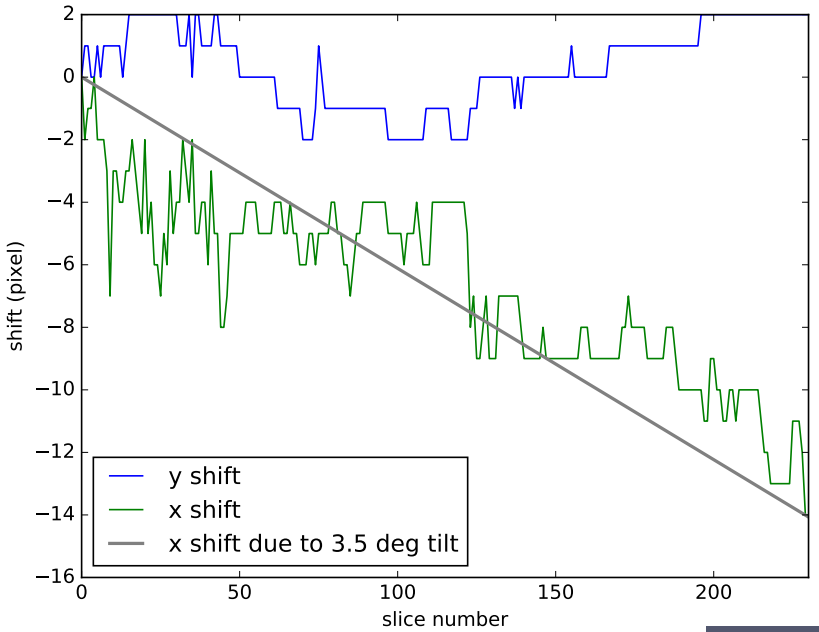
Compute cross correlation :





