CENTRE DES MATERIAUX
P.M. FOURT

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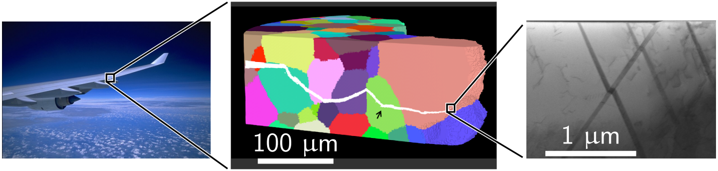
Reconstruction 3D de microstructures polycristallines, approches destructive par coupes successives vs non destructive par tomographie X

Henry Proudhon
MINES ParisTech, PSL research university, Centre des Matériaux, France

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 = 4D experiments : characterise the 3D microstructure **and** observe damage growing in situ under mechanical loading.

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

3D characterization of structural material microstructures

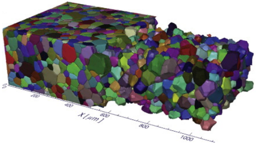
Difficult but important measurements

Coupled to *in situ* experiments and computational mechanics calculations, it is a promising route to study, develop and validate our material models and material constitutive behaviors.

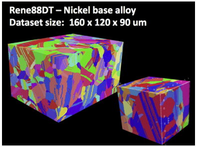
How to obtain grain geometries and orientations of a representative volume ($\sim 1\ \mathrm{mm}^3$) ?

- EBSD / Serial sectioning
- 3DXRD, HEDM or Diffraction Contrast Tomography

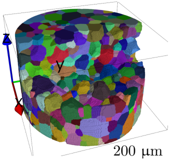
destructive — non-destructive



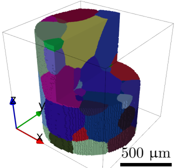
(a) β-Ti reconstruction by EBSD serial sectioning



(b) Tribeam reconstruction of a Rene88 sample



(c) DCT reconstruction of a pure Ti sample



(d) labDCT reconstruction of a test AlLi sample

[Rowenhorst et al., 2010, Echlin et al., 2015, Proudhon et al., 2016]

Contents

Thanks to

Collaborators :

- Nicolas Guéninchault (former Ph. D. student, now with Xnovotech)
- S. Forest (CDM), W. Ludwig (ESRF/INSA Lyon)
- UCSB Pollock group : M. Echlin, W. Lenthe, T. Pollock

Funding :

MINES ParisTech

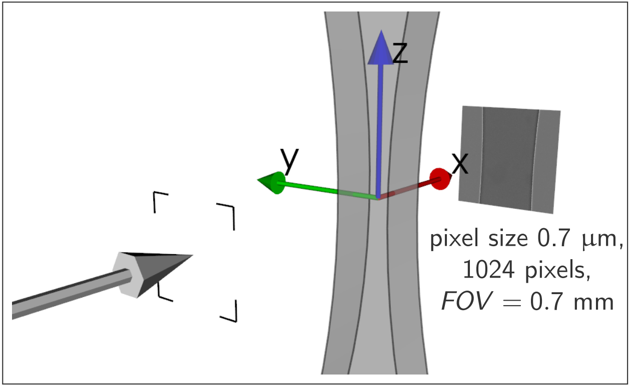
cnrs dépasser les frontières

ANR - PROJET FINANCÉ PAR L'ANR - PROJECT FUNDED BY THE ANR

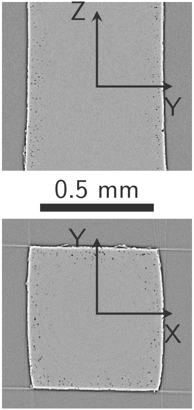
Contents

Tomography of monophase materials

If electrons cannot go through a significant amount of material, hard X-rays can. At 40 keV, transmission through 1 mm of materials is **66% in Al** and **35% in Ti**.

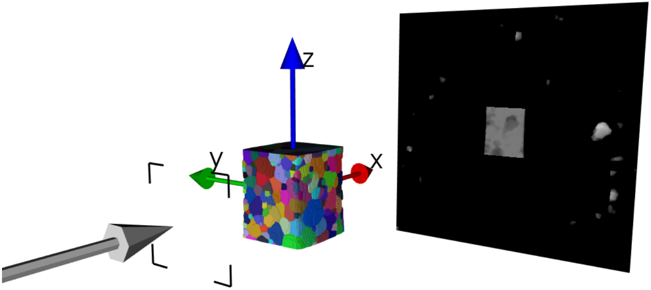


pixel size 0.7 μm,
1024 pixels,
FOV = 0.7 mm



Absorption/Phase contrast do not depends on crystallographic orientation → **no contrast between grains of the same phase**.

