3D grain mapping using 3D detector at beamline ID11 at ESRF

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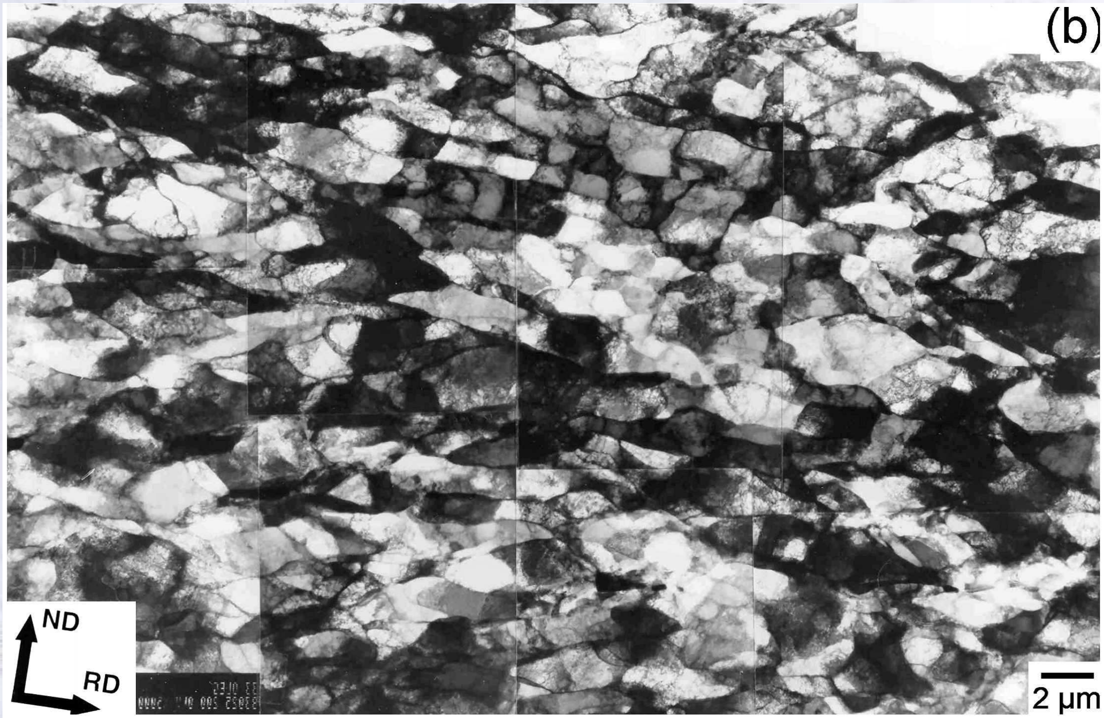
Outline

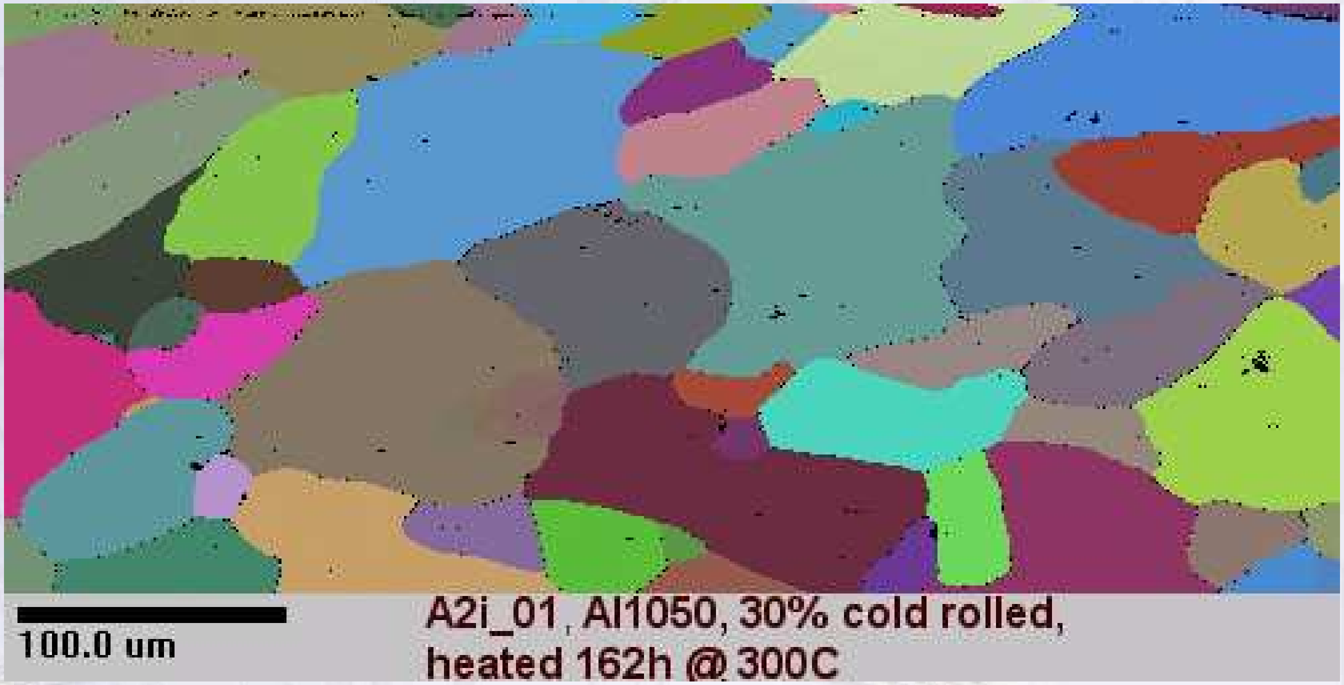
- Introduction
- Center of mass grain mapping
- 3D grain boundary mapping
- Results
- Future

TEM



Transmission through ~1 µm sample

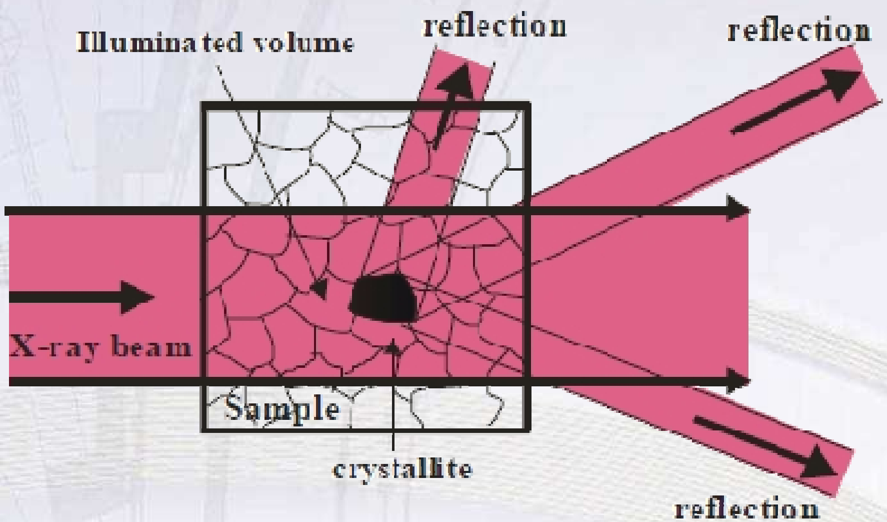
EBSD



- EBSD is a powerful surface technique
- Slices to make a 3D map

Why X-ray diffraction mapping?

- Grain evolution
- Many grains simultaneously
- X-rays scattered from crystal fulfilling the Bragg condition
- Monochromatic beam



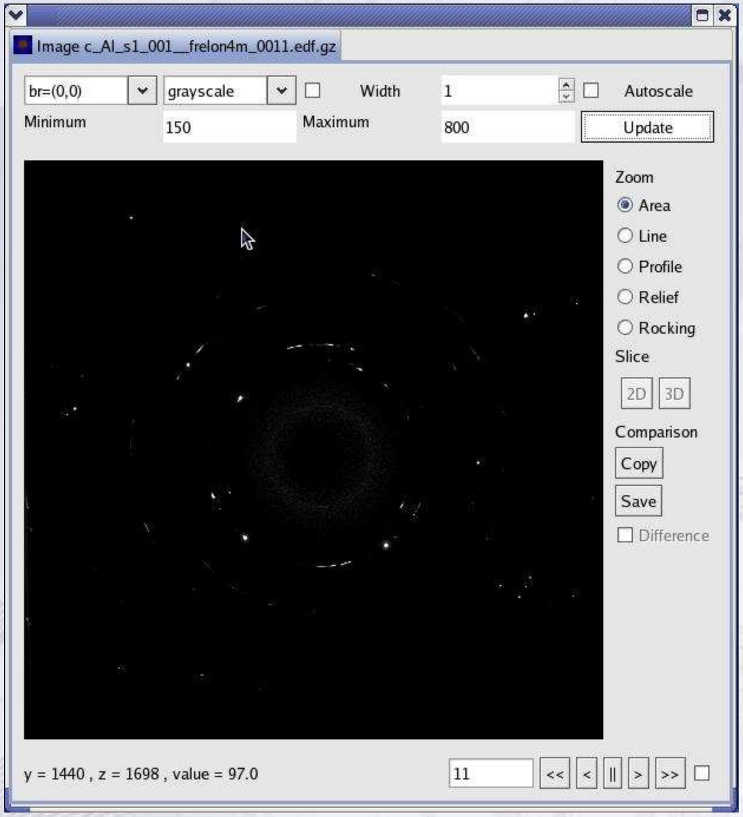
Grain positions, orientations, lattice parameters all simultaneously refined from multi-crystal data

Experimental setup

- Optics
  - Focosing bent single laue crystal
  - Double bent laue-laue monochromator
- Detectors
  - High resolution, Quantix (5 μm pixels)
  - Low resolution, Frelon (50 μm pixels)

