

A Light for Science



European Synchrotron Radiation Facility



3D grain mapping of polycrystals

Carsten Gundlach

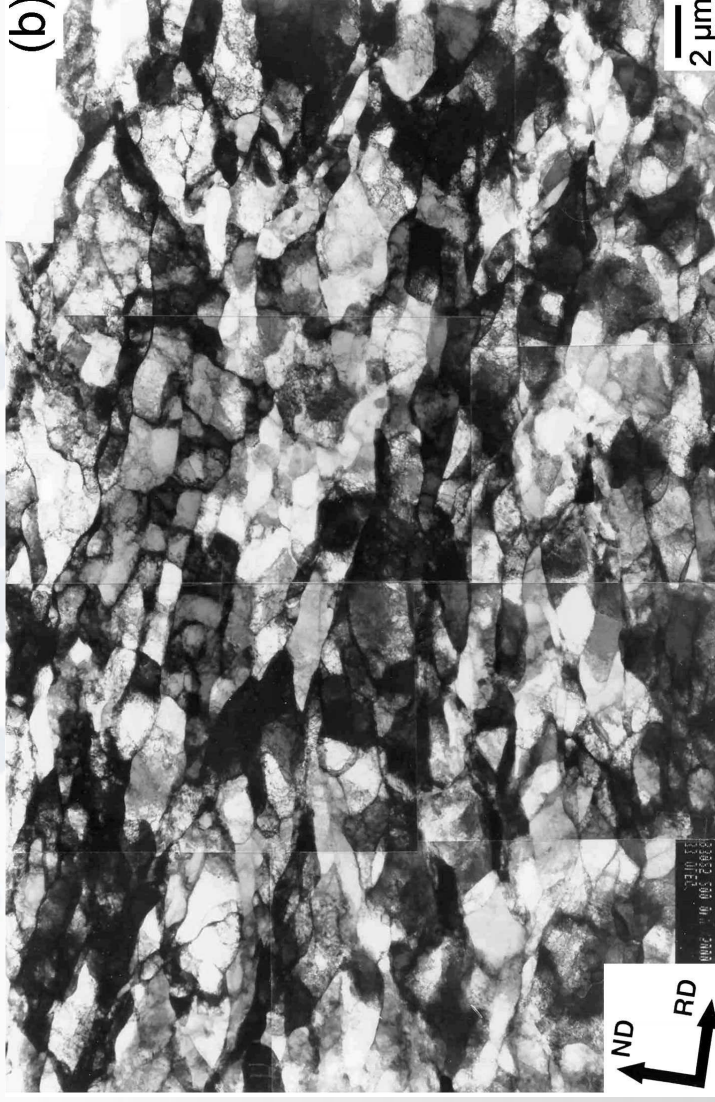
Outline

- Introduction
- Center of mass grain mapping
- 3D grain boundary mapping
- Grid computing - Age of Enlightenment?