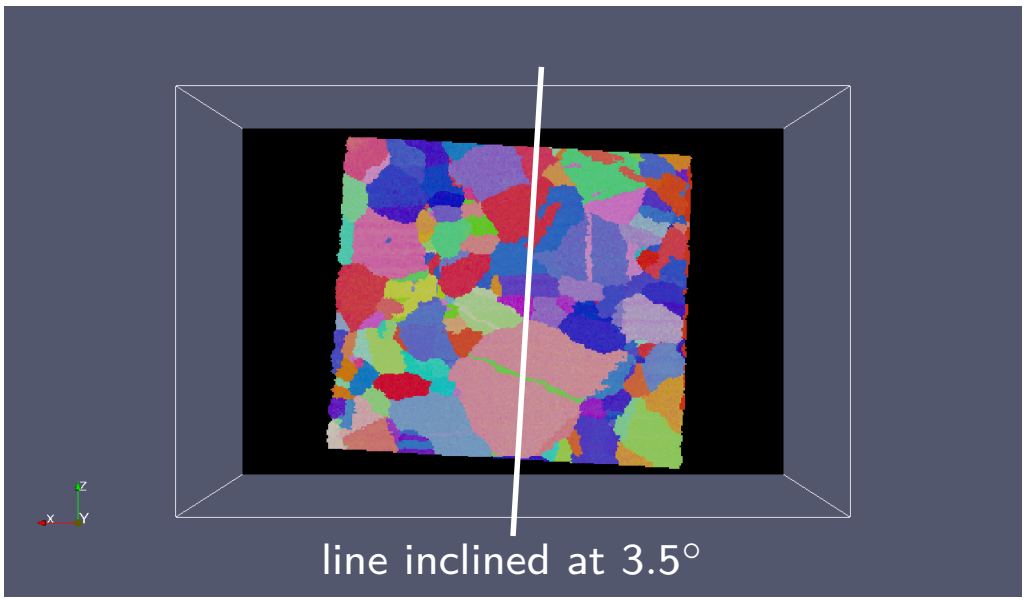


Stack aligned



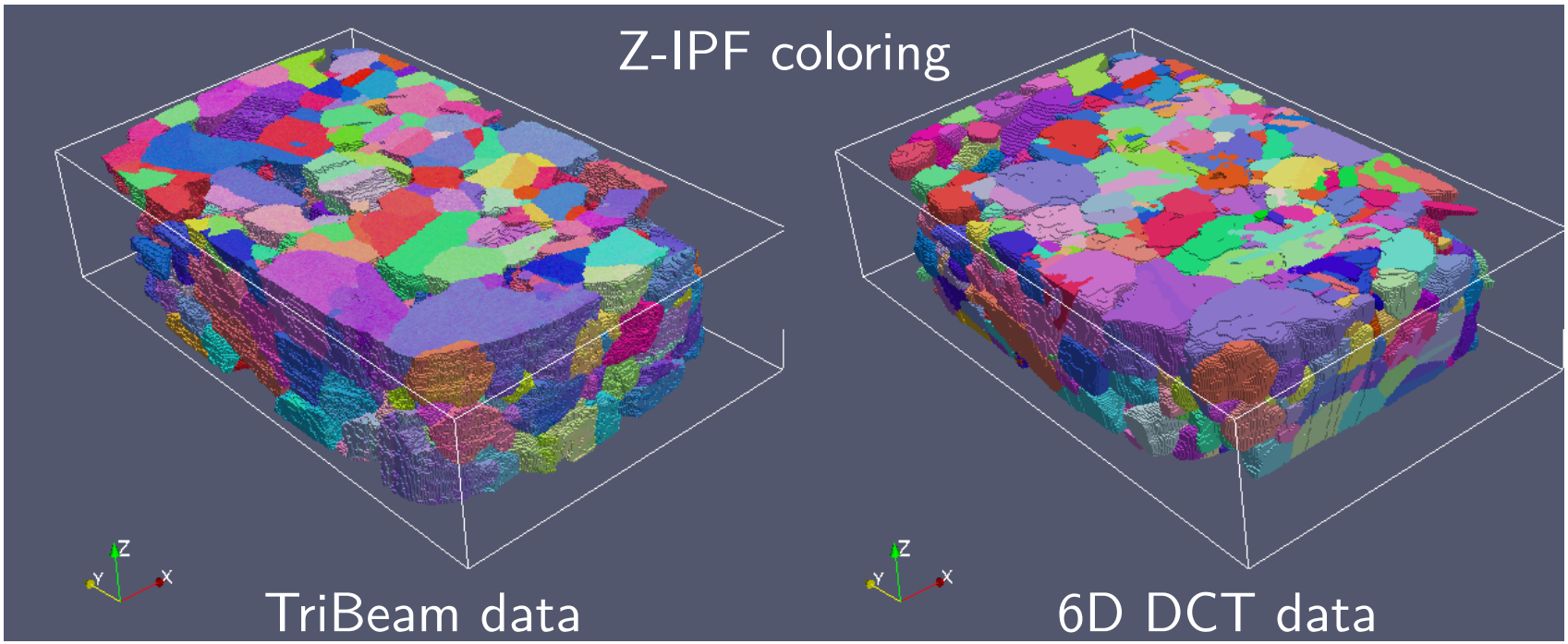
shifts primarily along the X axis (correspond to a sample tilt in the SEM chamber)

after applying the shifts, the reconstruction shows the tilt in the YZ plane, evaluated at 3.5°