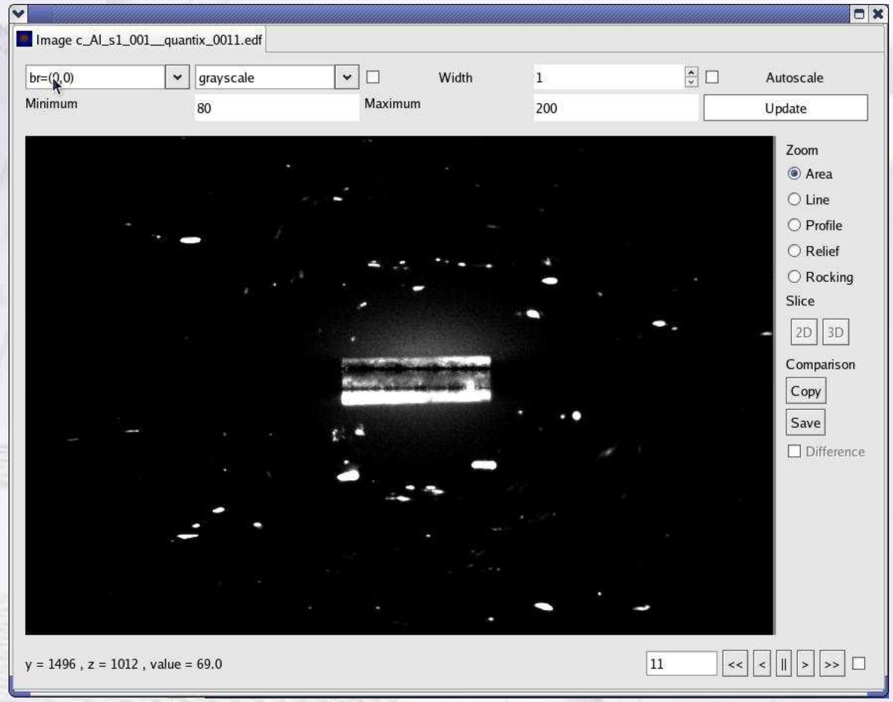
Detector images

- Frelon detector



2048x2048 pixels
102.4x102.4 mm

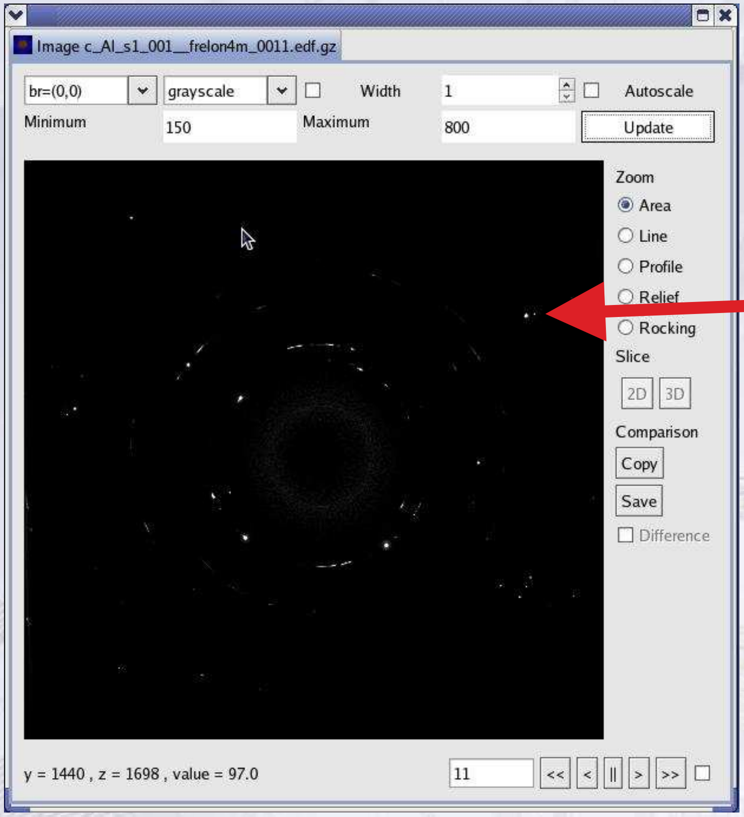
- Quantix detector



1536x1024 pixels
~6.6x4.4 mm

Detector images

- Frelon detector

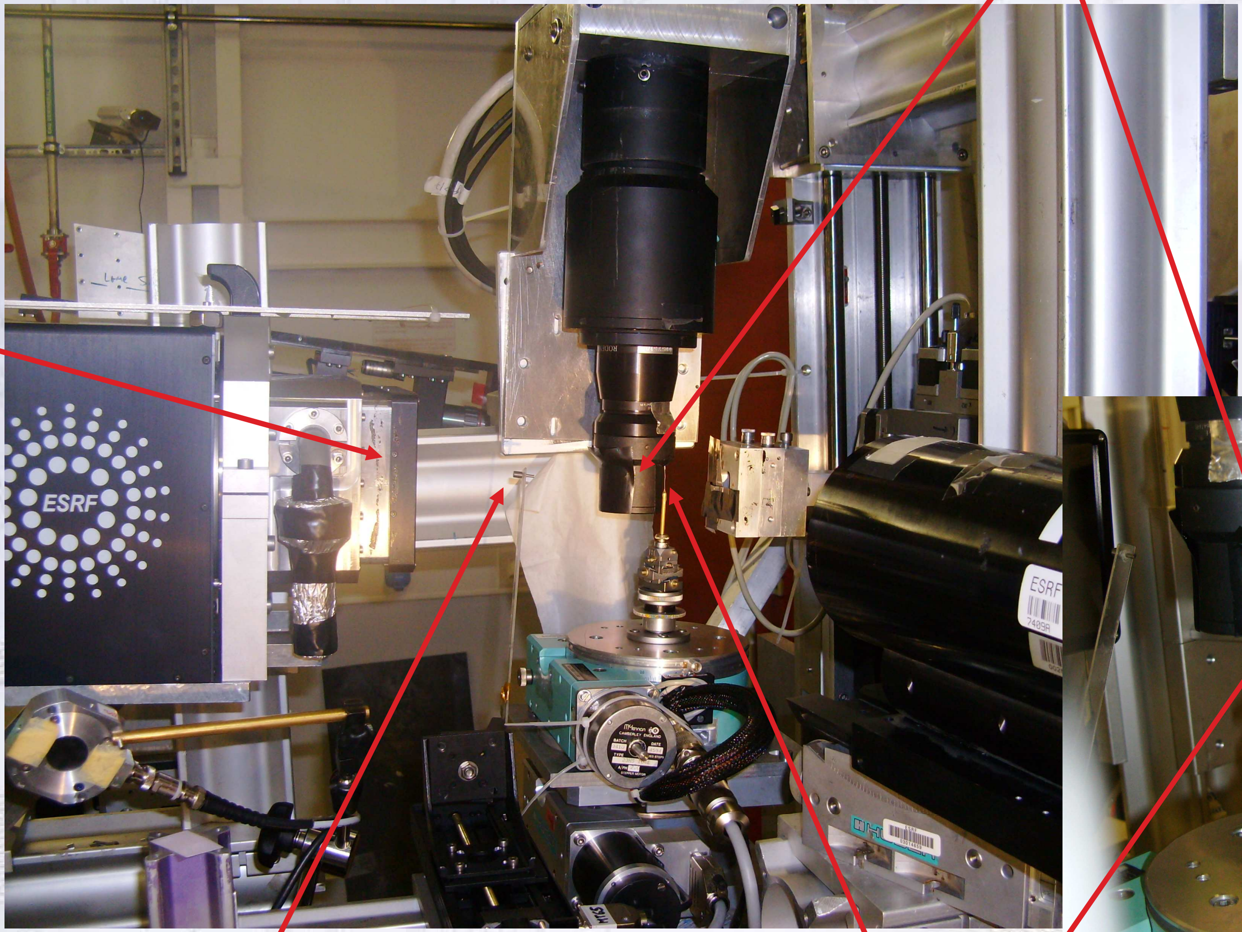


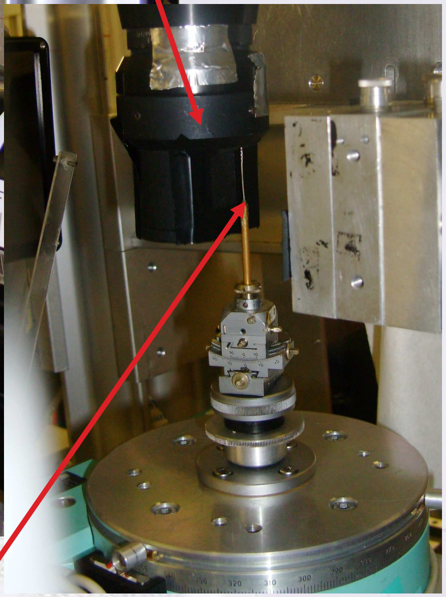
2048x2048 pixels
102.4x102.4 mm

- Quantix detector

1536x1024 pixels
~6.6x4.4 mm

3D detector





Quantix detector

Frelon detector

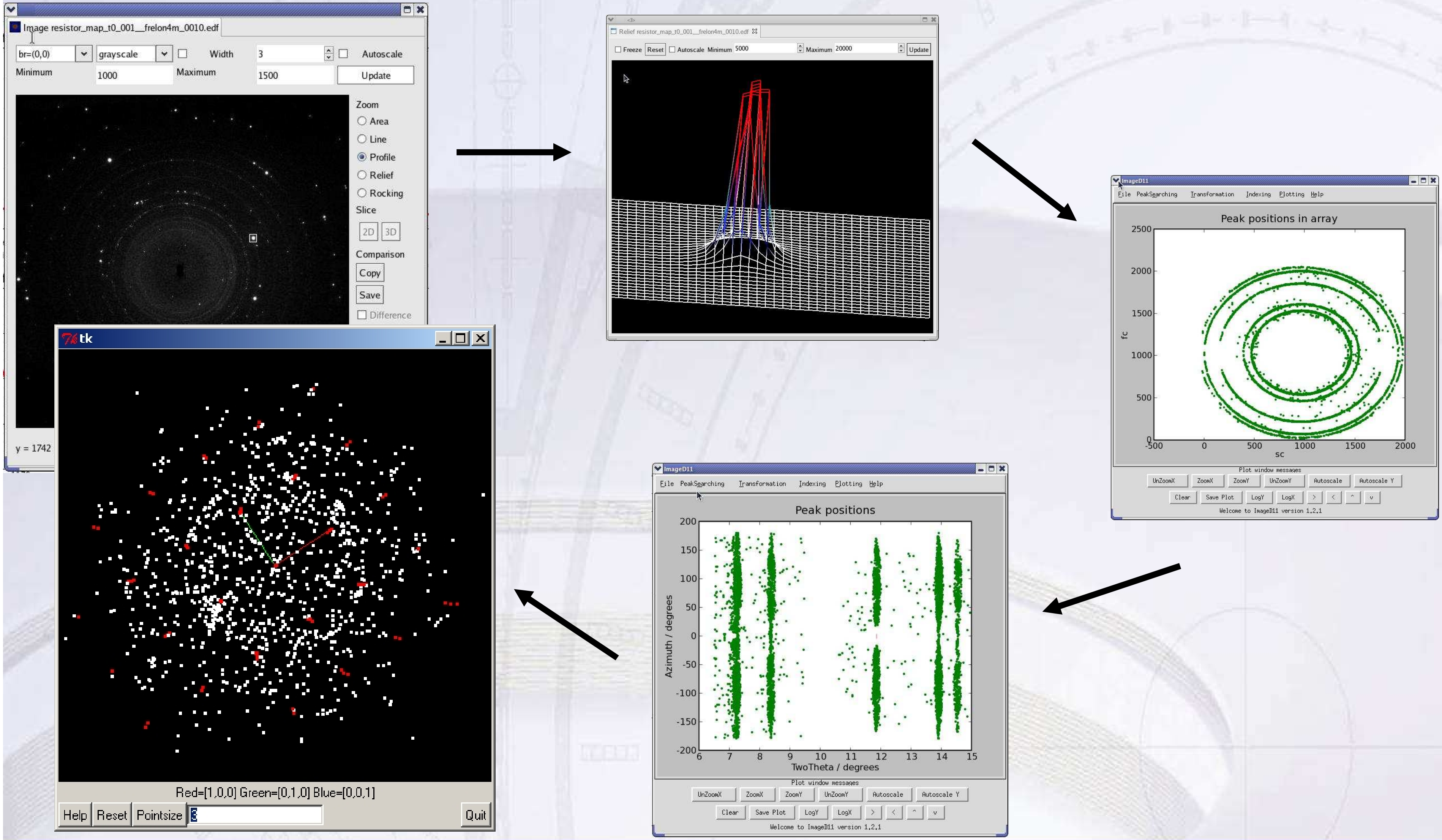
Beamstop

Sample

Center of mass grain mapping

- Starting with farfield data
  - Peaksearching
  - Calculate G-vectors from peak positions
  - Assign G-vectors to grains
  - Refine the assignment
  - Evaluate the grains