Introduction

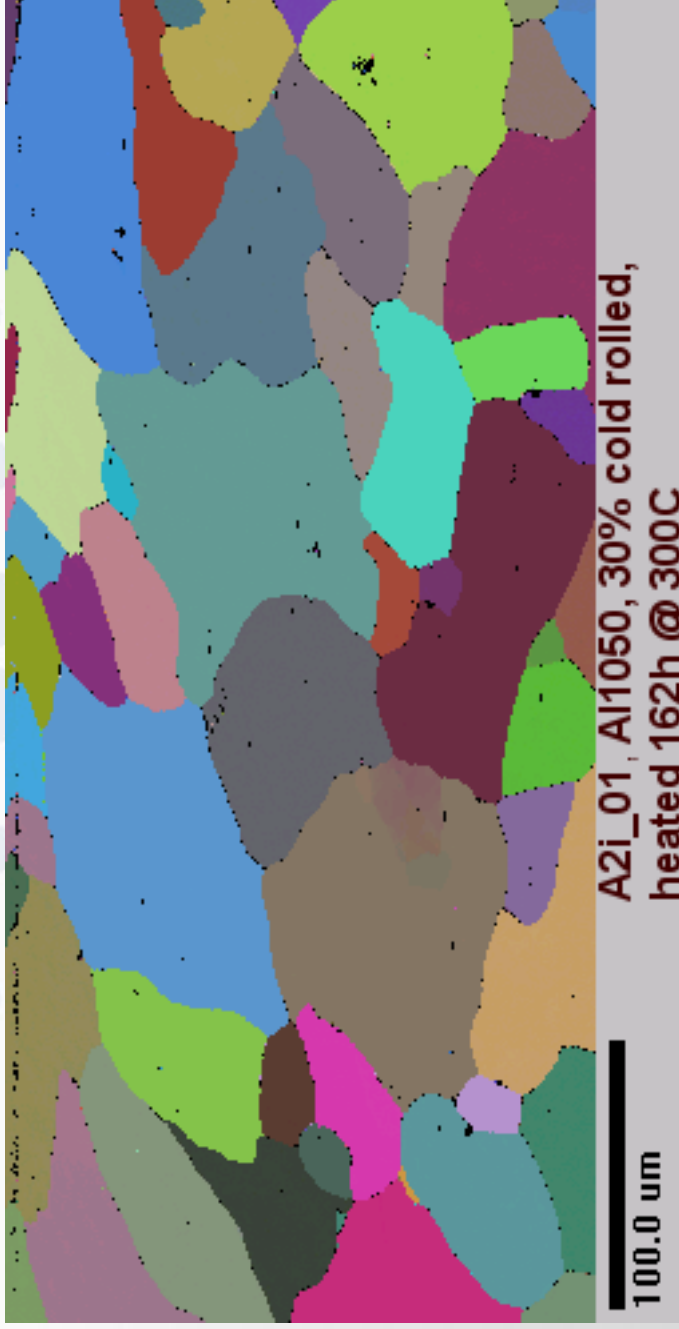
- ID11 – Multi purpose beamline based on diffraction
- Metals as crystals – grains as domains
- Metals – microstructure – properties - design

