Measuring the elastic strain of individual grains in a polycrystalline material

Jette Oddershede, Søren Schmidt, Henning Friis Poulsen
Risø DTU

and Walter Reimers
TU Berlin

Measuring the elastic strain of individual grains in a polycrystalline material

- Why?
  - To study grain-grain interactions in deformed materials – Does the strain depend on the grain orientation and/or the neighbouring grains?
  - To study residual stresses
  - To study crack formation and propagation and the role of reinforcements for the process

- How?
  - Farfield 3DXRD

- July 2008 workshop on 3DXRD software for strain in grains:
  - C. Aydiner, J. Bernier, J. Wright, U. Lienert, P. Reischig (W. Ludwig)
  - M. Miller, A. Borbely

- FitAllB – Fable package for fitting grain resolved centre of mass positions, orientations and elastic strains

FitAllB

2 3 6 hkl

$$\sum_{i,j(i)} \left(\Gamma_{ij}^{-1}\overline{G}_{ij} - \frac{\lambda}{2\pi} U_i B_i \overline{G}_{hkl,ij}\right)^T V_{ij}^{-1} \left(\Gamma_{ij}^{-1}\overline{G}_{ij} - \frac{\lambda}{2\pi} U_i B_i \overline{G}_{hkl,ij}\right)$$

$$\overline{G}_{ij} = \left[\frac{\bar{d}_{ij}}{|\bar{d}_{ij}|} - \begin{pmatrix}1\\0\\0\end{pmatrix}\right]$$

3

$$\bar{d}_{ij} = R \begin{pmatrix}0\\(y_{det,ij} - y_{det,0})p_y\\(z_{det,ij} - z_{det,0})p_z\end{pmatrix} + \begin{pmatrix}D\\0\\0\end{pmatrix} - \Gamma_{ij}\begin{pmatrix}x_{0,i}\\y_{0,i}\\z_{0,i}\end{pmatrix}$$

Observations
3 per reflection

Global parameters
10

Grain parameters
12 per grain

Fable input

- peaksearch:
  - images →
  - filtered peaks file (.flt: ω, dety, detz, spotid, intensity)

- transformation:
  - peak positions (.flt) + detector parameters (.par) →
  - scattering g-vectors (.gve)

- GrainSpotter:
  - g-vectors →
  - oriented grains (.log: spotid, h, k, l, orientations, positions)

FitAllB input

- flt_file al20_peaks_t25.flt
- par_file al20_detector.par
- log_file al20_grainspotter.log
- structure_file al.cif
- dety_size 2048
- detz_size 2048
- w_step 0.5
- w_limit -22.5 22.5 67.5 112.5
- crystal_system cubic
- c11 10.8e10
- c12 6.22e10
- c44 2.84e10
- skip 4 # skip grain 4 in al20_grainspotter.log
- ia 0.2
- min_refl 60

FitAllB input

- w 0 # Fit omega stage tilt parameter wy (wedge)
- center 0 # Fit beam centre on detector in y direction, cy
- pixel 0 # Fit pixel size py and pz
- tilt 0 # Fit detector tilt parameters tx, ty, tx
- L 0 # Fit sample-to-detector distance

- rod 1 # Fit orientations (Rodrigues vector)
- xyz 1 # Fit positions
- eps 1 # Fit strain tensors

FitAllB output

- A parameter file containing the following parameters for each grain:
  - grainno mean_IA grainvolume x y z
  - rodx rody rodz U11 U12 U13 U21 U22 U23 U31 U32 U33
  - eps11 eps22 eps33 eps23 eps13 eps12
  - eps11_s eps22_s eps33_s eps23_s eps13_s eps12_s
  - sig11 sig22 sig33 sig23 sig13 sig12
  - sig11_s sig22_s sig33_s sig23_s sig13_s sig12_s

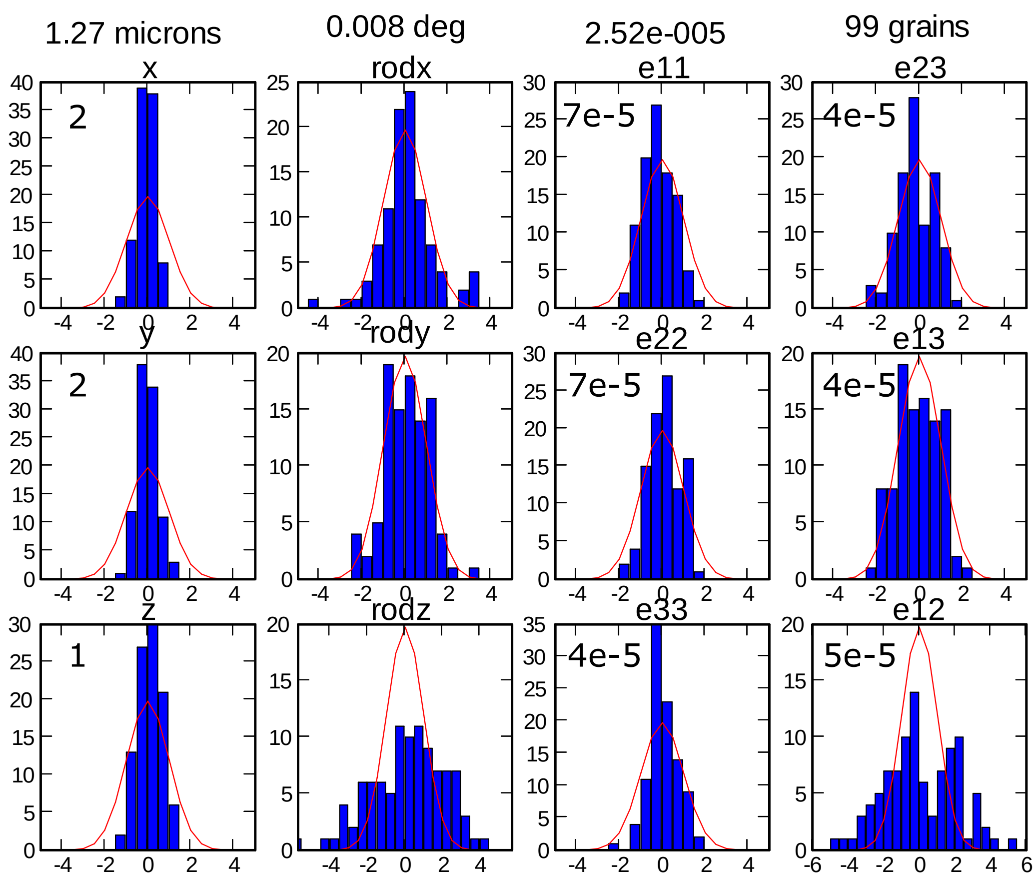
- An error file containing the estimated errors of the above parameters