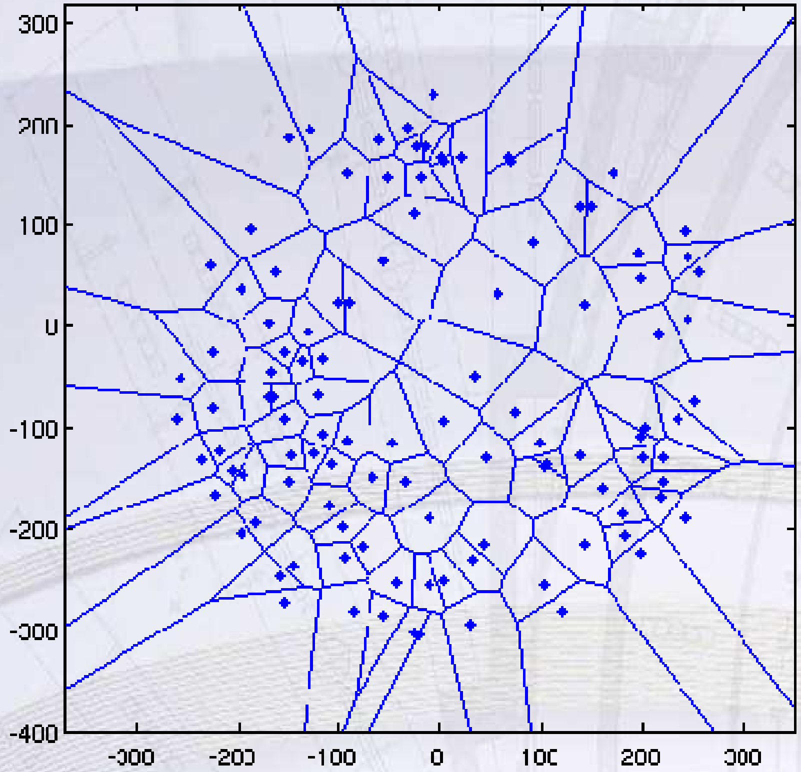
Center of mass analysis



Center of mass grain mapping

- Starting with farfield data
  - Peaksearching
  - Calculate G-vectors from peak positions
  - Assign G-vectors to grains
  - Refine the assignment
  - Evaluate the grains
- Finding grains with the near field detector
  - Peaksearching
  - Calculate G-vectors from peak positions
  - Refine grains from farfield in the set of G-vectors from nearfield
  - Evaluate the grains

Center of mass grain map

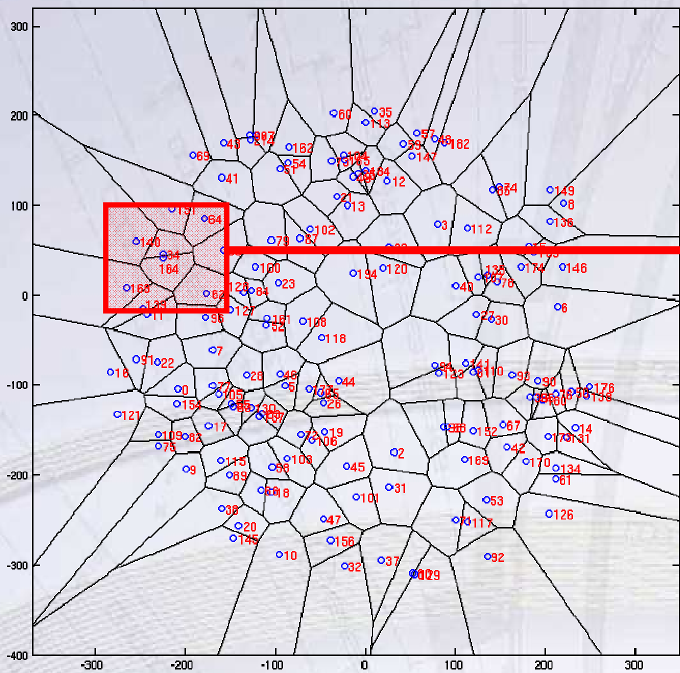


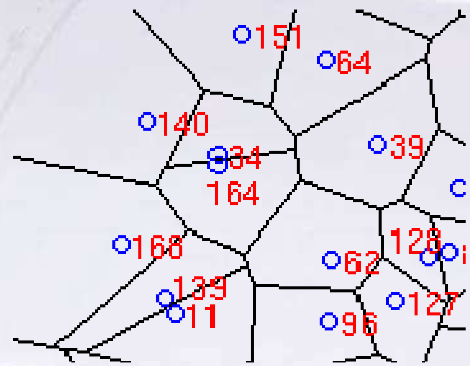
Grain positions, orientations, lattice parameters all simultaneously refined from multi-crystal data

"grain boundaries" from Voronoi calculation: if the grain centre falls in the middle of the reconstructed grain, perhaps nothing is missing.

Center of mass grain map

- Some grain centres almost perfectly overlap, but have different orientations. Twins?



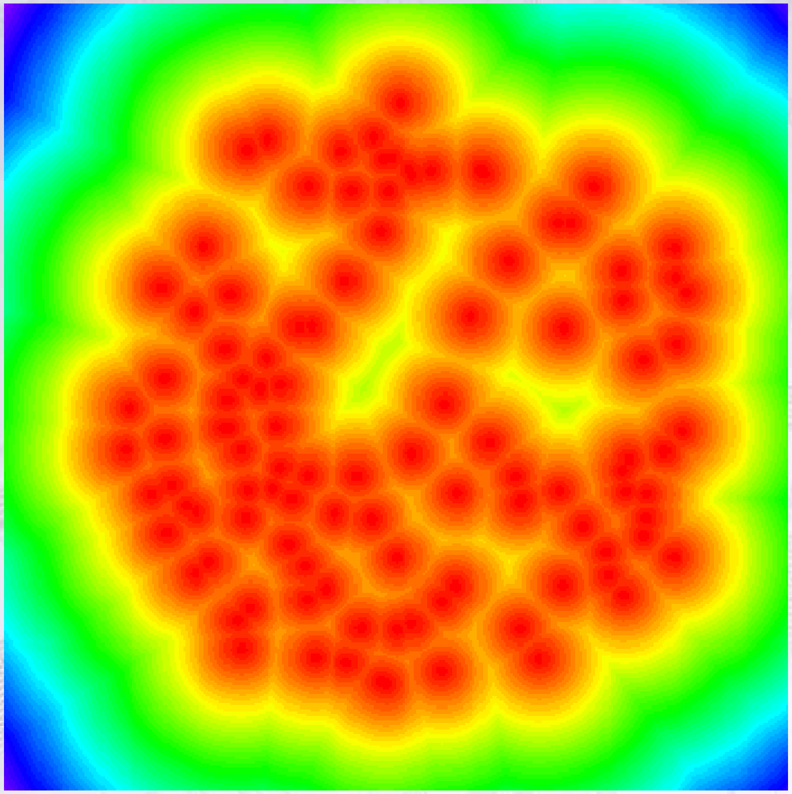


Furthermore, the average position would be at the centre of the total crystal

In this case, they share all <311>

Center of mass grain map

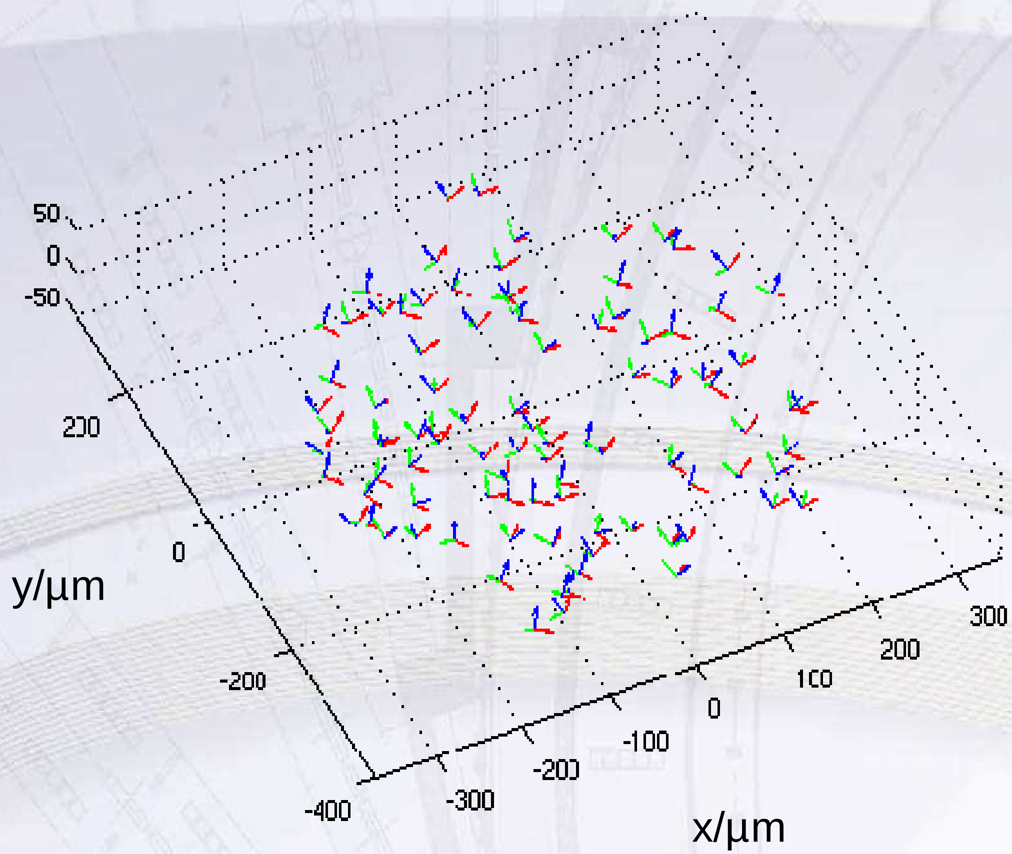
Better representation?