TEM



Transmission through $\sim 1 \mu\text{m}$ sample

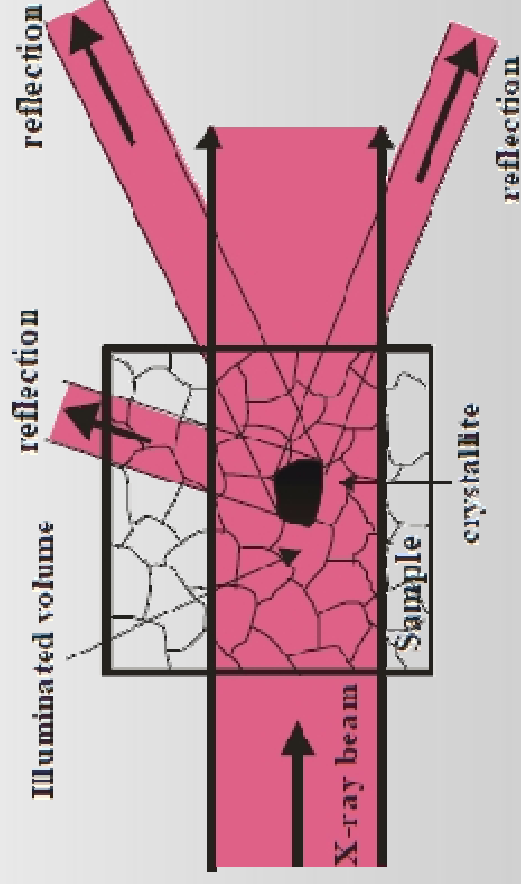
EBSD



- 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



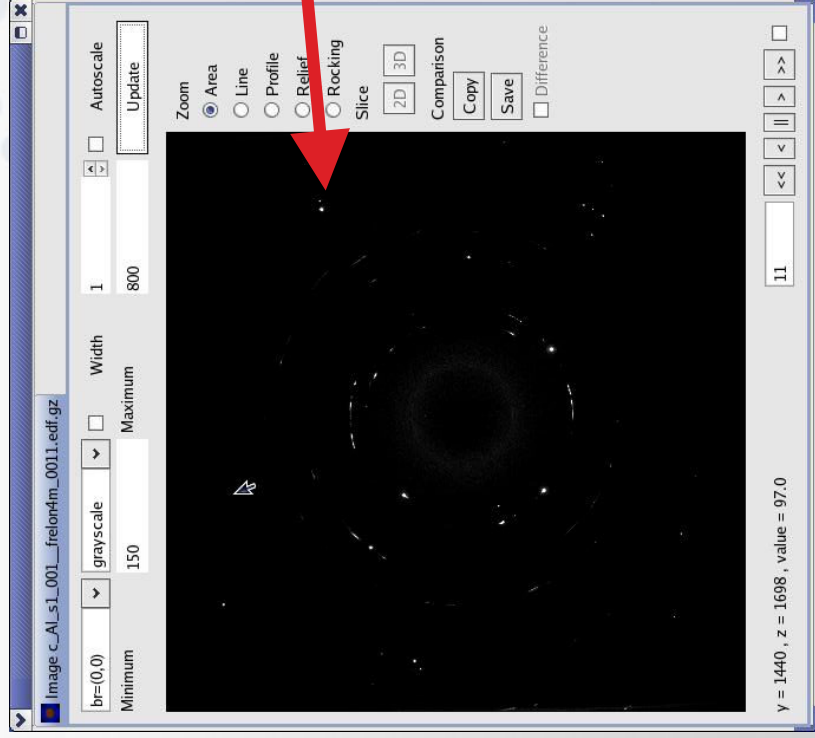
Grain positions, orientations, lattice parameters all simultaneously refined from multi-crystal data

Experimental setup

- Optics
 - Focusing 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
- Quantix detector