Tests on simulated data - PolyXSim

- Model systems
  - F.C.C. or B.C.C. metals
  - 100-200 illuminated grains
  - Positions randomly distributed in cylinder with Ø=0.5-1 mm and h=0.01-0.1 mm
  - Random orientations
  - Lognormal distribution of grain sizes
  - Random strain, Gaussian distribution with $\mu=0$ and $\sigma=0.001$
  - ~70 keV, 2048×2048 pixels detector, 50×50 µm pixels (ID11 Frelon4M) sample-to-detector distance to give 5 full diffraction rings

- Present example
  - 100 grains of IF steel (B.C.C)
  - ω-ranges: $-22.5 \rightarrow 22.5^\circ$ and $67.5 \rightarrow 112.5^\circ$ in steps of 0.5°

Simulated data and error estimation
Idealised geometry
Detector discretisation



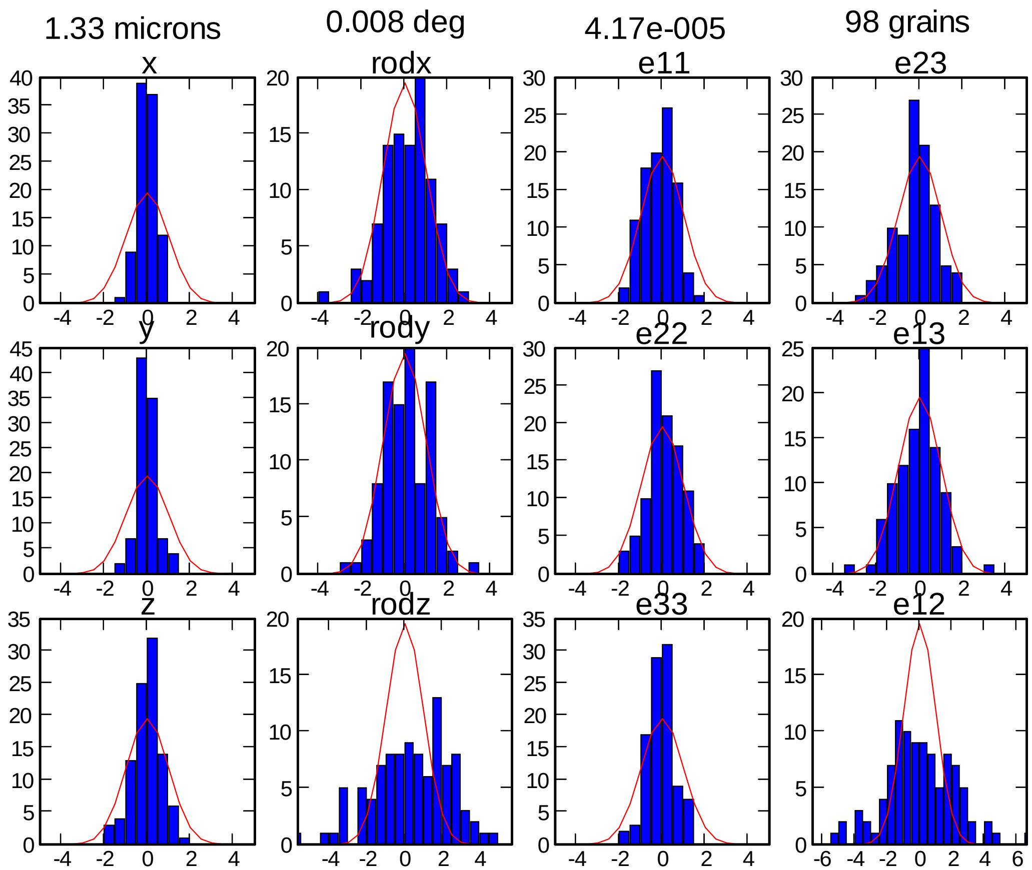
For each of the 12 grain parameters:

x-axis:
(refined – true value)/ estimated error

y-axis:
Number of observations

Red curve:
Gaussian with $\mu=0$ and $\sigma=1$, expected for correct error estimation

Idealised geometry
Diffractometer vibrations in y and z
(2D-Gaussian, σ=1 μm)



For comparison:

1.27 microns
0.008 deg
2.52e-5

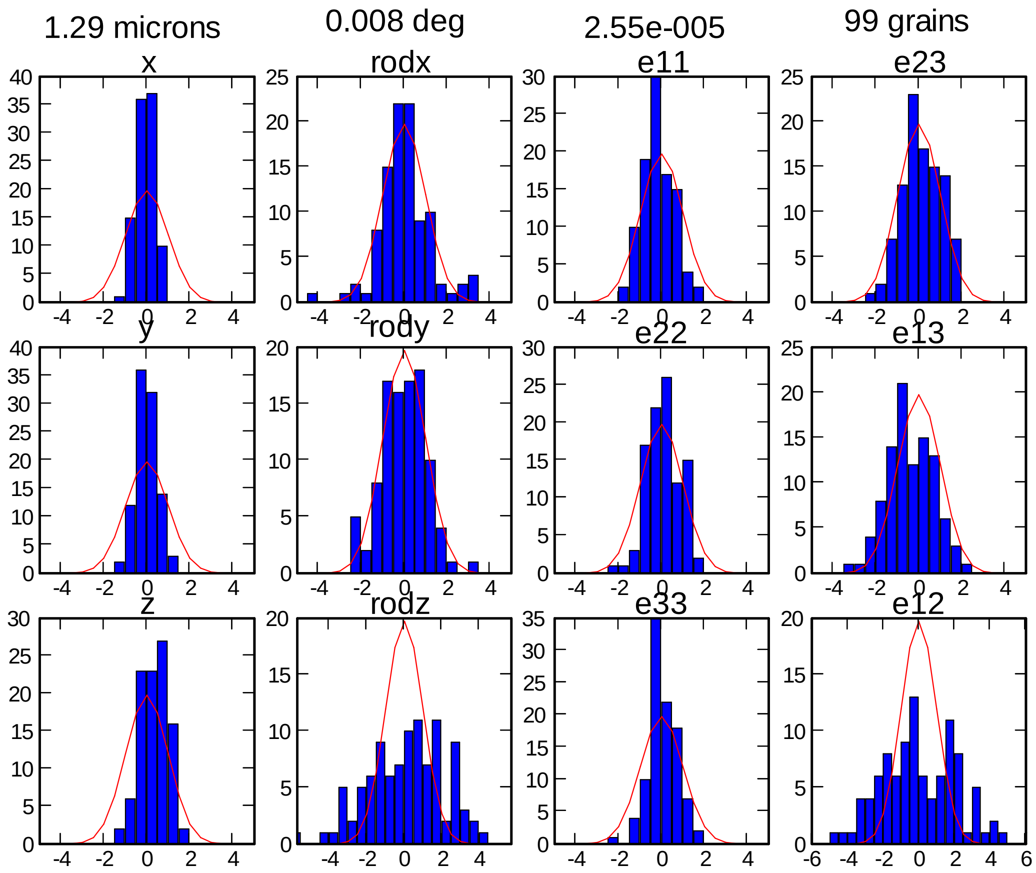
Fitting global parameters

- Fitglobalgrain or Fitgloball for multigrain global parameter refinements
- Same input file as for FitAllB, but with the following options:

- w 1 # Fit omega stage tilt parameter wy (wedge)
- center 1 # Fit beam centre on detector in y direction, cy
- pixel 0 # Fit pixel size py and pz
- tilt 1 # Fit detector tilt parameters tx, ty, tx
- L 1 # Fit sample-to-detector distance

- rod 1 # Fit orientations (Rodrigues vector)
- xyz 1 # Fit positions
- eps 0 # Fit strain tensors

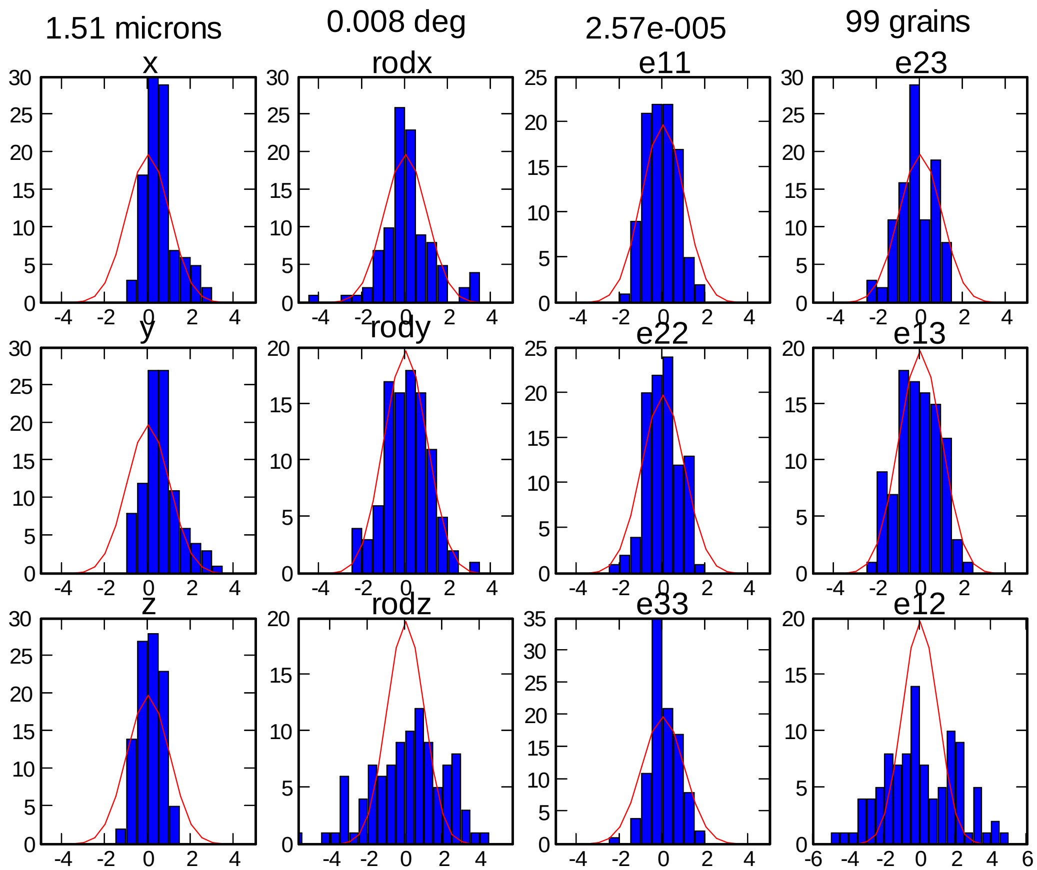
Sample tilt (wedge) off by 0.001°



For comparison:

1.27 microns
0.008 deg
2.52e-5

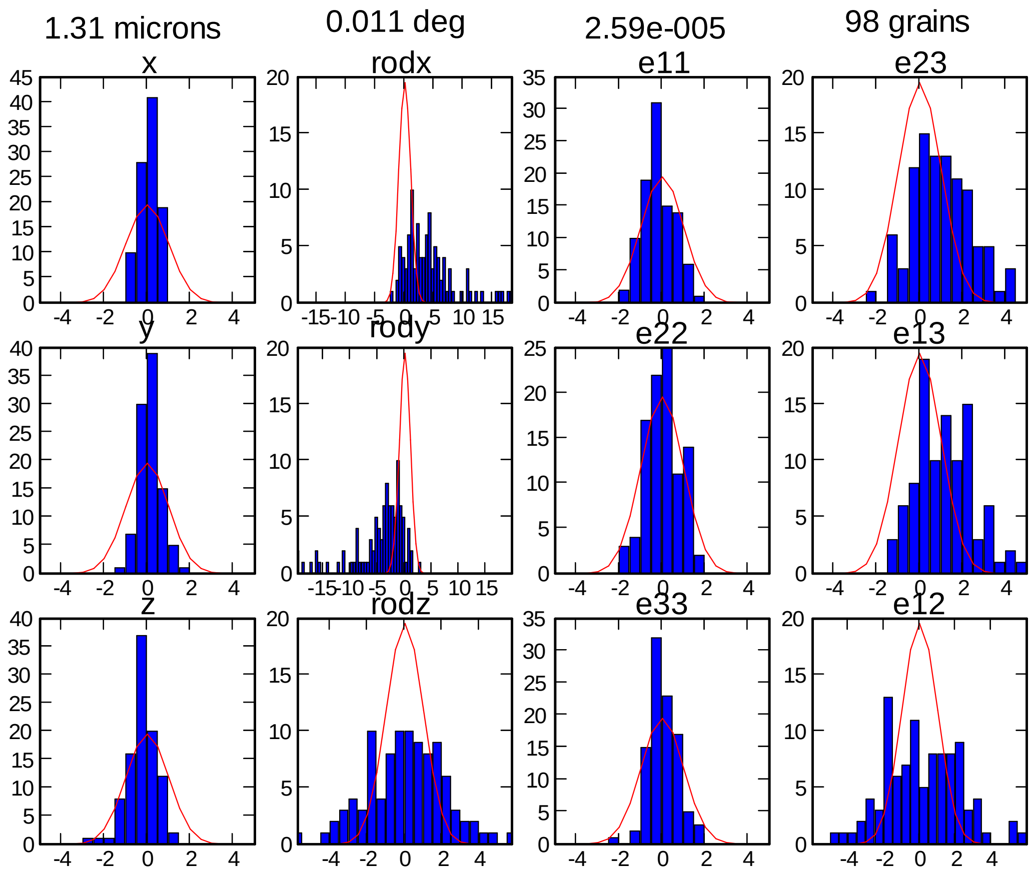
Beam centre on detector off by 0.01 pixels in y direction (0.5 μm)