Here the colours fall away from the centre of mass and end at the boundaries – so that missing or vacant areas become more apparent…

Center of mass grain map

Could add some orientational information

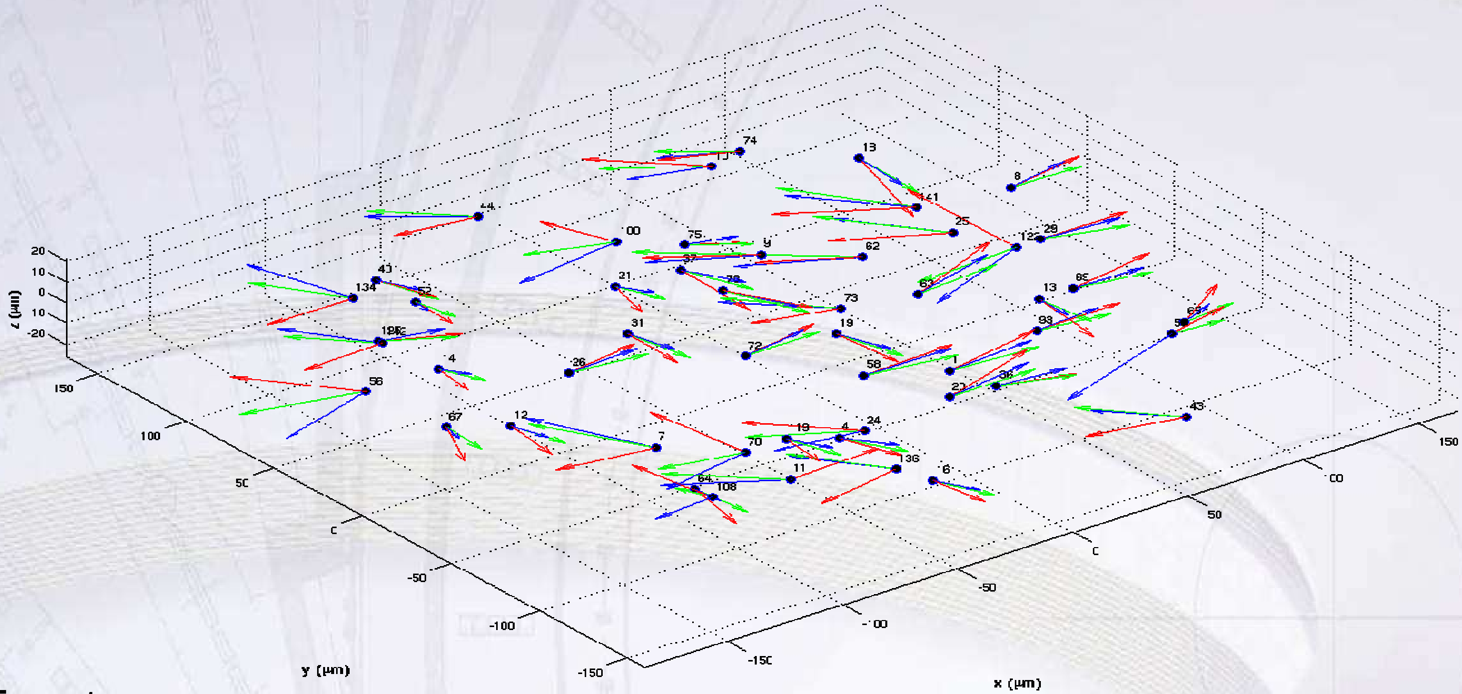


The axis are placed at the crystal centre.

Center of mass grain map

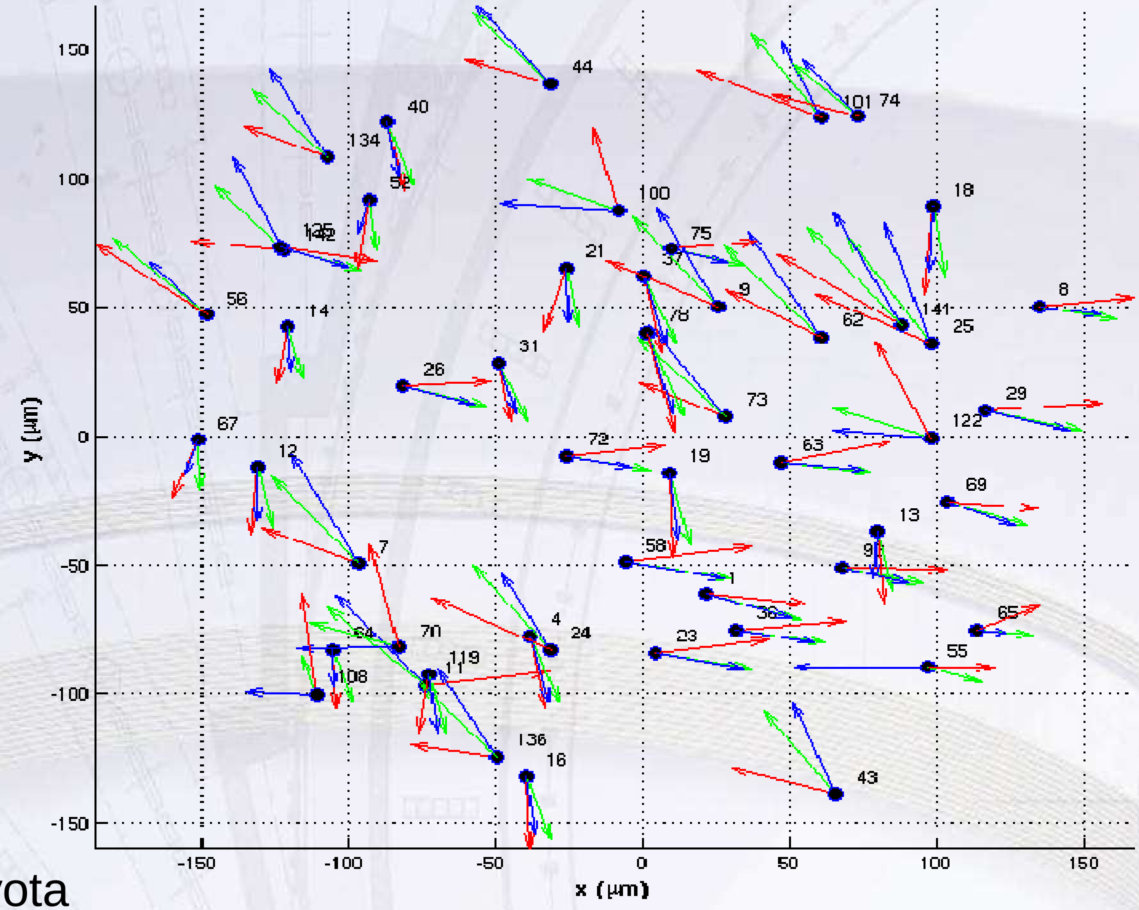
Rotations after each step

- Depicted are the Rodrigues vectors of subsequent rotations after straining a sample