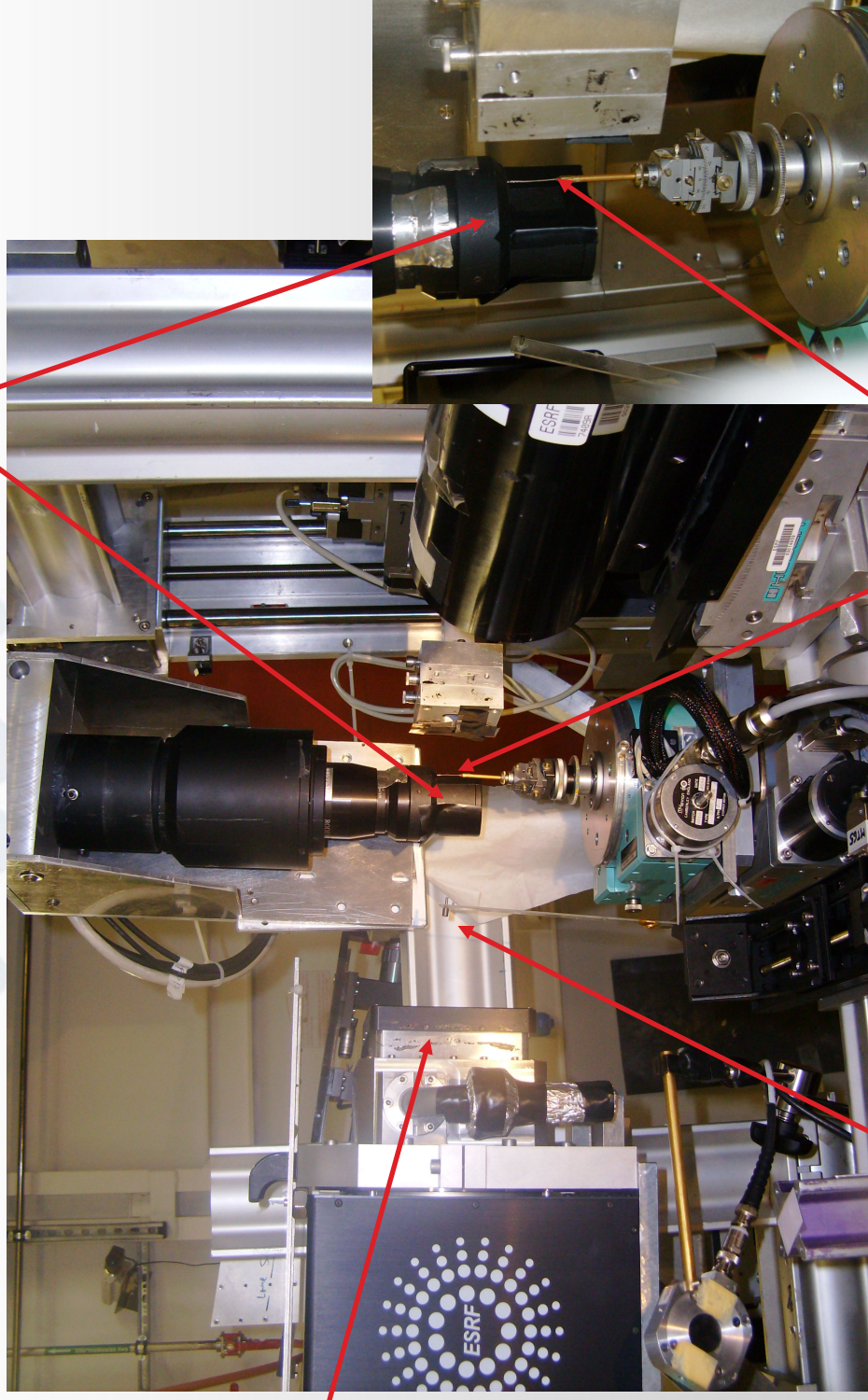


2048x2048 pixels
102.4x102.4 mm
8.1 Mb



1536x1024 pixels
~6.6x4.4 mm
3.1 Mb

3D detector



Frelon detector

Quantix detector