Diffraction contrast tomography

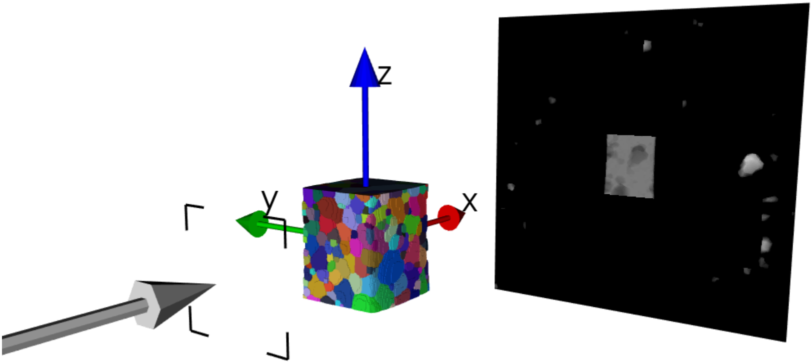


As summarized in [Poulsen, 2004], according to the Bragg's law, one reflection $\underline{\boldsymbol{G}}$ of a crystallographic grain will be in diffracting condition if :

$$\frac{2\sin^2(\theta)}{\lambda} = -\left[{}^{\cdot}_{\sim}\underset{\sim}{\mathbf{g}}^{-1}\underset{\sim}{\mathbf{B}}\underline{\boldsymbol{G}}\right]_1$$

here θ is the (known) Bragg angle defined for the given reflection of the crystal lattice and the considered wave length λ.

Animation of simulated DCT on AlLi sample



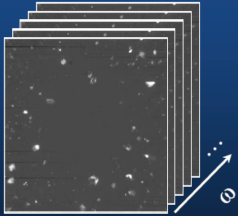
⏮ < ◁ ▷ > ⏭ − ⧓ +

ω step $= 0.5^\circ$, $\{111\}$, $\{002\}$, $\{022\}$ and $\{113\}$ families included (FCC crystal structure).

principal steps of DCT data analysis

(~ 1 day / scan)

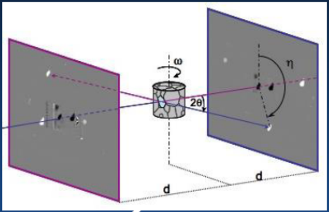
Acquisition



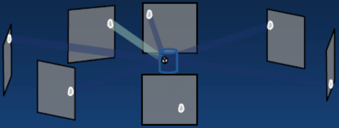
Raw data (7200 images)
~ 100.000 spots

Segmentation

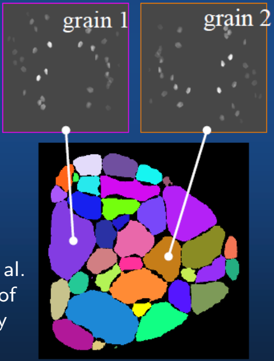
Pair Matching



Indexing



Reconstruction



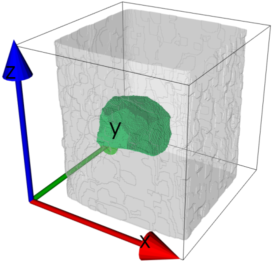
ART reconstruction
1000 grains

Developed at ESRF by W. Ludwig et al.
→ Non destructive characterization of 3D grain microstructures (plastically undeformed, monophase materials)
[Ludwig et al., 2009, Reischig et al., 2013]

Real example : AlLi specimen, grain 4

- Position : $[-0.0882, 0.0276, -0.0793]$ mm
- Orientation : $\underline{\boldsymbol{r}} = [0.0499, -0.3048, 0.1040]$
- The grain orientation matrix is :

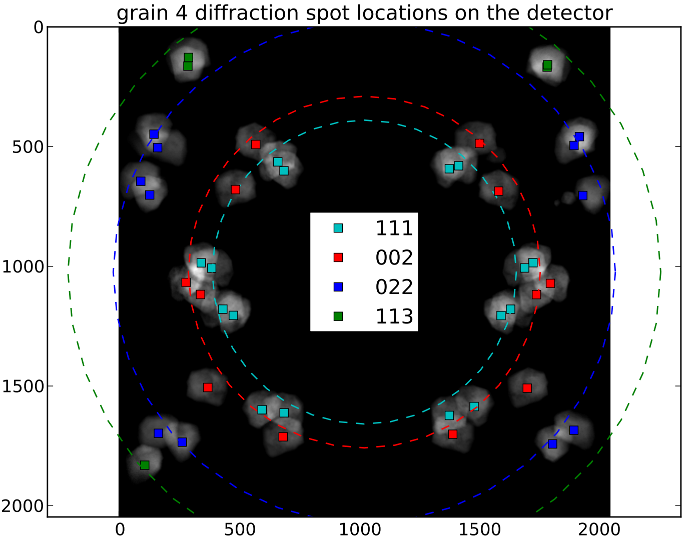
$$g^{-1} = \begin{pmatrix} 0.812 & -0.215 & -0.541 \\ 0.160 & 0.975 & -0.147 \\ 0.560 & 0.033 & 0.827 \end{pmatrix}$$