Project with Toyota

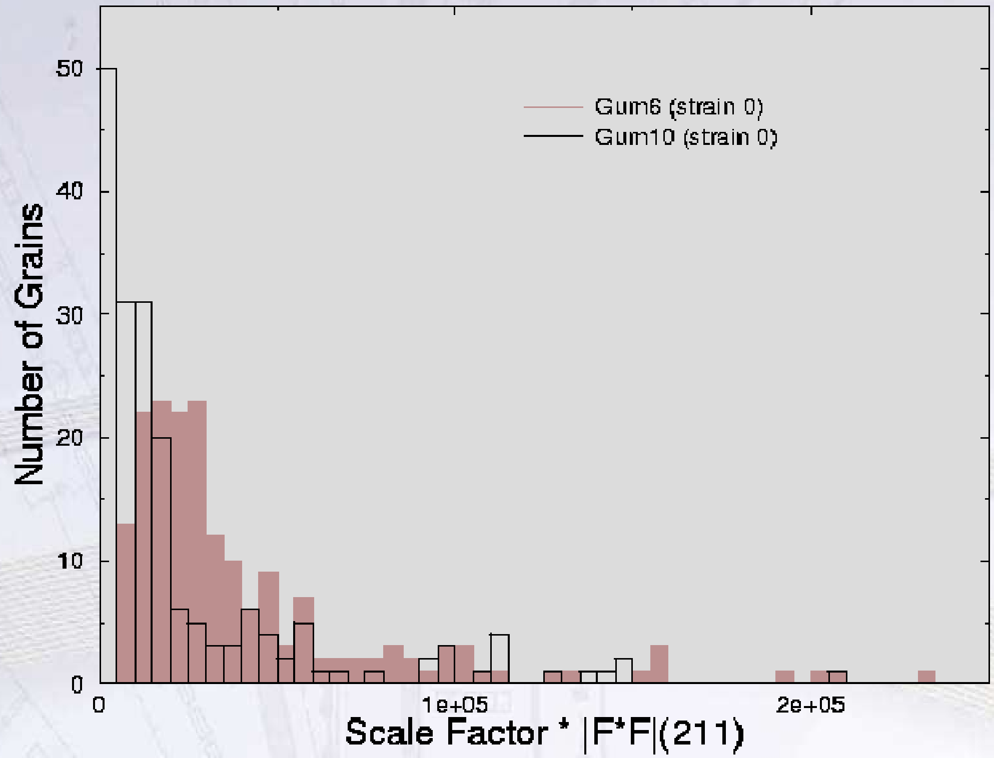
Center of mass grain map



Project with Toyota

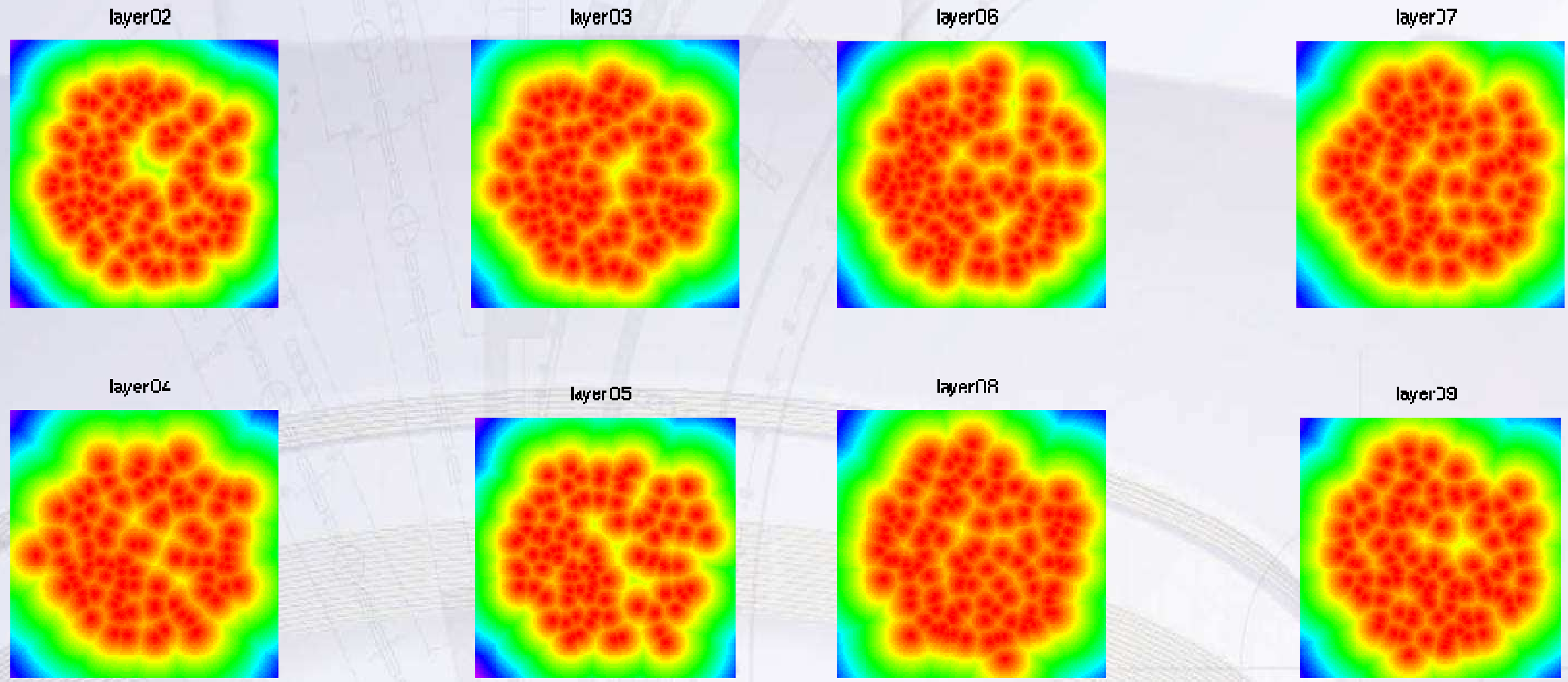
Center of mass grain map

Also correlate with grain size distribution



Center of mass grain map

Layer by layer maps



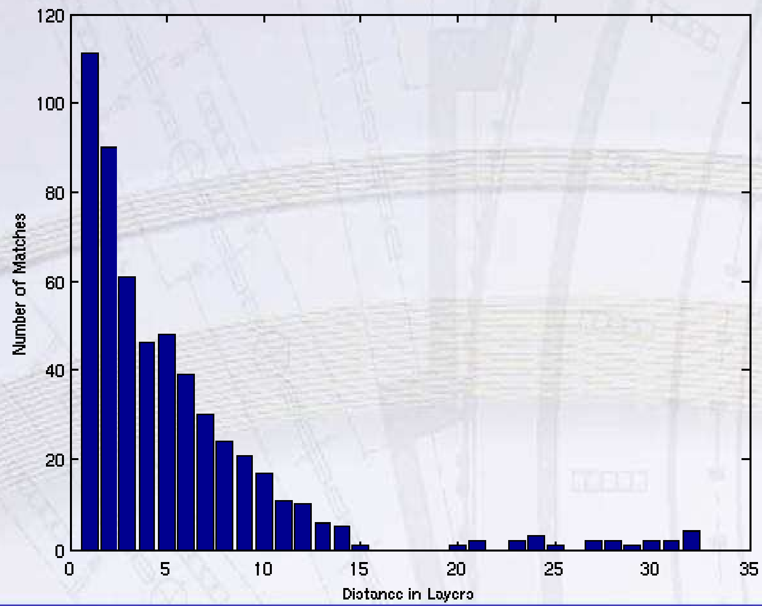
- These are constructed using only grains with a match above and/or below

Center of mass grain map

Resistor: Compare different layers

- The grains found in different layers are compared based on their orientations and refined positions in order to try to locate the same grain in different layers.

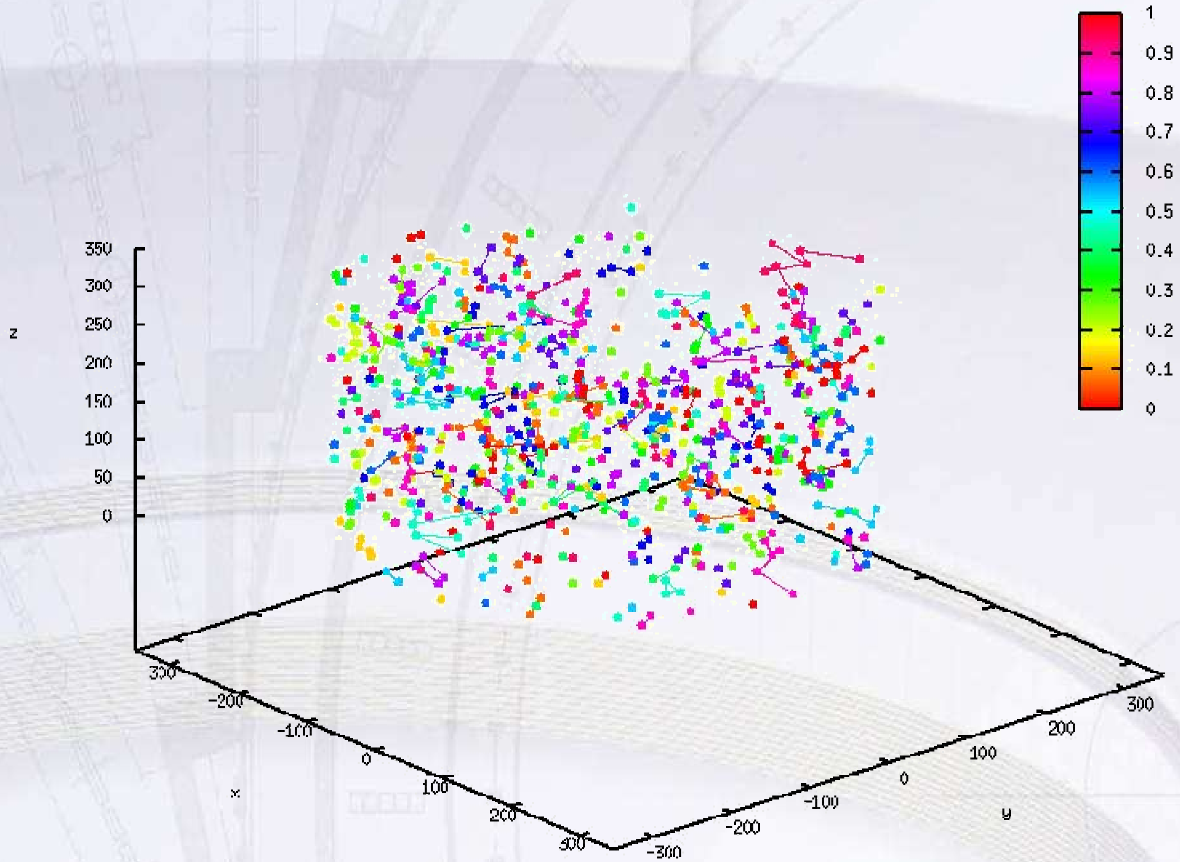
- The results in this case are quite complete and unique: most grains match only one other grain in the next layer with precision <0.3º and 50 µm.



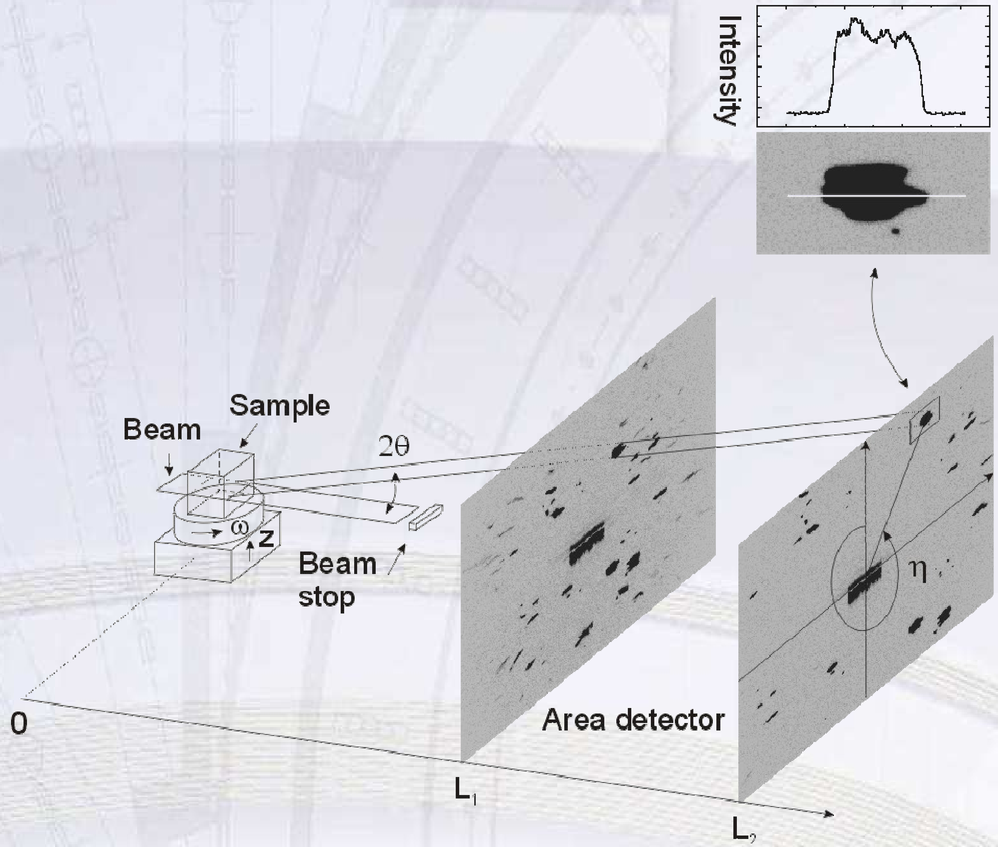
This can also give a sort of grain size distribution in z.