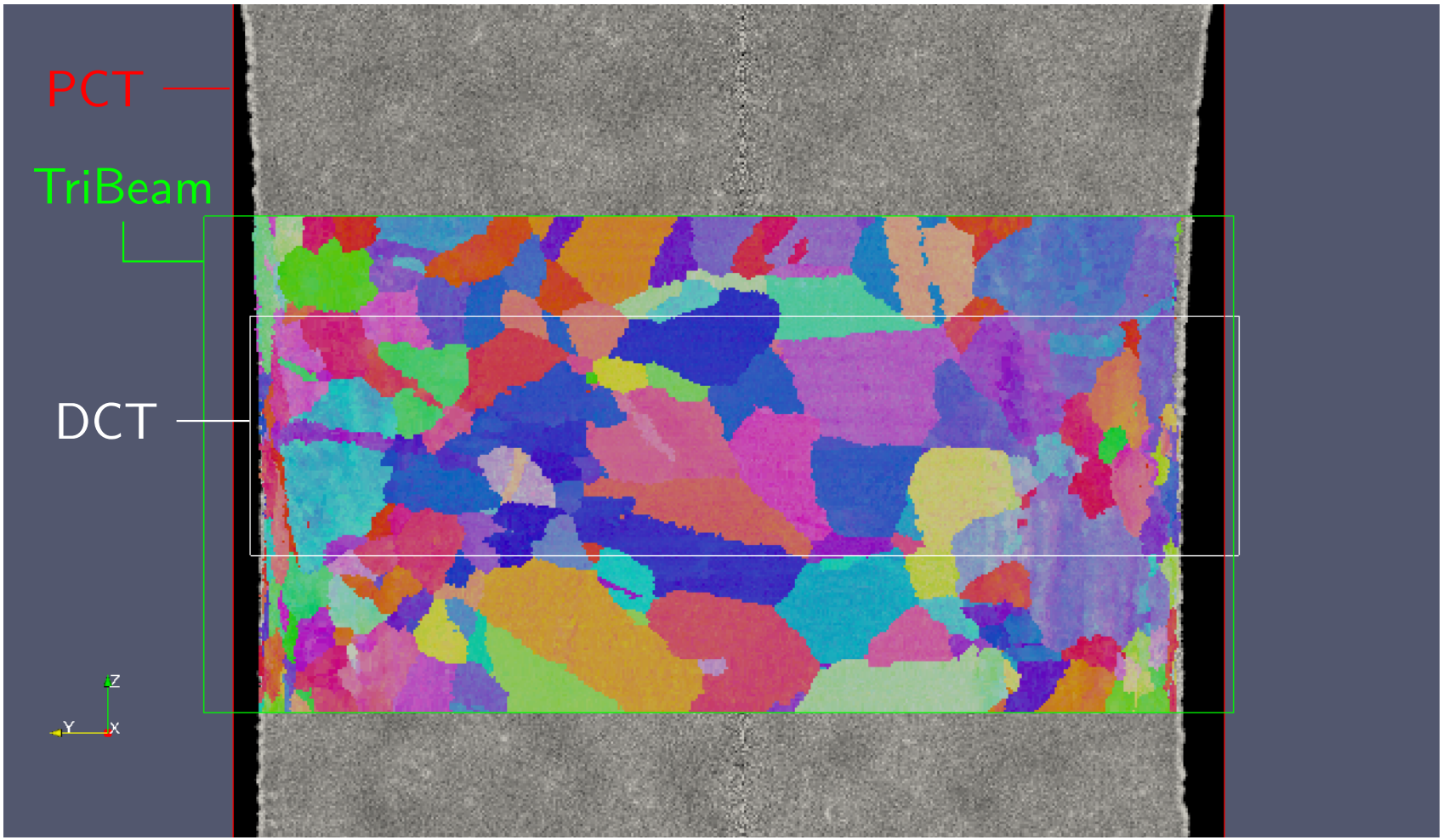
Registration of the TriBeam data set with the DCT

Precise registration between DCT and Tri-
Beam data sets using method from W. Lenthe [Lenthe et al., 2015] ;