For comparison:

1.27 microns
0.008 deg
2.52e-5

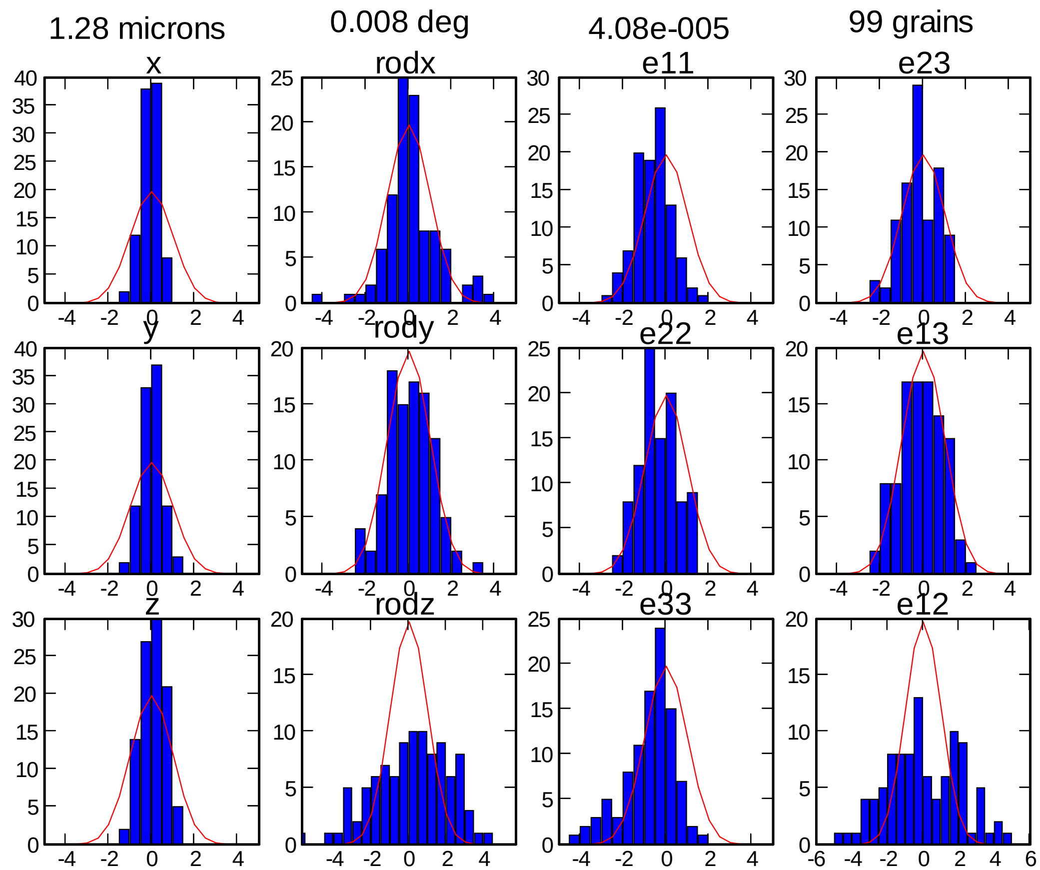
Detector tilt off by 0.006°



For comparison:

1.27 microns
0.008 deg
2.52e-5

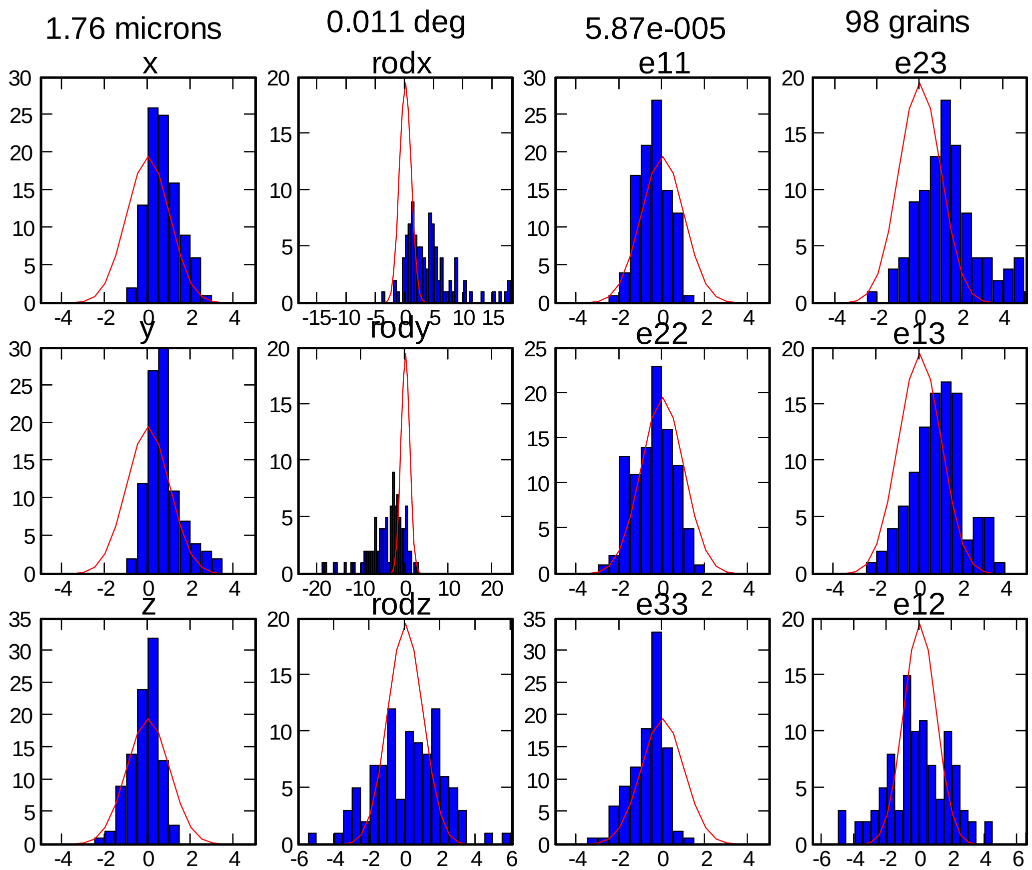
Sample-to-detector distance off by 2 µm on 200 mm (1e-5)



For comparison:

1.27 microns
0.008 deg
2.52e-5

Fitted geometry
Detector discretisation
Diffractometer vibrations



For comparison:

1.27 microns
0.008 deg
2.52e-5

With vibrations:

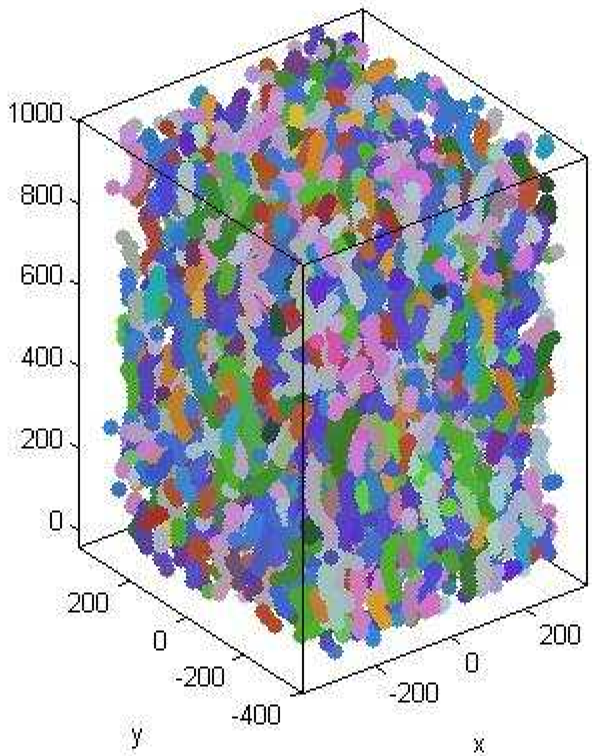
1.33 microns
0.008 deg
4.17e-5

Experimental data
Refining global parameters

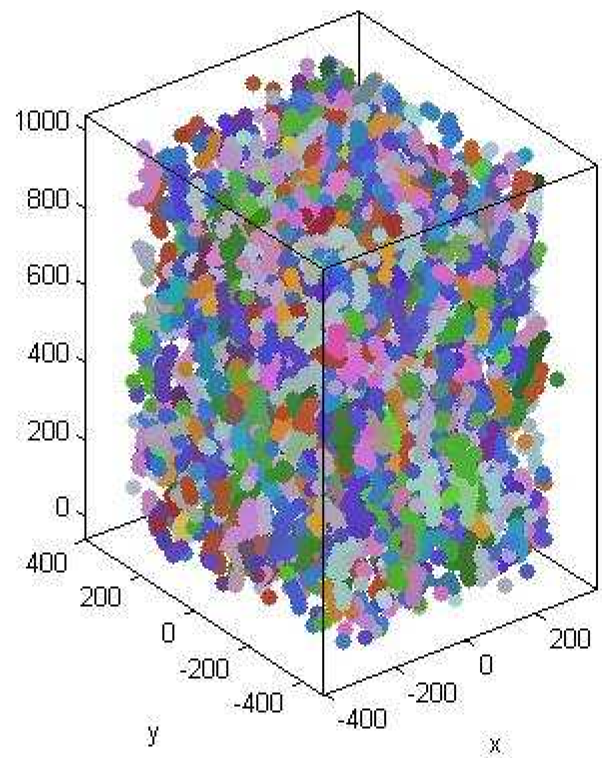
- Global parameters for Al reference single crystal
  - wedge = -0.0206°
  - y_center = 1019.06
  - tilt_x = -0.00041
  - tilt_y = 0.00303
  - tilt_z = -0.01337
  - L = 252.355 mm
  - Possible to index ~35% of the reflections

- Global parameters for 5 layers of undeformed Cu, 161 grains
  - wedge = -0.3552(7)°
  - y_center = 1018.743(11)
  - tilt_x = -0.00066(1)
  - tilt_y = 0.00414(10)
  - tilt_z = -0.01063(10)
  - L = 252.358(4) mm
  - Possible to index ~45% of the reflections

Experimental data, level 1:
Centre of mass position and orientations
IF steel, ex situ deformed



Undeformed
1939 grains



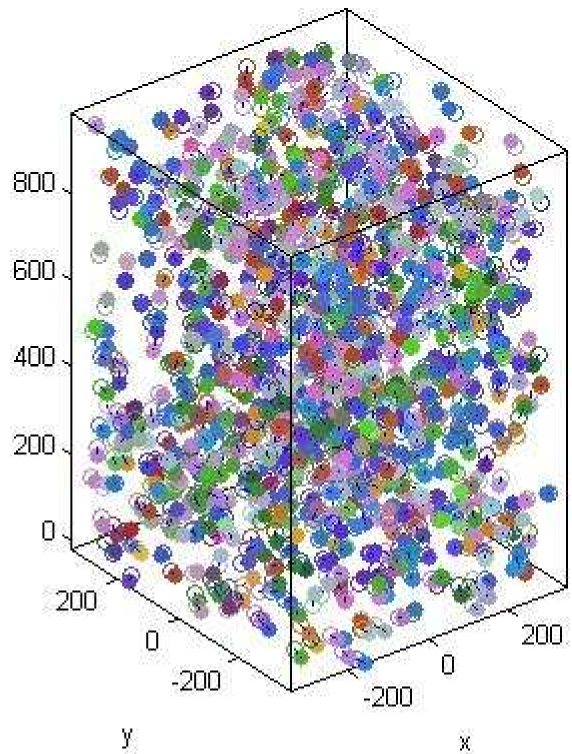
3% deformed
1766 grains

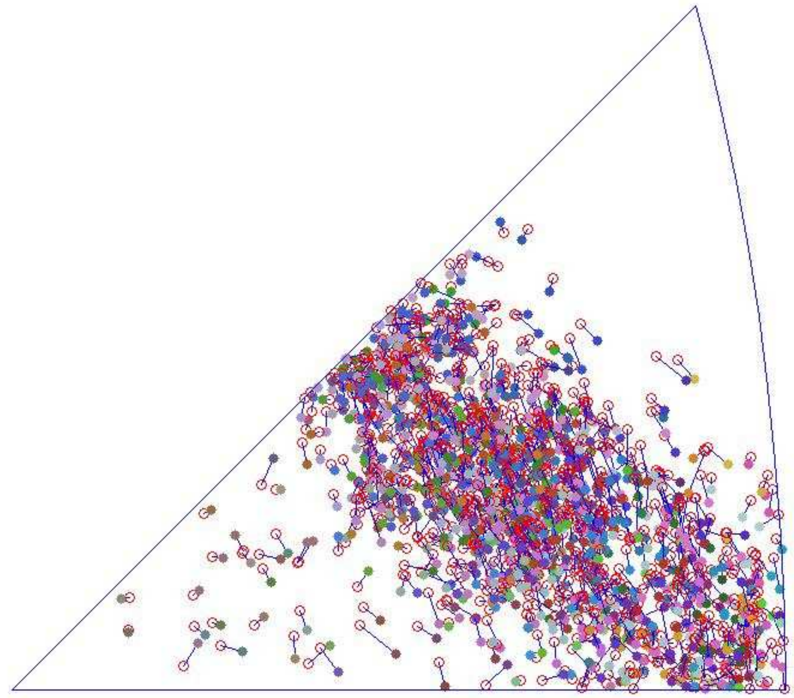
Est. error on pos.: 3 µm — 5 µm

NB! Position fit using near-field data

G. Winther, H.F. Poulsen, L. Margulies, M. Kobyashi, J. Oddershede, S. Schmidt, J. Wright – in progress

Experimental data, level 1: Centre of mass positions and orientations IF steel, ex situ deformed