Center of mass grain map

Combine layers to 3D center of mass grain map



Reconstruction/Tracking

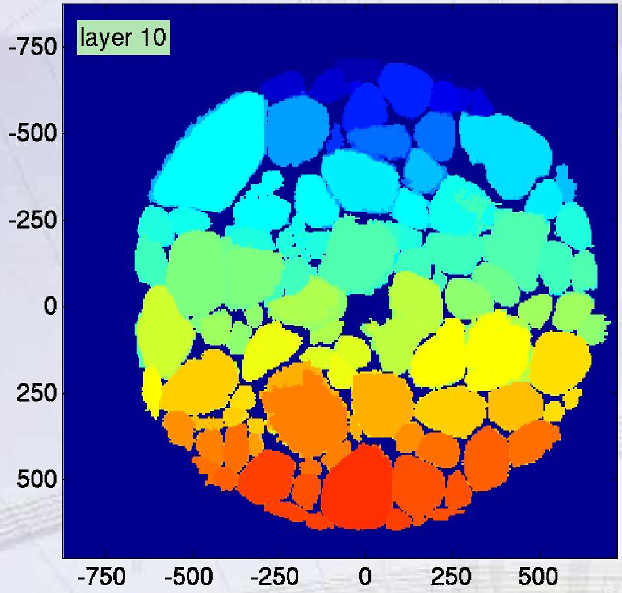


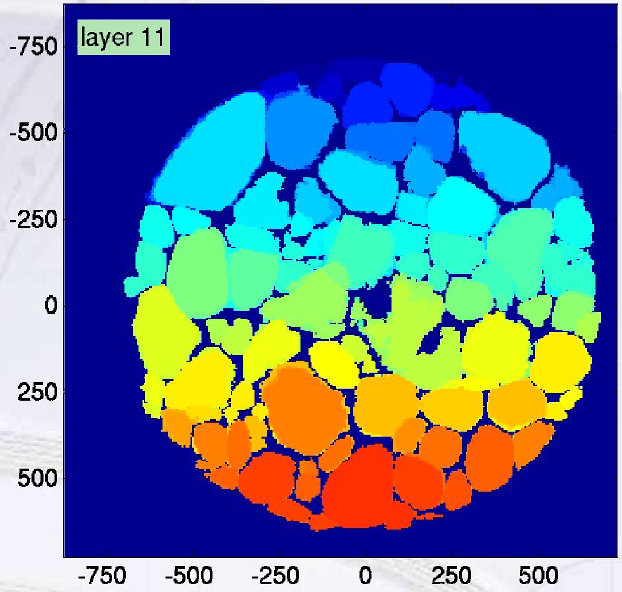
Reconstruction of grain maps by back projection and algebraic reconstruction

GrainSweeper

- Define sample grid
- Scan orientation space
- Forward projections
- Grain completeness
- Connectivity search in sample grid
- Reconstruction of grain

Layer by layer

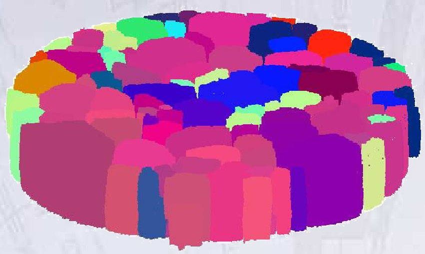




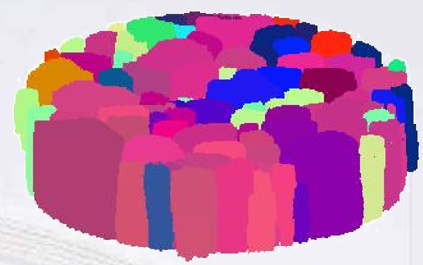
Merge layers

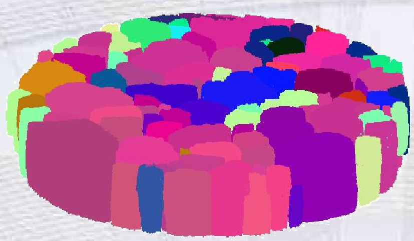
- 3D connectivity search
- Merge on orientation and position

Layer #10



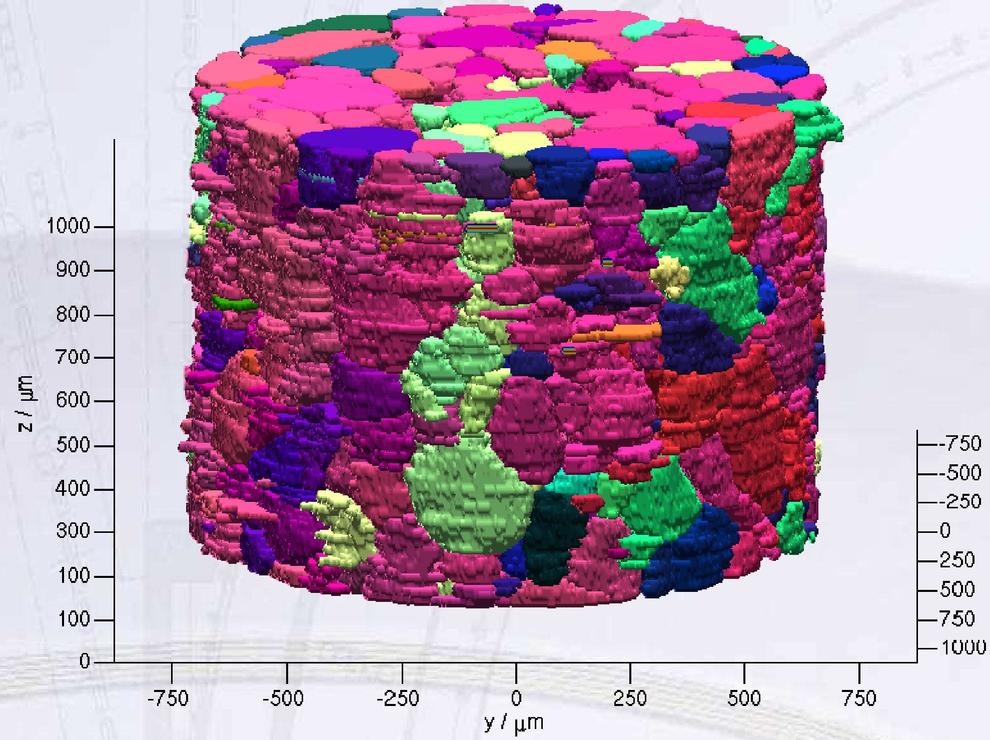
Combined layer #11-12





Layer #11

Results



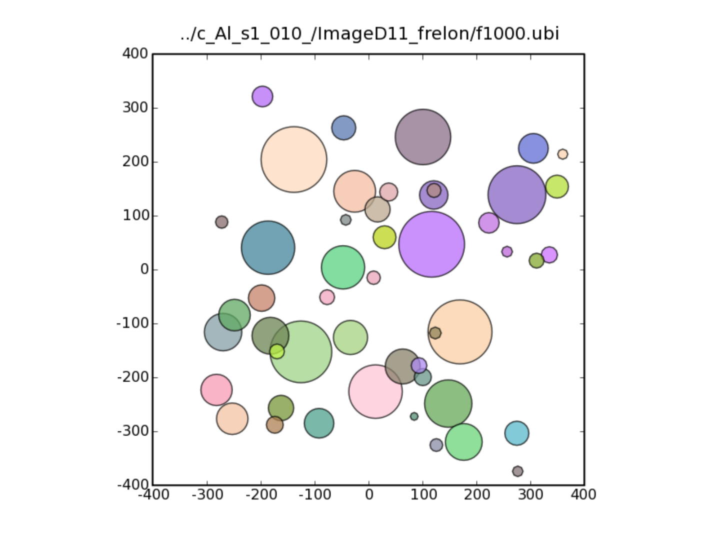
- Grain Growth with C.E. Krill et al., Ulm University
- 1.4 mm diameter
- 1 mm height
- 100 layer with 10 µm height beam
- More than 1000 grains

Standards

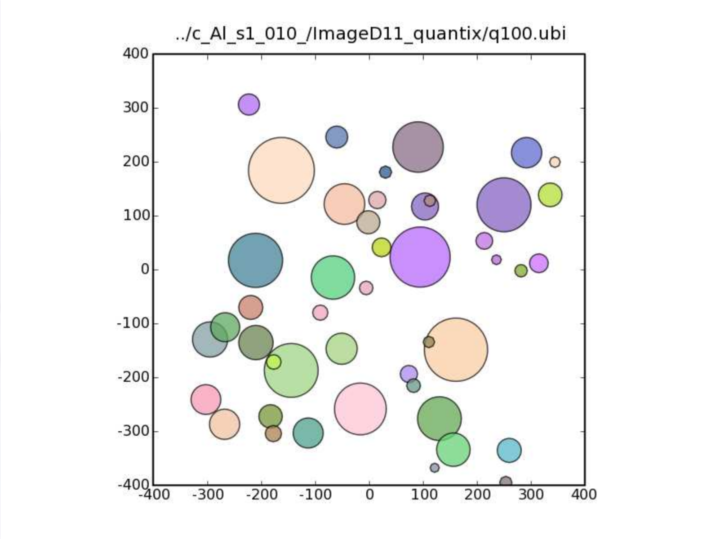
- Within 24 hours of starting the setup it was possible to map 600 μm in height of a sample.
- Started setup at ~16.00
- 4 hours after at ~22.30 the first data was taken
- ~9.00 next morning data measurement was stopped
- This gives a center of mass grain map for each layer
- 3D grain reconstruction

Center of mass

Frelon4m



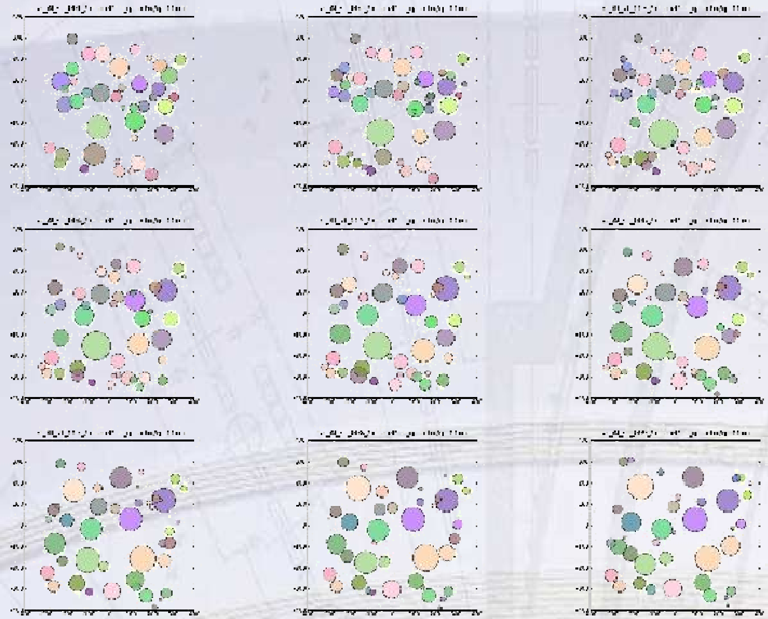
quantix

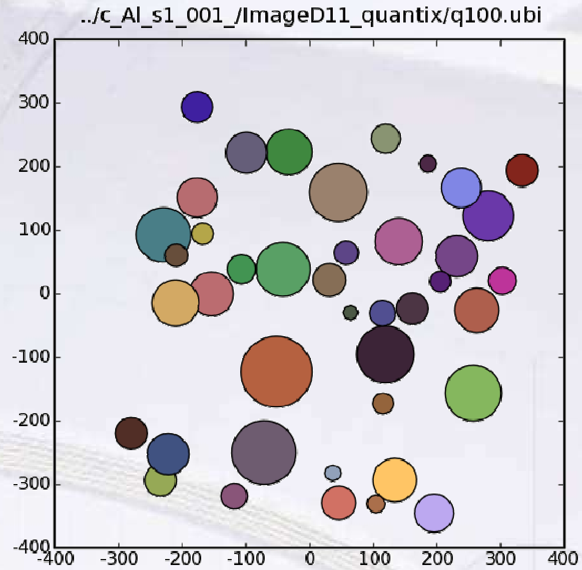


+ A higher resolution on the quantix provides a more precise center of mass position for the grains.
- Inefficient phosphor screen on quantix leads to higher detection limit