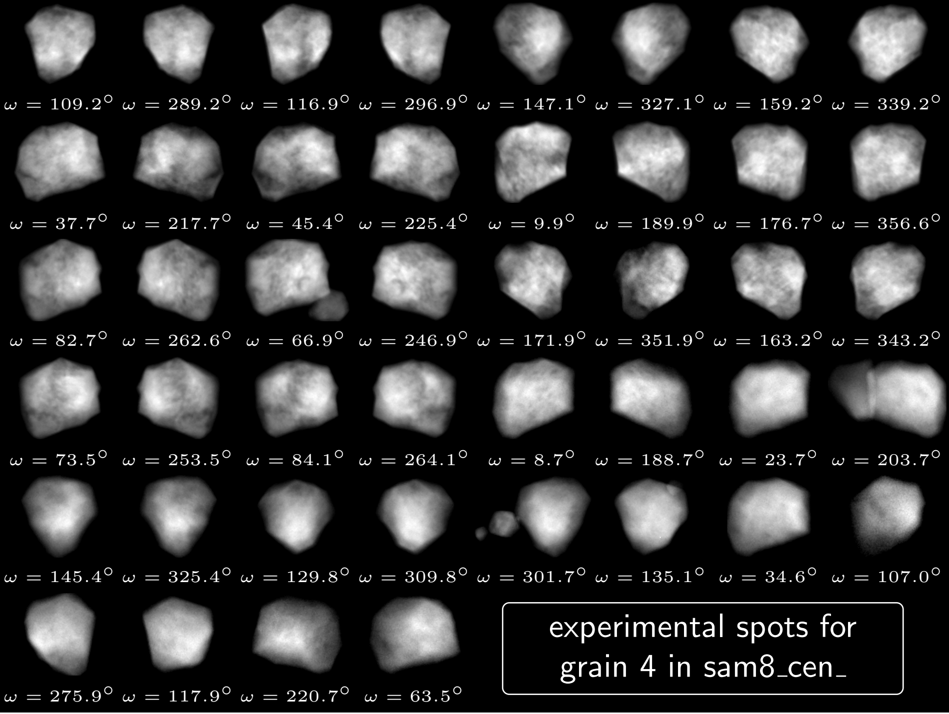
First diffraction angles predicted :

hkl	ω_1 (°)	ω_2 (°)
111	176.5	9.9
-111	109.2	296.9
1-11	327.1	159.3
11-1	217.7	45.4
002	66.9	262.6
020	163.2	351.9
200	253.5	84.1
022	129.7	325.4
0-22	8.7	203.7
220	200.9	34.6
-220	121.7	315.1
202	232.9	121.6
-202	70.9	263.5

Experimental spot positions for grain 4

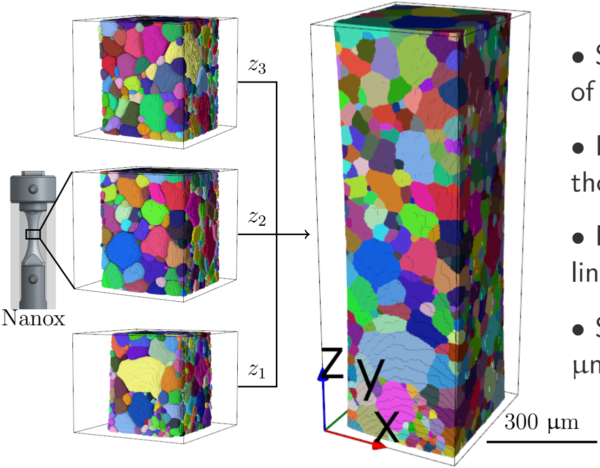


Experimental positions



experimental spots for
grain 4 in sam8_cen_

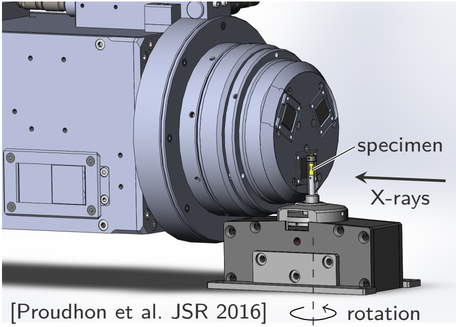
Final experimental grain map with AlLi sample



- Study the mechanics of polycrystals
- Data sets with a few thousand grains
- Links with FE modelling (crystal plasticity)
- Spatial resolution ∼ µm

Beyond 3D : 4D automated in situ mechanical testing

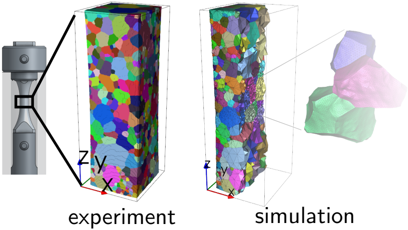
Nanox : unique 4D capabilities :

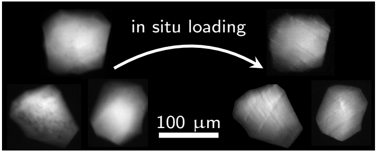


[Proudhon et al. JSR 2016]

4D experiments are becoming mature and provide a **new way to elucidate microstructure/properties relationships**.