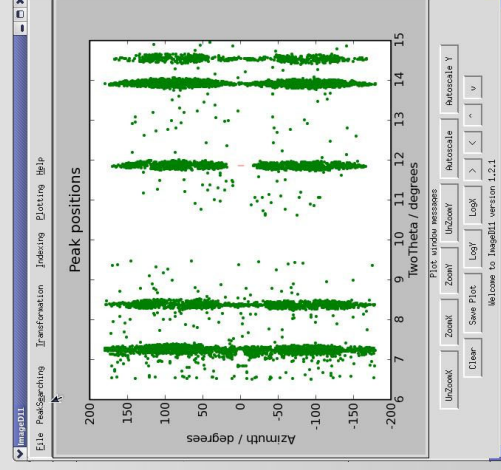
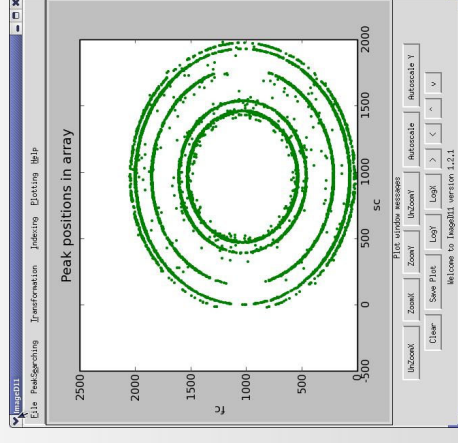
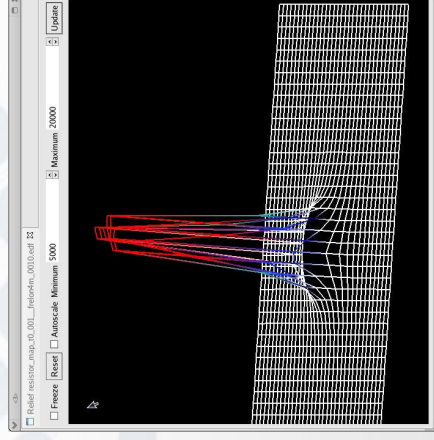
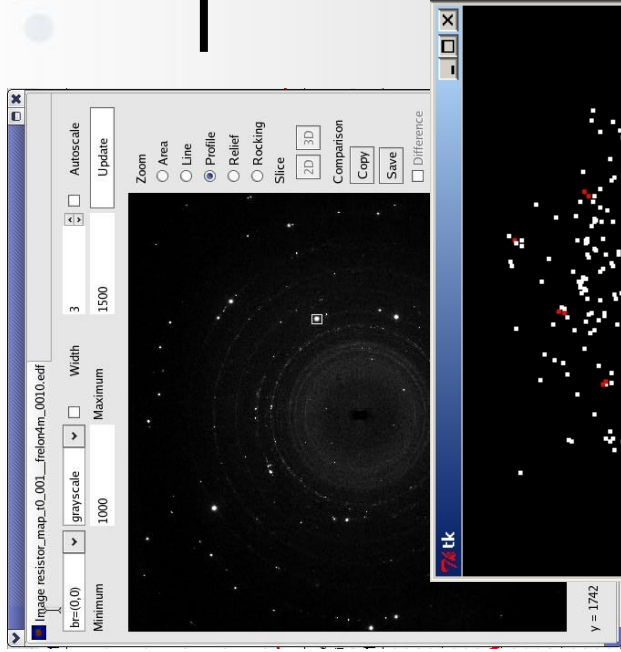
Beamstop