TotalCryst

Center of mass

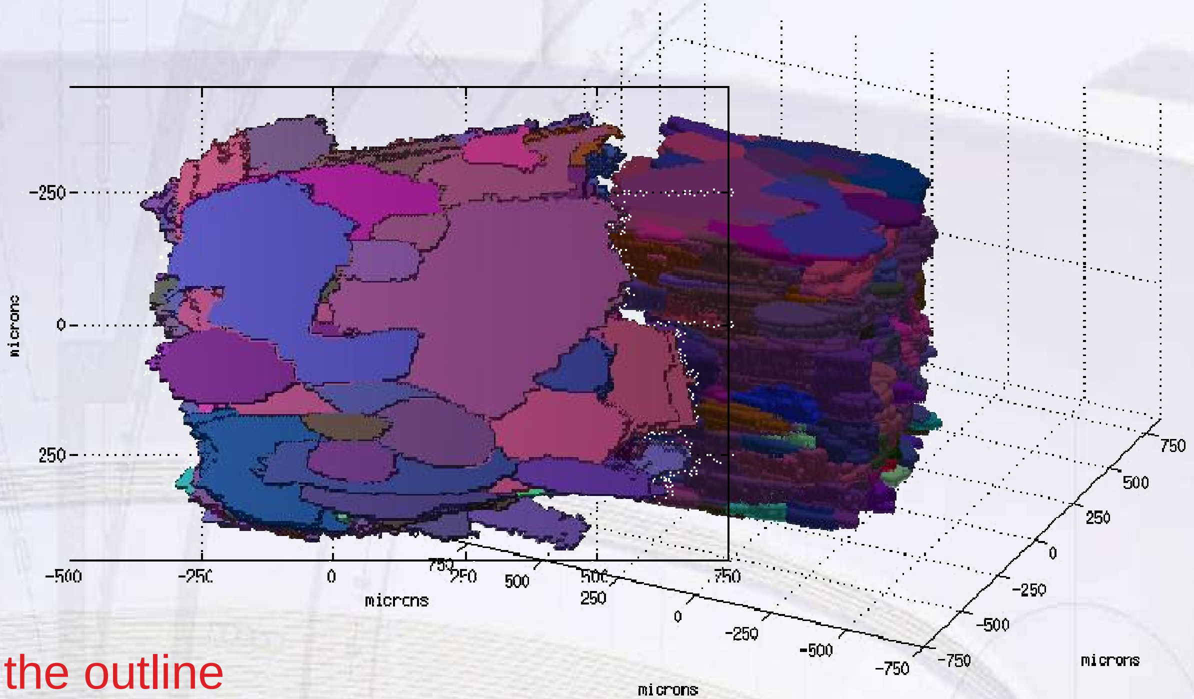




Layer 1 to 9 of Al (AA1050)

Reconstruction

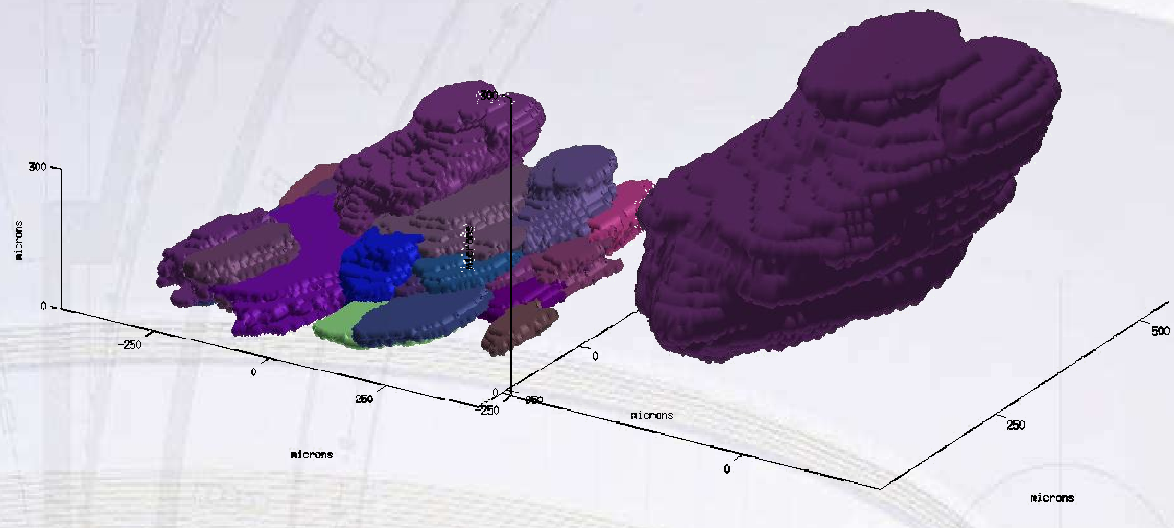
Top view   Side view



The grains for the outline of the sample

Reconstruction

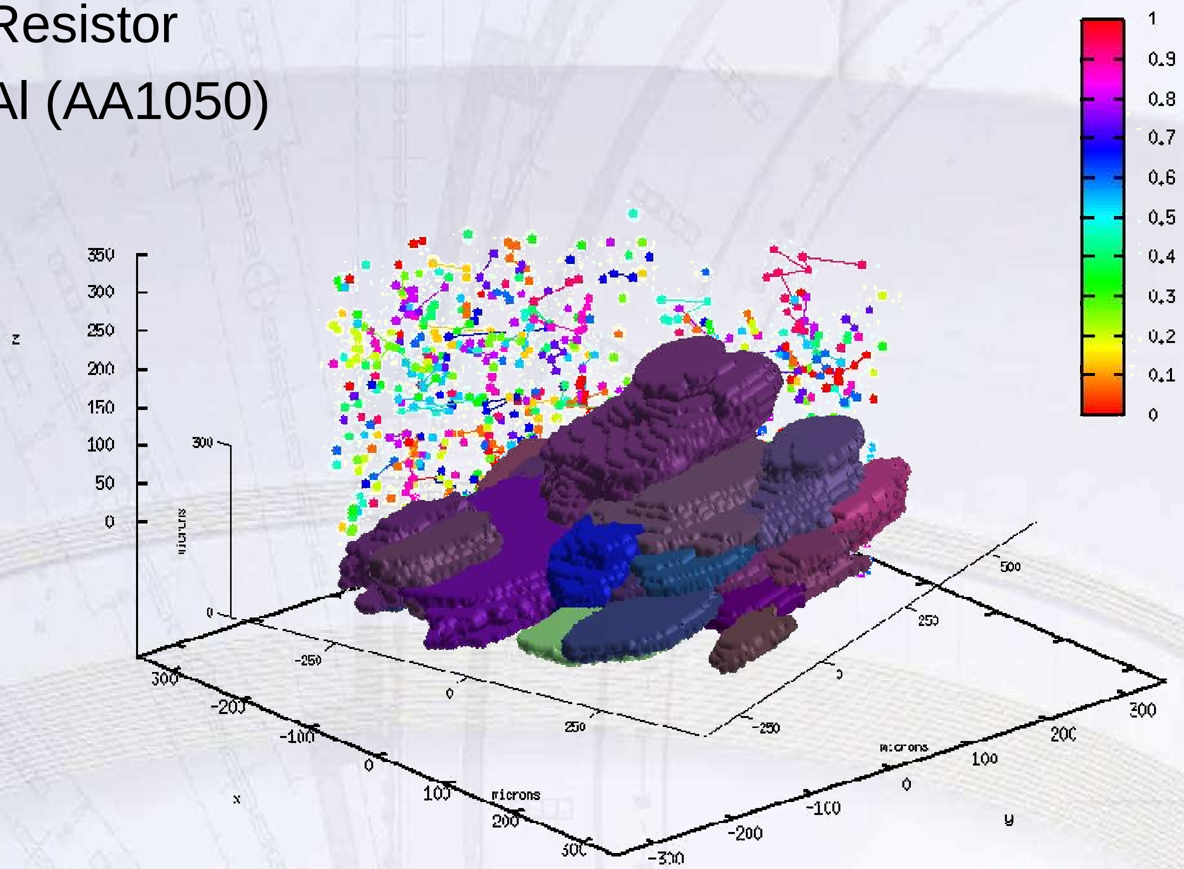
40 grains Grain #32



Euler angles: 4.1974 85.1124 100.1058
Size: 61900 voxels (61900x5x5x10 μm^3)
Height 31 layers

Projects

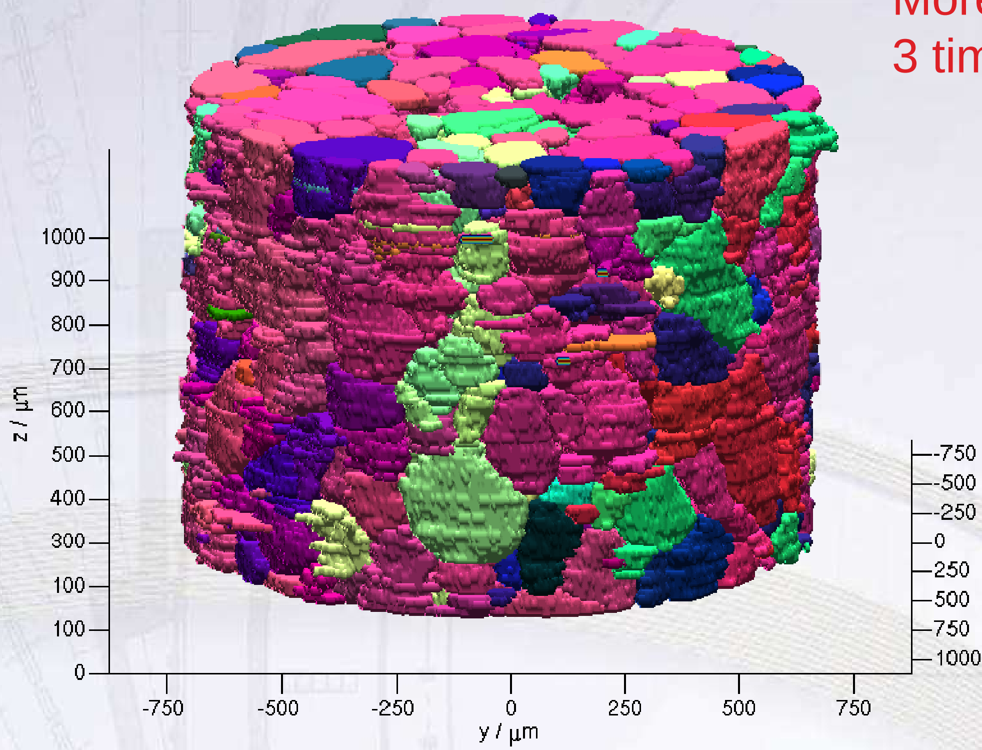
- ID11 grain mapping of different samples
  - Resistor
  - Al (AA1050)