Limit : only a few specimens can be tested due to limited beam time.



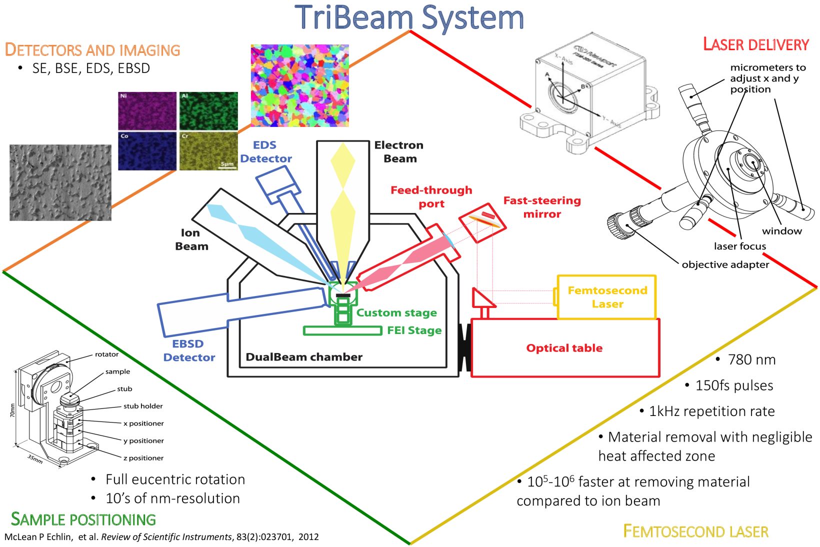


Initial state Deformed state

- Fully automated multi-day testing at the synchrotron
- First observation of plasticity in the bulk of a polycrystalline material

Contents

Tribeam tomography at UCSB



DETECTORS AND IMAGING

- SE, BSE, EDS, EBSD

LASER DELIVERY

- 780 nm
- 150fs pulses
- 1kHz repetition rate
- Material removal with negligible heat affected zone
- 10^5-10^6 faster at removing material compared to ion beam

FEMTOSECOND LASER

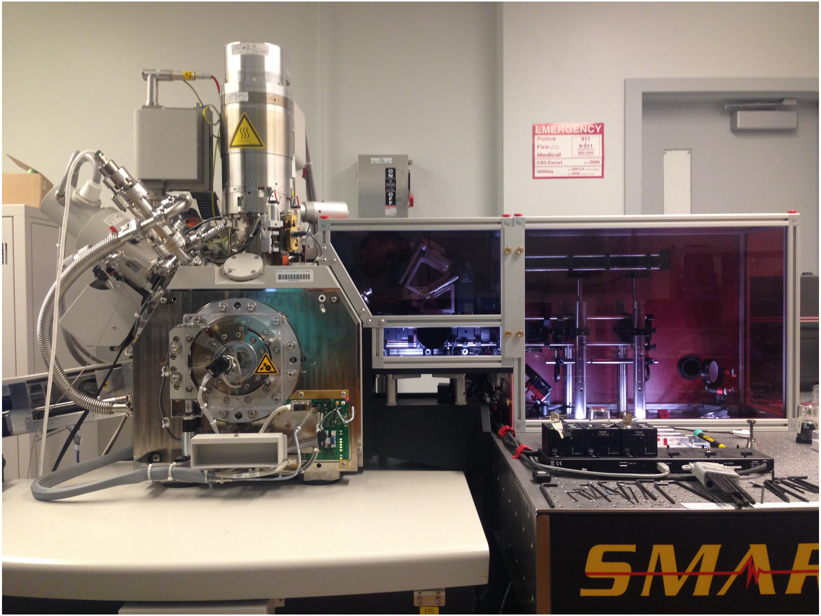
- Full eucentric rotation
- 10's of nm-resolution

SAMPLE POSITIONING

McLean P Echlin, et al. *Review of Scientific Instruments*, 83(2):023701, 2012

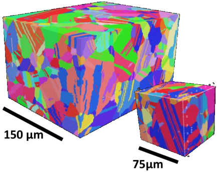
[Echlin et al., 2012, Echlin et al., 2015]

The tribeam system at UCSB

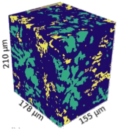


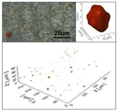
Tribeam tomography of structural materials