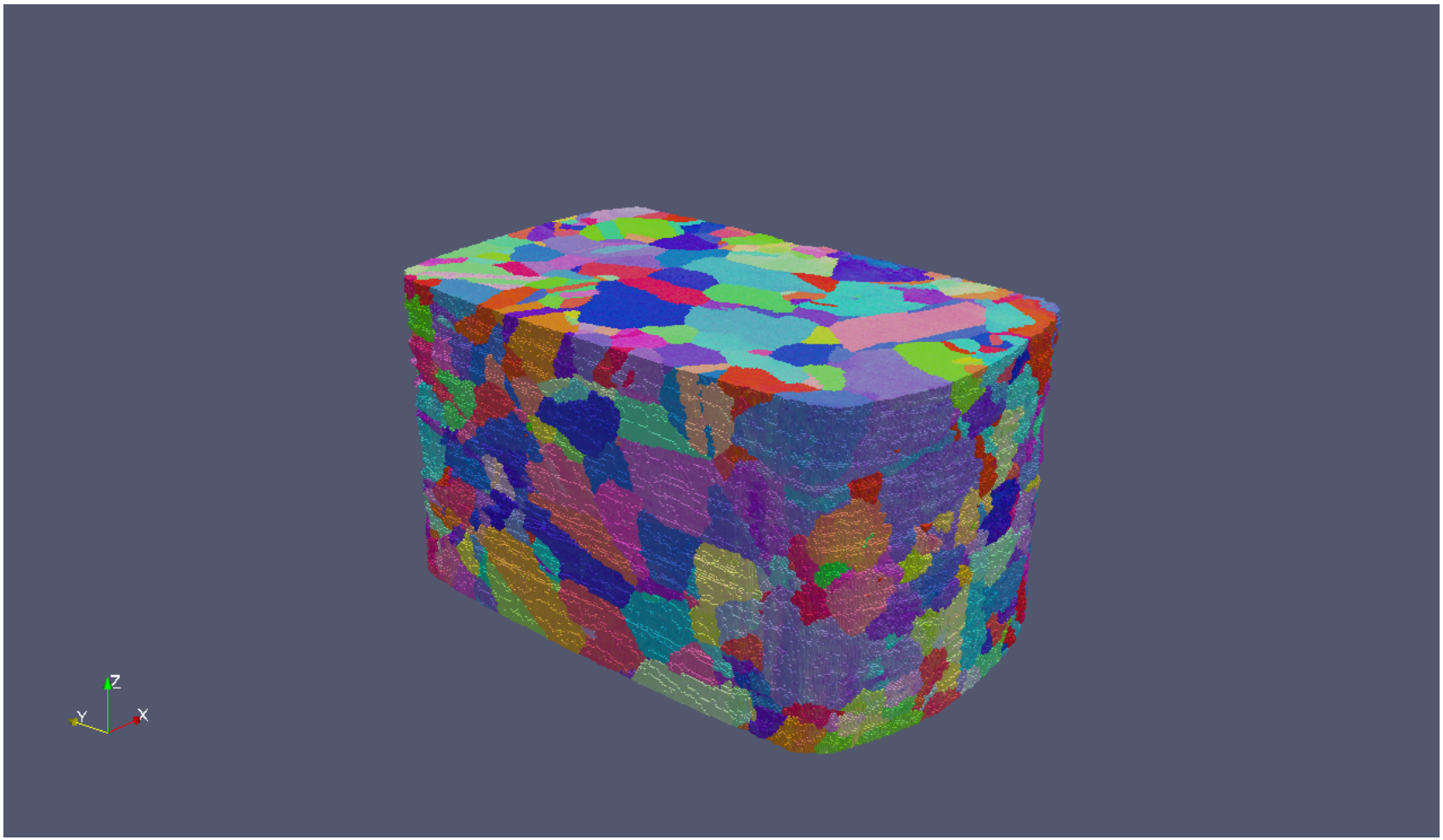
$\rightarrow$ Affine transform to bring the TriBeam data in the same coordinate system as the DCT data. Transformation is $\sim$ a rotation of 3.5° around Y.