Projects

AlMg

- Grain Growth - C.E. Krill, Ulm University

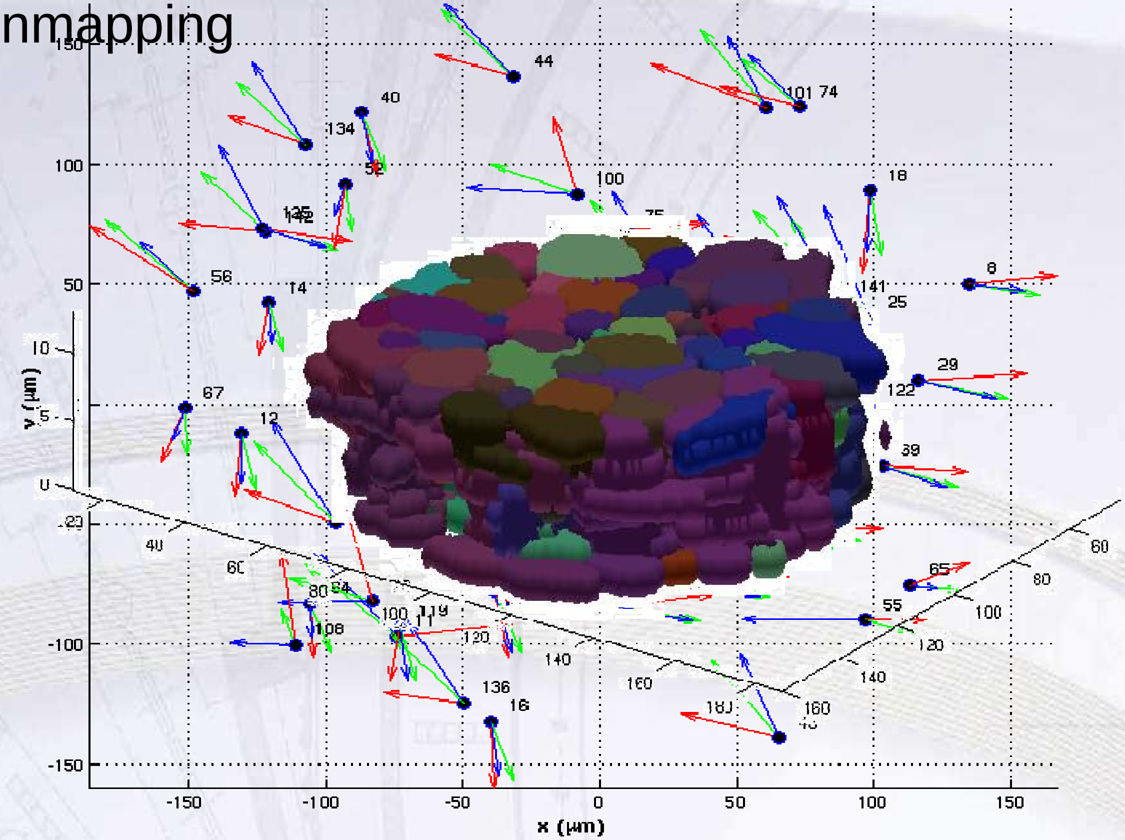


More than 1000 grains
3 time steps

Projects

Gum metal from Toyota

- Grain rotations and grain break up
- Grainmapping



Projects

- ID11 grain mapping of different samples
  - Resistor
  - Al (AA1050)
  - Sucrose (part of the TotalCryst)
- AlMg
  - Grain Growth - C.E. Krill, Ulm University
- Gum metal from Toyota
  - Grain rotations and grain break up - Yoshiharu Hiriose
- Steel
  - Phase transitions – S.E. Offerman, TUDelft
- NaCl
  - Grain growth - Sandra Piazolo, Stockholm University

Summery

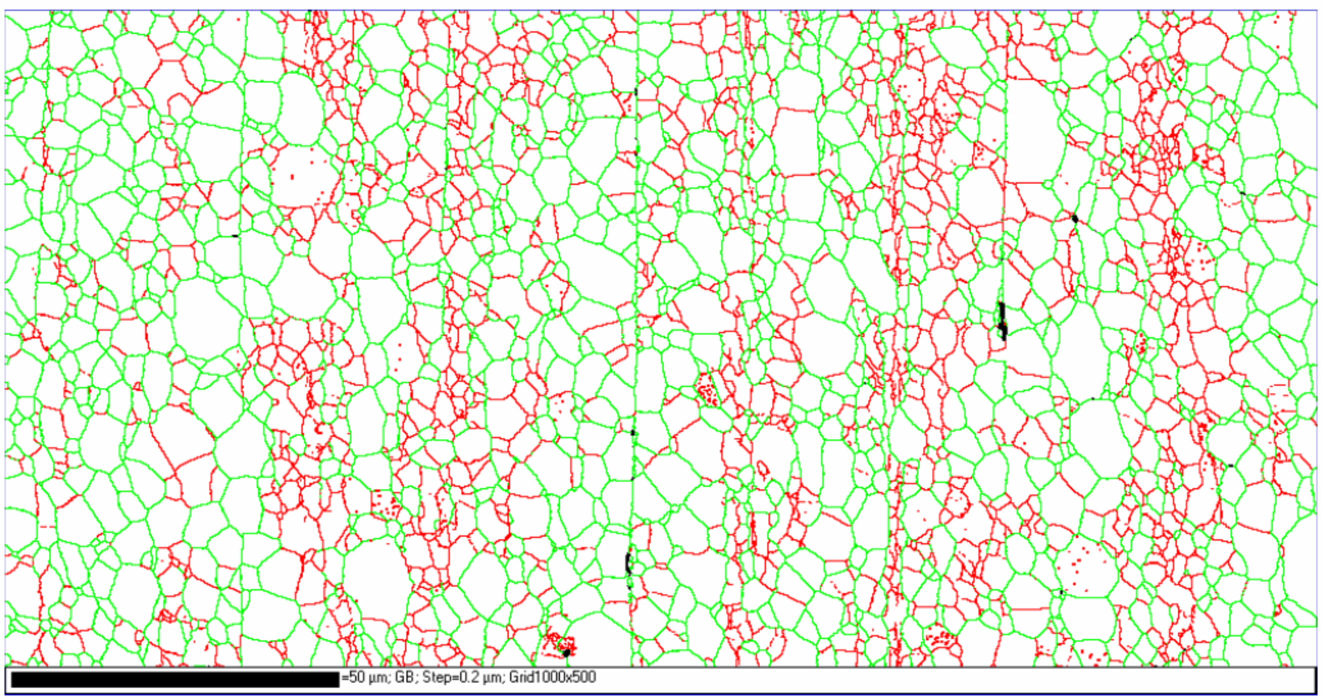
- Different grain mapping depend on needs
- Center of mass grain maps can be fast
- Grain boundary map for more than 1000 grains reconstructed
- 3D detector combining farfield and nearfield

Towards nano

What about smaller sizes, like a few microns?

One solution could be pointscanning

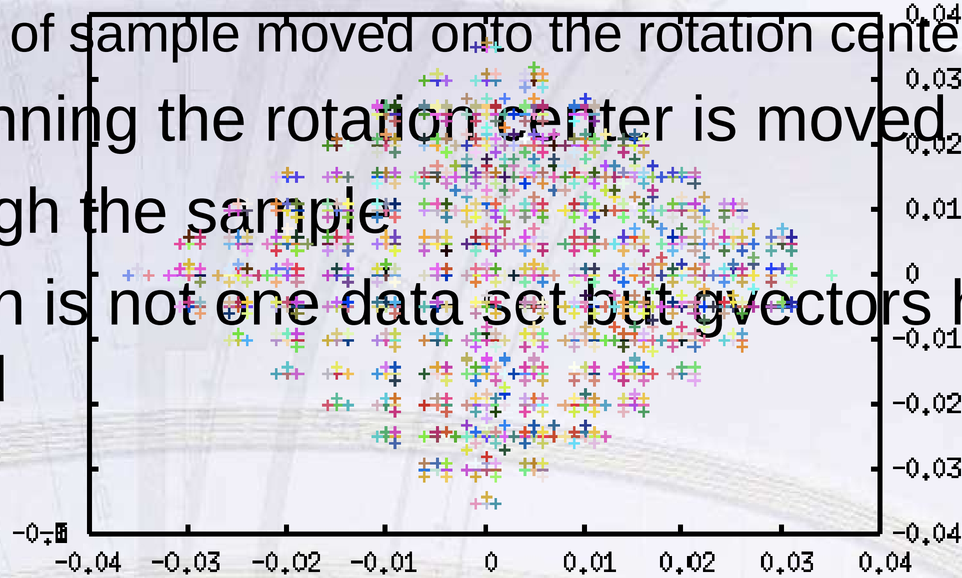
4N-Al_6cARB_Ann_EBSD



175°C 6h+250°C 0.5h

PointScanning

- Normally the beam illuminates the interesting area of the sample.
    - New area of sample moved onto the rotation center
- In pointscanning the rotation center is moved
- Lines through the sample
- One rotation is not one data set but gvectors has to be recombined



Can be used with nano sized beam

Beamsize 5 mµ x 5 mµ

Future

- High speed CMS mapping (few minuts each layer)
- Going from ‘layer to layer’ to box beam (100 µm)
- Deformed 3D mapping
  - Software (new version of GrainSweeper)
  - High resolution 3D detector
- Micron and submicron mapping
  - Pointscanning with submicron beam

Collaborators

- ID11 / ESRF
  - Gavin Vaughan
  - Jon Wright
  - Aleksei Bytchkov
  - Caroline Curfs
  - Henri Gleyzolle
  - Jean-Michel Reynal
  - Andy Götz
  - Gaëlle Suchet
  - Jean Michel Chaize
  - Michel Rossat
  - Denis Van Brussel
- Totalcryst team at Risø
  - Søren Schmidt
  - Henning F. Poulsen
  - Henning Osholm Sørensen
- Ulm University
  - Carl E. Krill
- Toyota
  - Yoshiharu Hiriose
- TUDelft
  - Sven E. Offerman
- Stockholm University
  - Sandra Piazolo