Nickel Superalloy



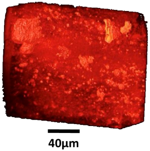
NiTiSn





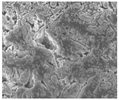
High Strength Steels

Geological Samples

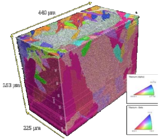


Strontium Titanate

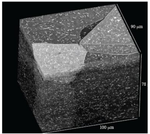
Cu-Polymer Composite

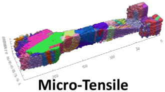


Ti-6-4 Beta Anneal

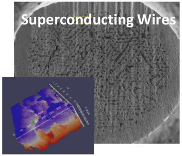


Laves Phases in Fe-based Alloy

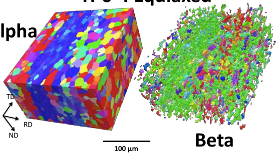




Micro-Tensile Samples

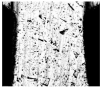


Ti-6-4 Equiaxed



Alpha

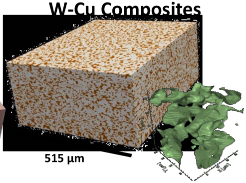
Beta



Ti_2AlC Ceramic

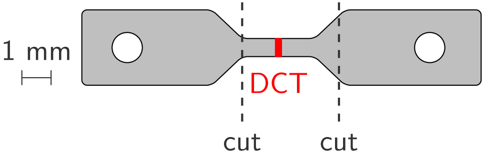
Carbon Fiber Epoxy Composite

W-Cu Composites



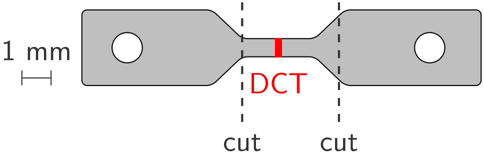
Tribeam experiment on a TiAl alloy

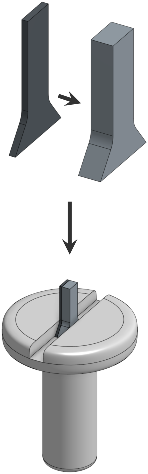
- small γ-TiAl (Ti-48Al-2Cr-2Nb) tension specimen characterized by DCT
- Saw cut of the gage length region to make a 600 µm × 400 µm pillar



Tribeam experiment on a TiAl alloy

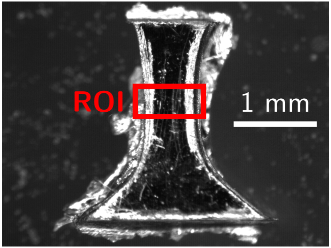
- small γ-TiAl (Ti-48Al-2Cr-2Nb) tension specimen characterized by DCT
- Saw cut of the gage length region to make a 600 µm × 400 µm pillar
- Back face protected by a 100 µm TiAl layer glued with silver paint
- Assembly mounted on a custom SEM stub

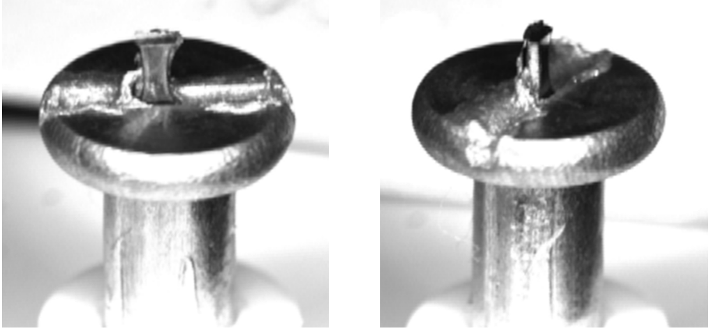




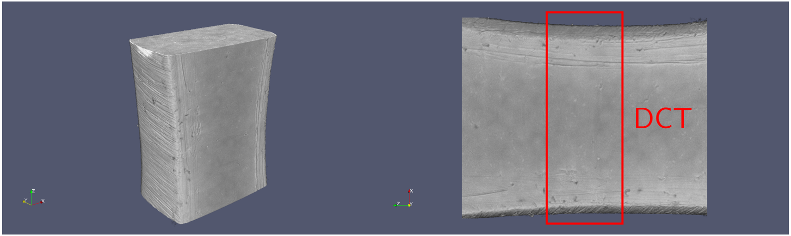
G2 pillar mounted on SEM stub

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

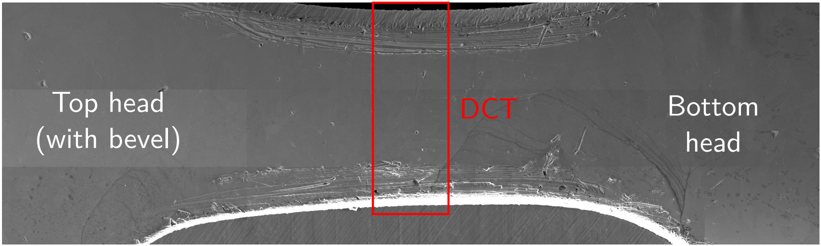




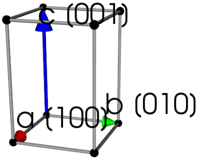
Registration with PCT volume

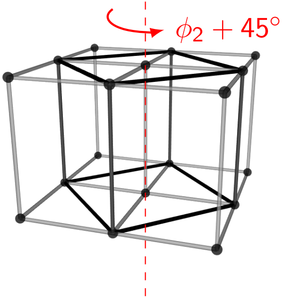


- Registration of absorption volume from the DCT acquisition onto a separate PCT volume.
- surface scratches in the PCT volume allo to locate the DCT region precisely ($\sim$ in the middle).