TriBeam reconstruction merged with PCT/DCT data sets

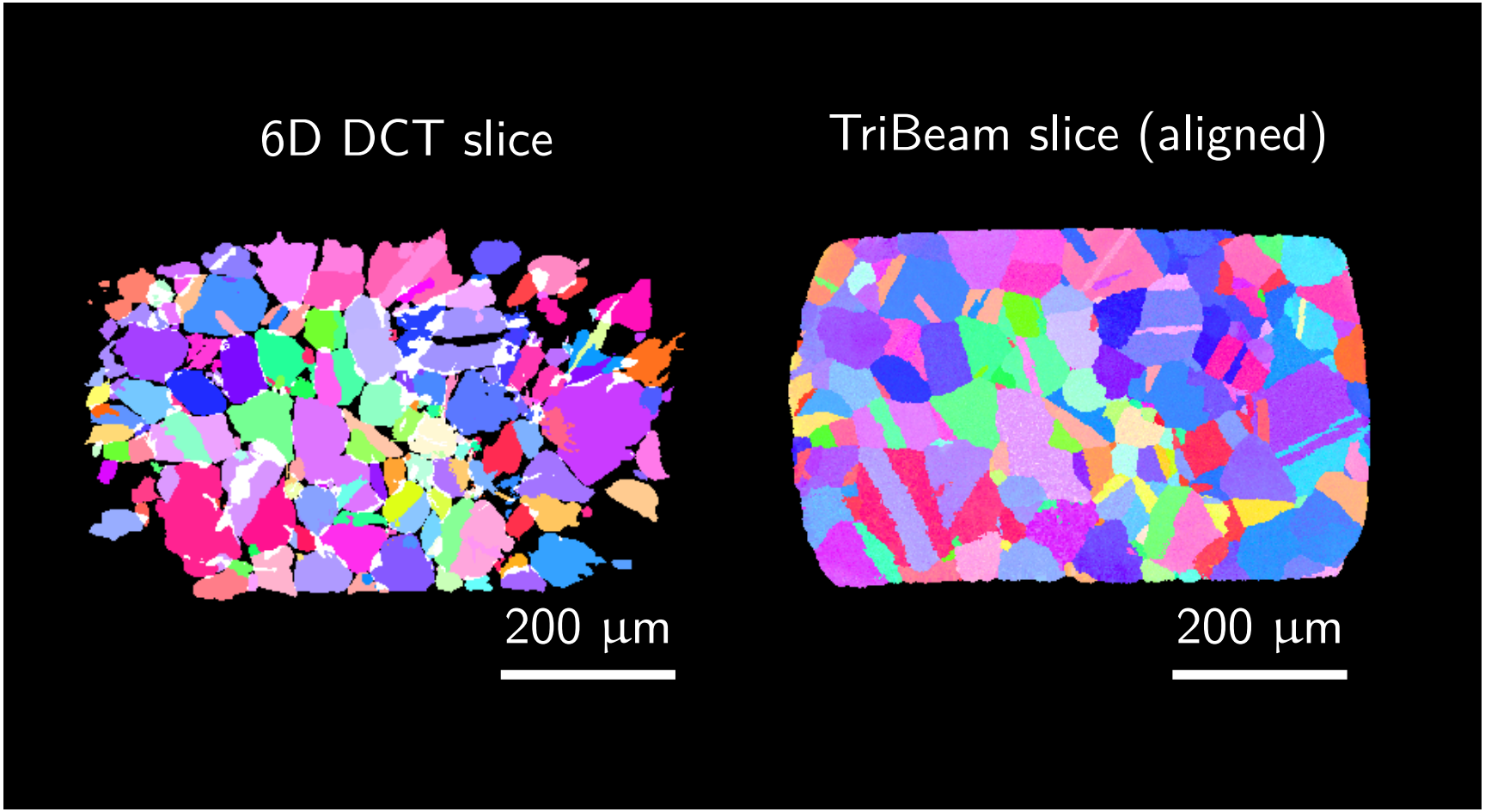


All 3 data sets merged into a single data container

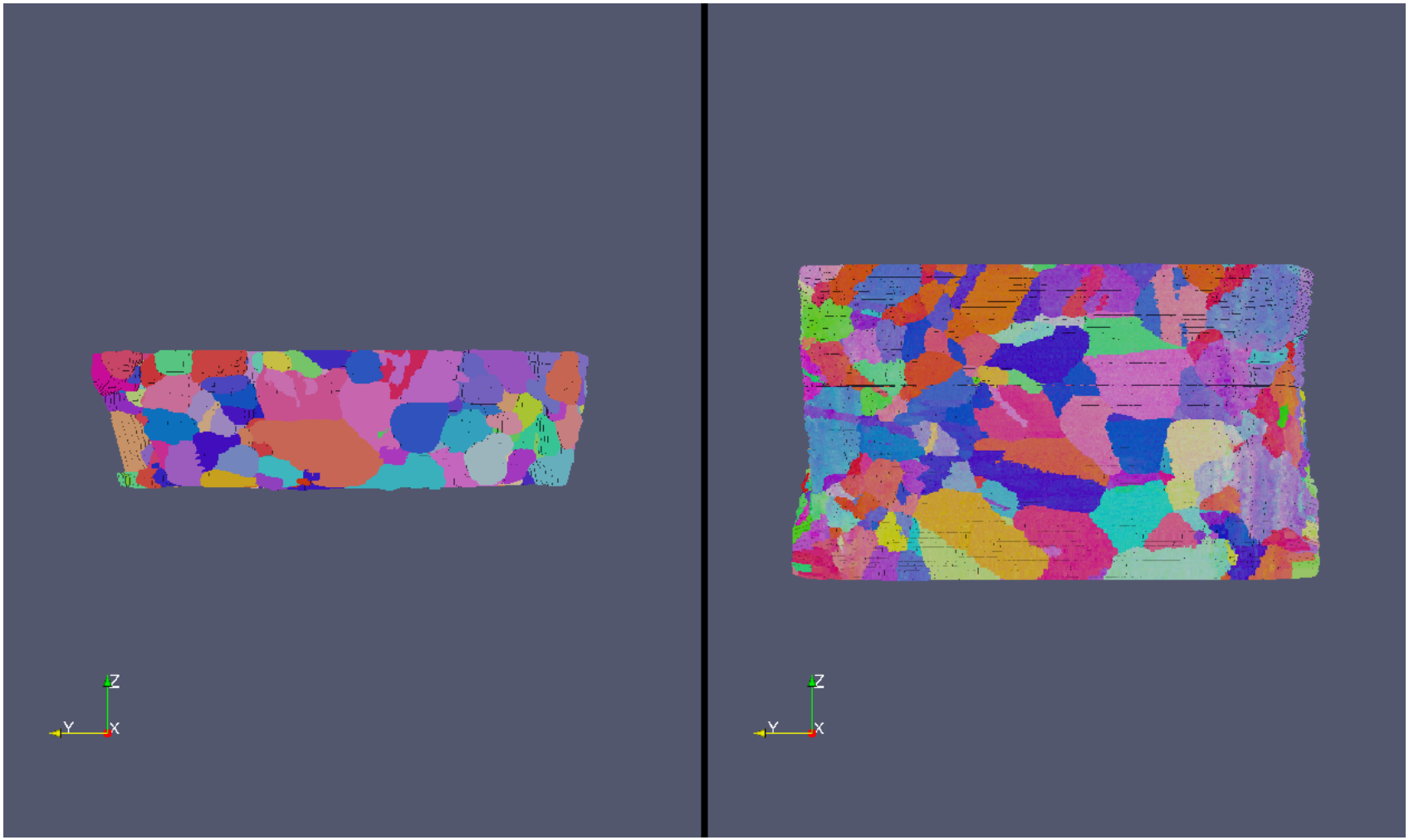
TriBeam reconstruction merged with PCT/DCT data sets



Comparison DCT/TRIBEAM



Overall comparison DCT/TRIBEAM



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4 Summary and outlook

Summary

3D grain mapping has become a reality for material scientists and is a great tool to study the mechanics of polycrystalline materials.

- **Serial sectioning** methods are imroving dramatically, and now allow access to thousands of grains strong microstructures with unprecedented precision.
- Among these, the **Tribeam method** can acess very large volumes of material ($\sim mm^3$).
- **Non destructive methods** also exist (DCT or HEDM) and are very complementary but have trouble with complex microstructures (twins, lamellaes, multiphase, intricated microstructures)
- Full-field simulations (FE but also FFT) with experimental microstructure is a key to **compare experiments and simulation** on a grain to grain basis.

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