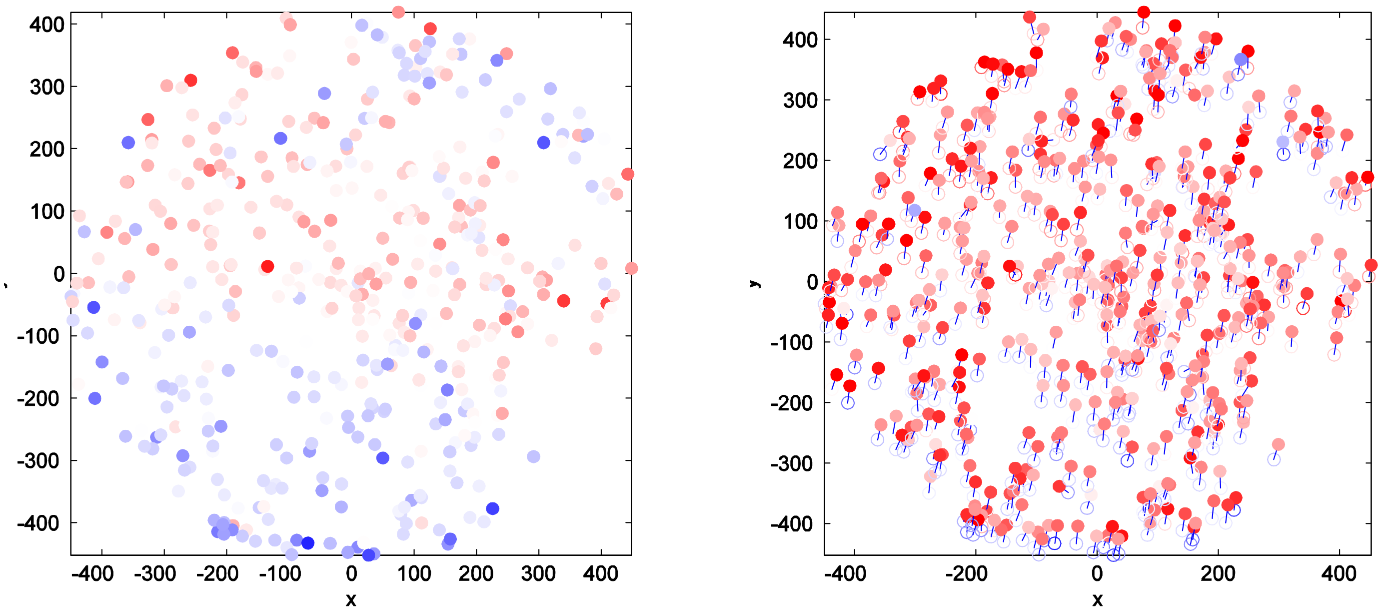


Possible to match 1186 grains (of 1939 and 1766)

To study grain rotations during deformation

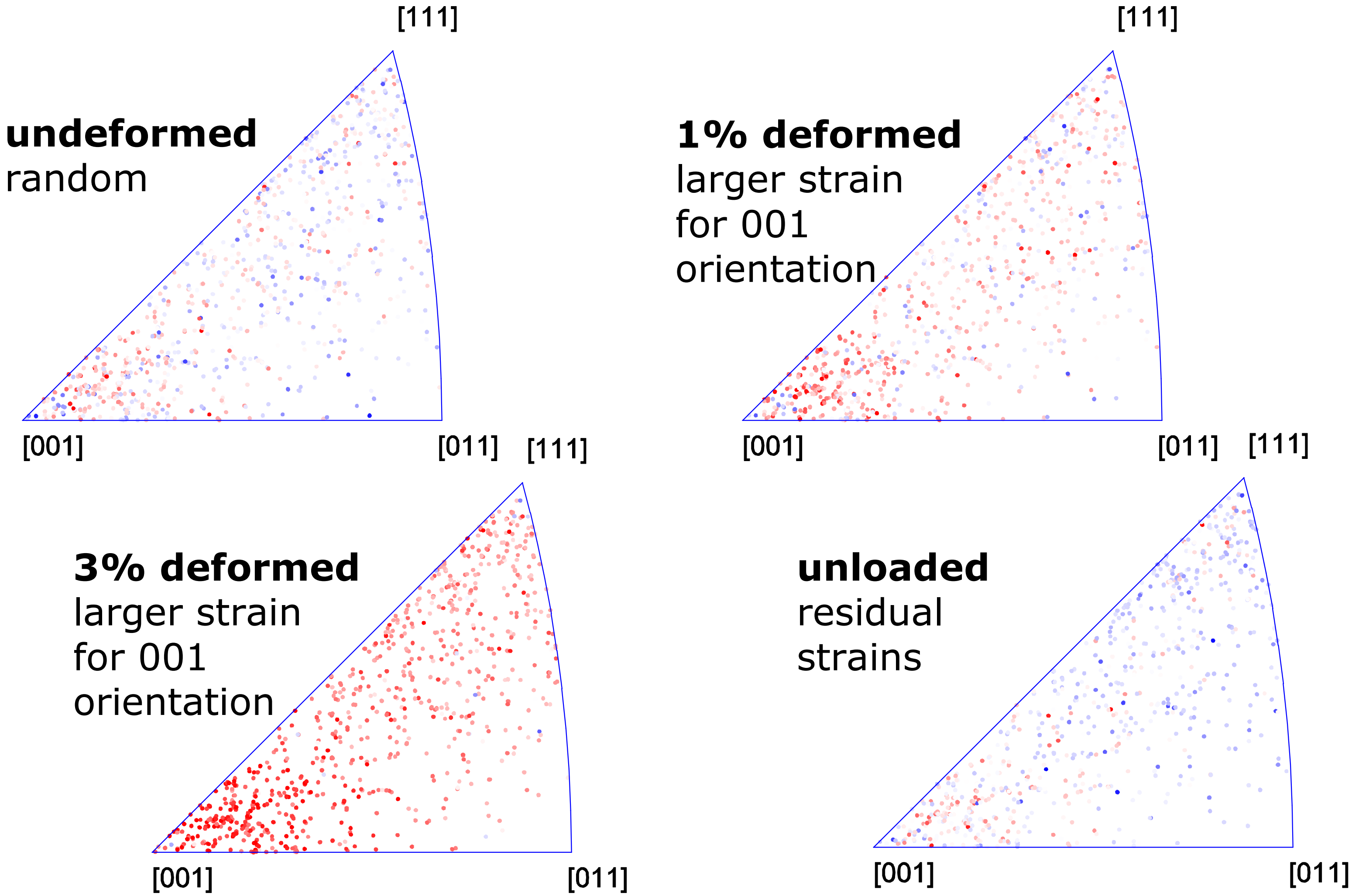
Experimental data, level 2: Positions, orientations and strains Cu, deformed tensionally in situ