Note : from tetragonal to pseudo-fcc lattice



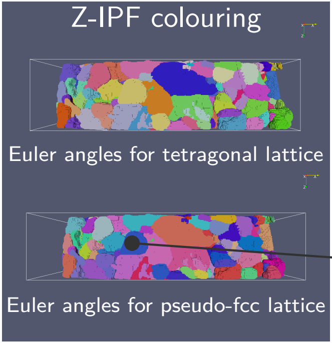


To account for the rotation around the c-axis, we need to add 45° to the ϕ_2 angle.
Ex for grain 9 (DCT numbering) :

- 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

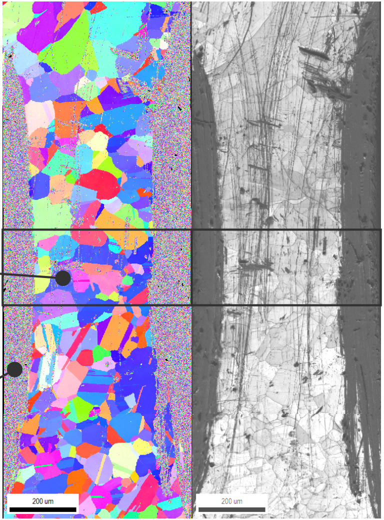
Grain shape at the surface in the current DCT reconstruction is approximate, this made it hard to identify the region. Registration of the EBSD image quality allowed to find it.



Z-IPF colouring

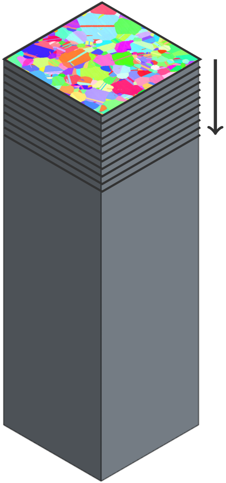
Euler angles for tetragonal lattice

Euler angles for pseudo-fcc lattice



no indexing due to rounded shape

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

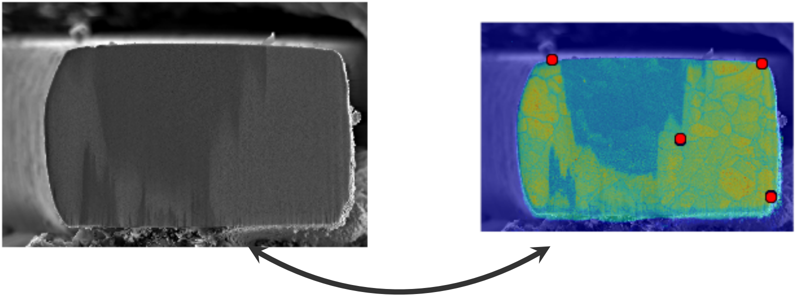
Done via Python and DREAM.3D
[Groeber and Jackson, 2014]
`http://dream3d.bluequartz.net`

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 → affine transform for the distortions during EBSD data collection.

$$\underset{\sim}{\mathbf{A}}^{-1} = \begin{pmatrix} M & 0. \\ A_{yx} & M \end{pmatrix} \quad \text{here, } M = 1.04 \text{ and } A_{yx} = 0.011$$

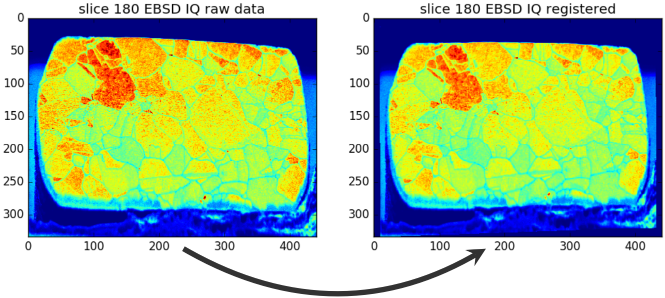


Compute affine transform

In plane distortion corrections

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

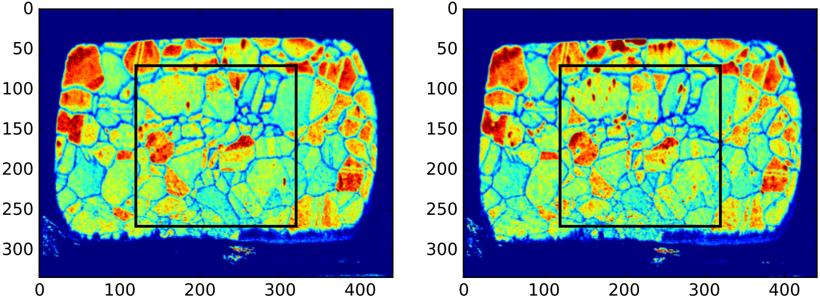
$$\underset{\sim}{\mathbf{A}}^{-1} = \begin{pmatrix} M & 0. \\ A_{yx} & M \end{pmatrix} \quad \text{here, } M = 1.04 \text{ and } A_{yx} = 0.011$$