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

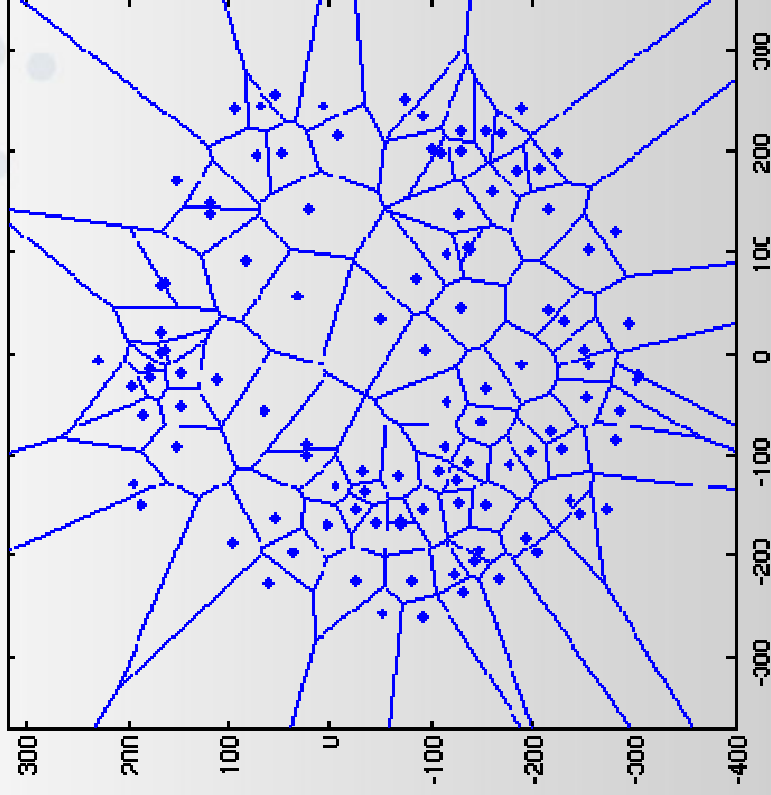
Center of mass analysis



Center of mass grain map

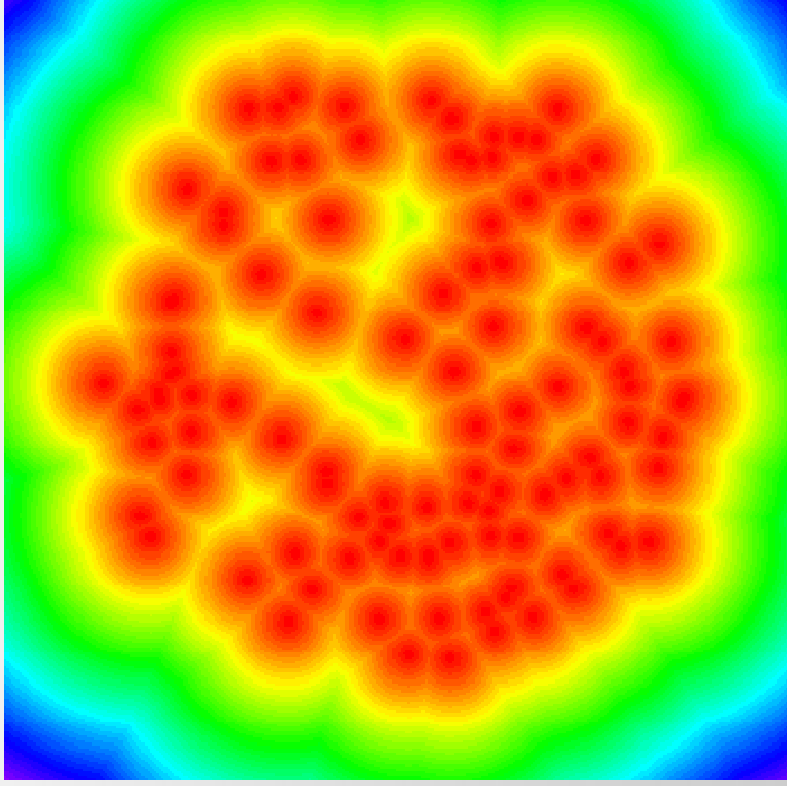
Grain positions, orientations, lattice parameters all simultaneously refined from multi-crystal data

“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

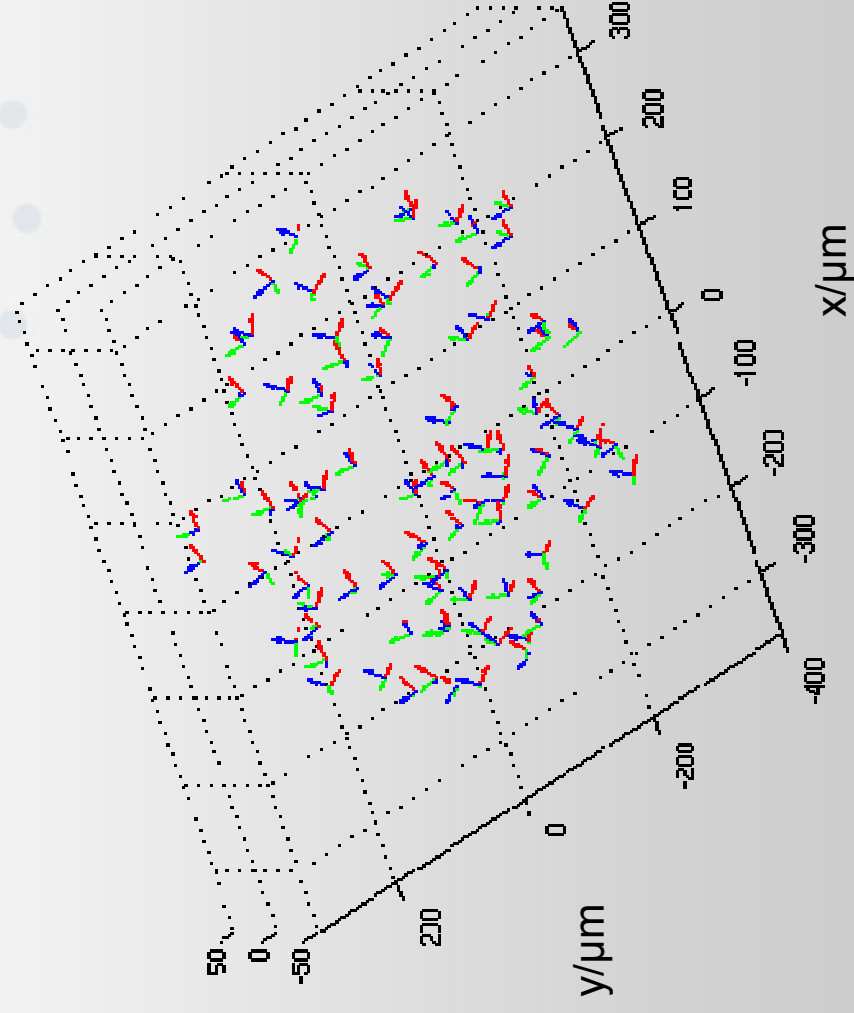
Better representation?