- Strain levels: undeformed, 1%, 3% and unloaded
- Sample diameter 1 mm, 5 layers of 0.1 mm mapped
- 800 large grains indexed and refined
- 450 of these match between undeformed and 3% deformation



J. Oddershede, G. Winther, H.F. Poulsen, L. Margulies, M. Moscicki, S. Schmidt, J. Wright – in progress

Experimental data, level 2a: Correlating strain and orientation Cu, deformed tensionally in situ



Experimental data, level 3: Laguerre tesselation, grain maps and grain interaction studies

- Laguerre tesselation:
  - Method to get 3D grain map from centre of mass positions and relative grain volumes

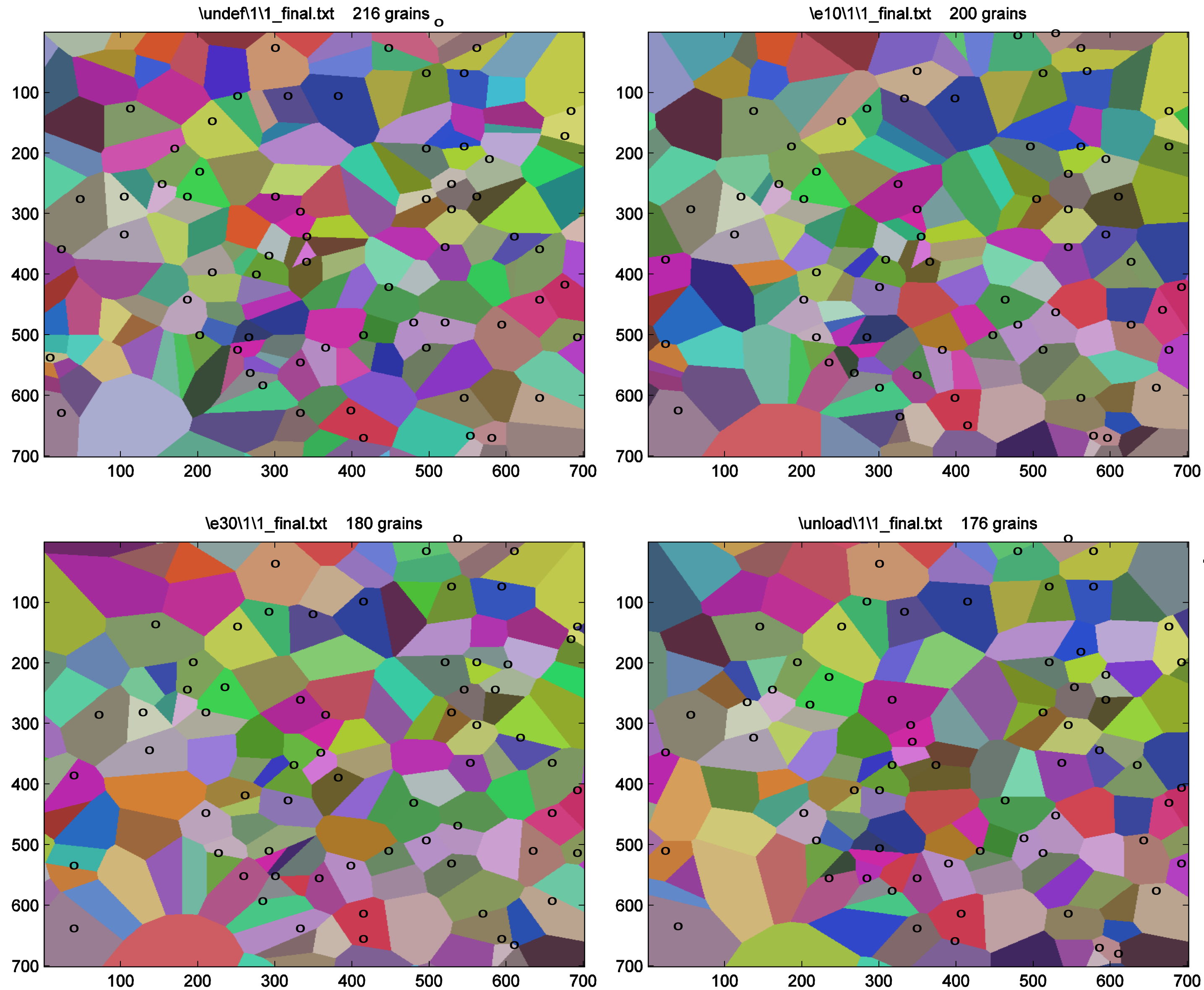
- Test on position, volumes and grain shapes from from microtomography on meta-stable beta-titanium alloy

  (A. Lyckegaard, E.M. Lauridsen, W. Ludwig, R.W. Fonda, H.F. Poulsen)

	Voronoi	Laguerre					
Error type	None	None	Volume	CMS			
Std. of error, 3 sigma	-	-	10%	2 µm	4 µm	7 µm	10 µm
% Correct labelled voxels	59.72	86.30	86.26	85.88	84.72	81.85	78.25
% grains with all neighbours correct	7.82	31.75	30.90	28.80	23.82	16.99	10.15
# erroneously extra neighbours/grain	1.87	0.58	0.59	0.62	0.73	0.93	1.23
# erroneously missing neighbours/grain	1.29	0.64	0.65	0.69	0.76	0.96	1.24
# total of wrong neighbours/grain	3.16	1.22	1.24	1.31	1.49	1.89	2.47

Table 1: Average similarity measures for the tessellations: Voronoi (N=1), Laguerre without errors (N=1), Laguerre with 10% volume errors (N=17) and Laguerre with 2 µm, 4 µm, 7 µm and 10 µm CMS errors (N=17).