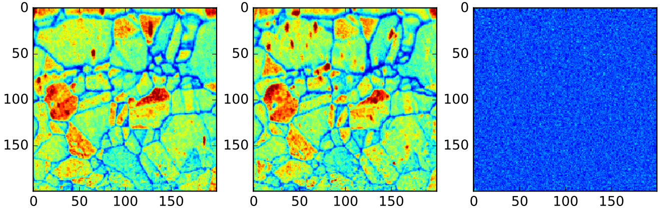
Apply affine transform

Stack alignment

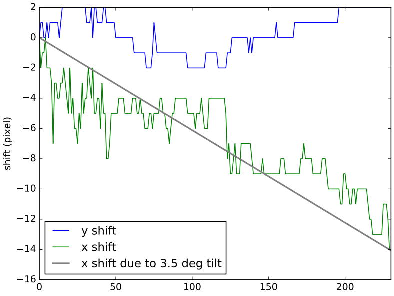
Leverage IQ contrast in the central region of the slices (for the example the shift has been increased 5 times) :



Compute cross correlation :

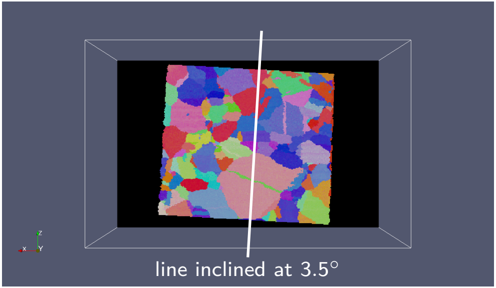


Stack aligned



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

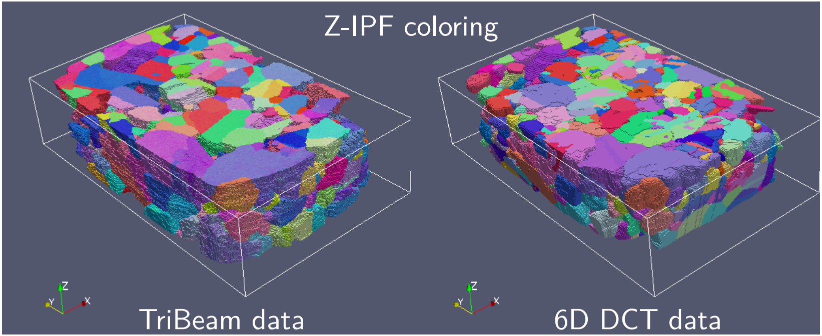
after applying the shifts, the reconstruction shows the tilt in the YZ plane, evaluated at 3.5°



line inclined at 3.5°

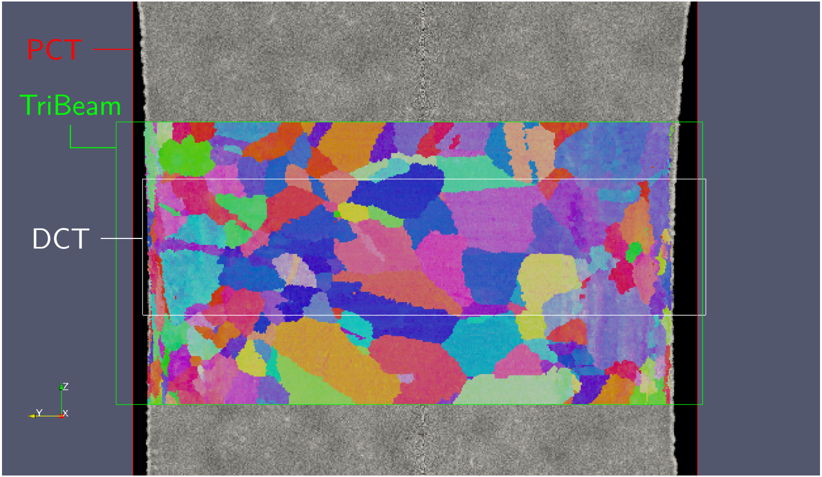
Registration of the TriBeam data set with the DCT

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



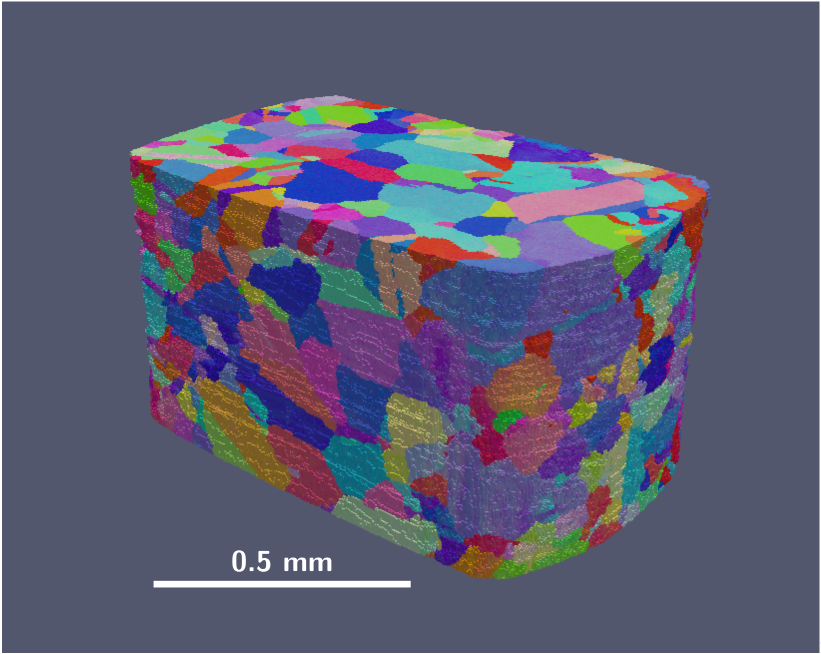
→ Affine transform to bring the TriBeam data in the same coordinate system as the DCT data. Transformation is $\sim$ a rotation of 3.5° around Y.

TriBeam reconstruction merged with PCT/DCT data sets

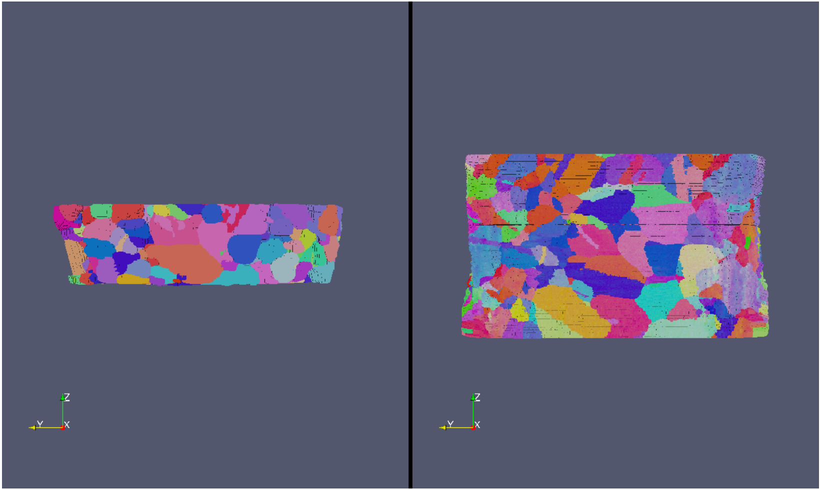


All 3 data sets merged into a single data container

TriBeam reconstruction merged with PCT/DCT data sets



Overall comparison DCT/TRIBEAM



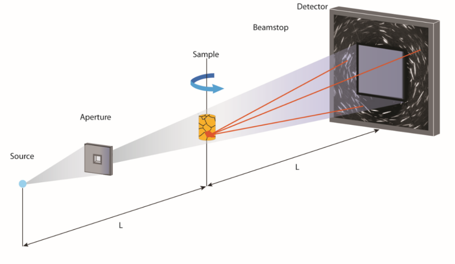
Contents

Summary

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

- Very complementary to serial sectioning methods, diffraction contrast tomography is **fast** ($\sim$ hour) and **non destructive** ; reconstruction can be tedious/impossible for complex microstructures, but algorithms constantly improve (6D-DCT).
- Design of **mechanical rigs** adapted for X-ray orientation imaging allow to study the (eg. polycrystal plasticity, fatigue, stress corrosion racking. . .) ;
- Full-field simulations (FE but also FFT) with experimental microstructure is a key to **compare experiments and simulation** on a grain to grain basis.

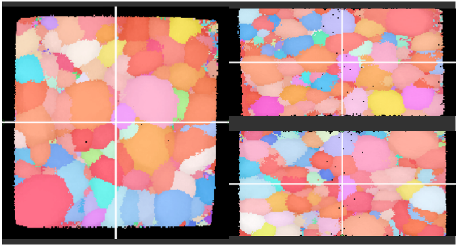
Outlook : DCT can now be done in the laboratory !

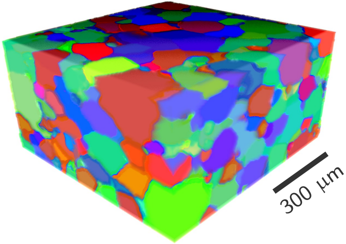


Versa 520 from Zeiss

LabDCT Laue focusing geometry

Example on a 1 mm Ti7Al specimen (3.4 µm resolution) :





Thanks to Hrishikesh Bale (Carl Zeiss, Pleasantown, CA.)

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