Experimental data, level 3:
Cu, deformed tensionally in situ



estimated error on position: 10 μm

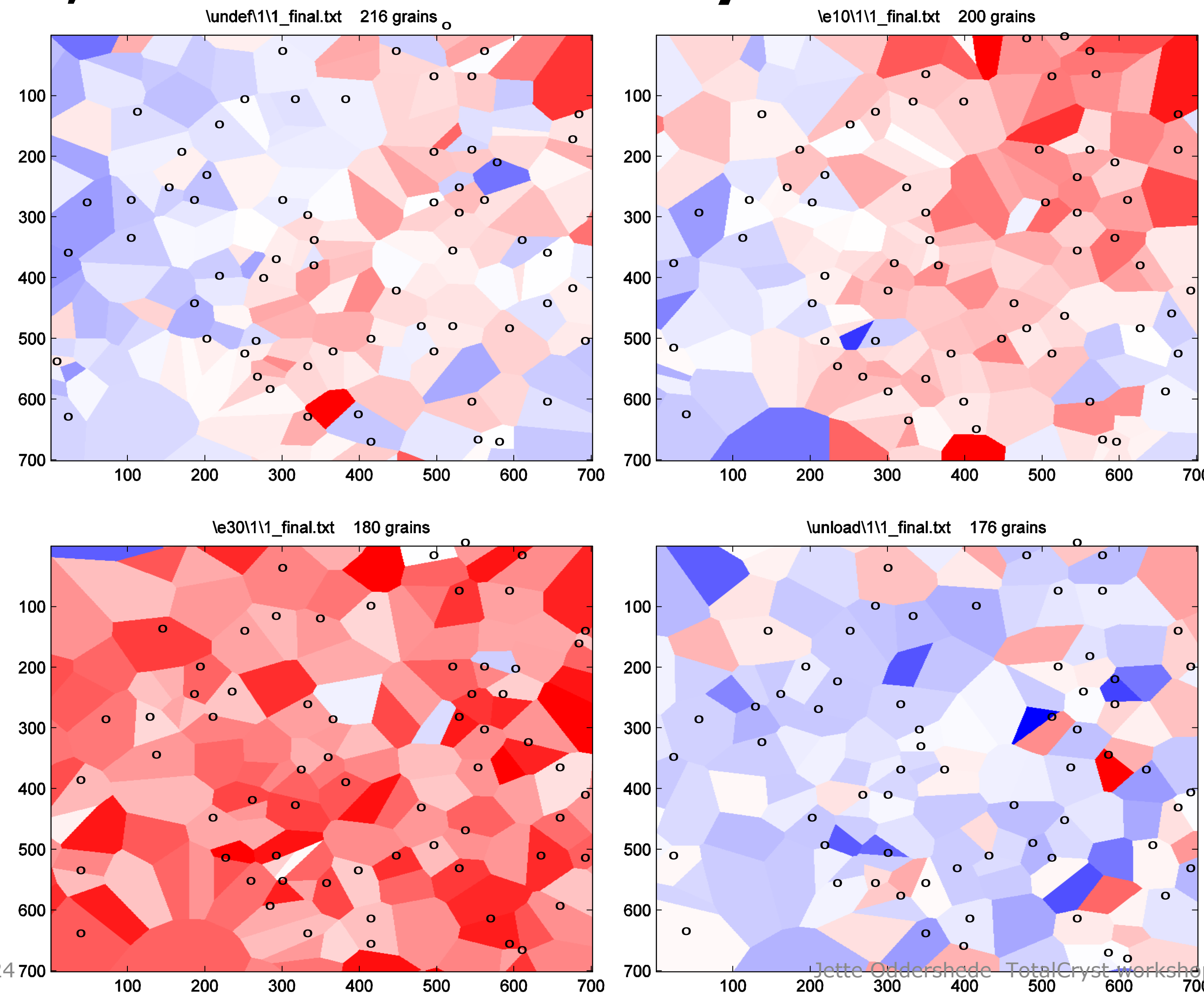
62 grains visually matched at all strain levels

Sample slip/ elongation

Twins!!

Combine knowledge from adjacent layers/strains to improve incomplete indexing

Experimental data, level 3
Cu, deformed tensionally in situ



undeformed
$\varepsilon_{33}^{avg} = 1.2e\text{-}5$
$\varepsilon_{33}^{err} = 1.1e\text{-}4$

1%
$\varepsilon_{33}^{avg} = 1.6e\text{-}4$
$\varepsilon_{33}^{err} = 1.1e\text{-}4$

3%
$\varepsilon_{33}^{avg} = 5.4e\text{-}4$
$\varepsilon_{33}^{err} = 1.2e\text{-}4$

unloaded
$\varepsilon_{33}^{avg} = -5.3e\text{-}5$
$\varepsilon_{33}^{err} = 1.2e\text{-}4$

Conclusions

- FitAllB for refining centre of mass grain positions, orientations and strain tensors and Fitglobalgrain/Fitgloball for refining global experimental parameters

- Simulated data used to validate error estimation and illustrate the necessity for accurate global parameters

- IF steel ex situ
  - ~2000 grains, good statistics
  - The use of near-field data for position fit significantly improved these

- Cu in situ
  - Average estimated error on ε_{33} strain in tensile direction $\leq$1.2e-4
  - Strain evolution along tensile axis detected
  - Orientation dependence of ε_{33} detected

Outlook

- IF steel ex situ:
  - Too low percentage of grains matched between undeformed and 3% deformed, must be improved
  - 6% and 9% data

- Cu in situ:
  - Twins, indexing
  - Strain and correction for spatial distortion of detector
  - Analysis of data measured at 0.2 % and 0.4 % deformation
  - Present data measured for ω-ranges: -150 → -30° and 30 → 150° in steps of 0.25°. Is one ω-range enough (speed gain)?
  - One layer remeasured in steps of 0.1°, is this an improvement?

- APS beamtime application for studying the grain resolved stress evolution around crack tips.

Acknowledgements

- J. Wright & H.O. Sørensen, my python and fable gurus
- G. Winther, everything on deformation theory
- Everyone who helped prepare the samples and collect the data
- M. Moscicki and A. Borbely, MPIE Düsseldorf, stress rig

Motivation

- Goal:
  - To determine the centre-of-mass elastic strains (and stresses - type II) in many (100+) grains to an accuracy of 10^{-4}

- To study what:
  - grain-grain interactions in elastically deformed materials
  - crack formation and propagation
  - residual stresses

- Approach
  - FitAllB – Fable package for fitting grain resolved centre of mass positions, orientations and elastic strains

Potential problems 2: Peak overlap

- Especially for textured and/or deformed materials

- Solutions:
  - Illuminate a smaller volume
  - Filter out peaks covering more than a certain number of pixels
  - Use several thresholds in peaksearch and merge the outcome

estimated error
on position:
10 μm