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

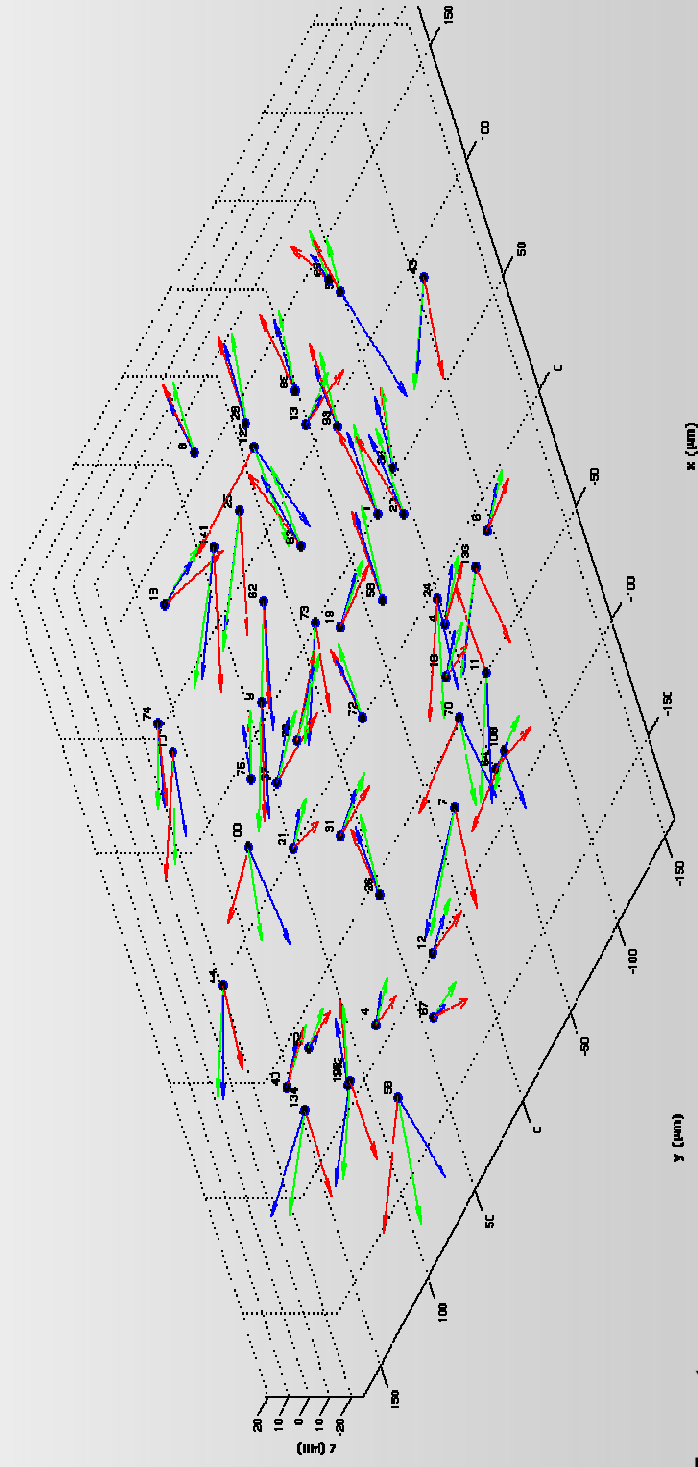


The axis are placed at the crystal centre.

Center of mass grain map

Rotations after each step

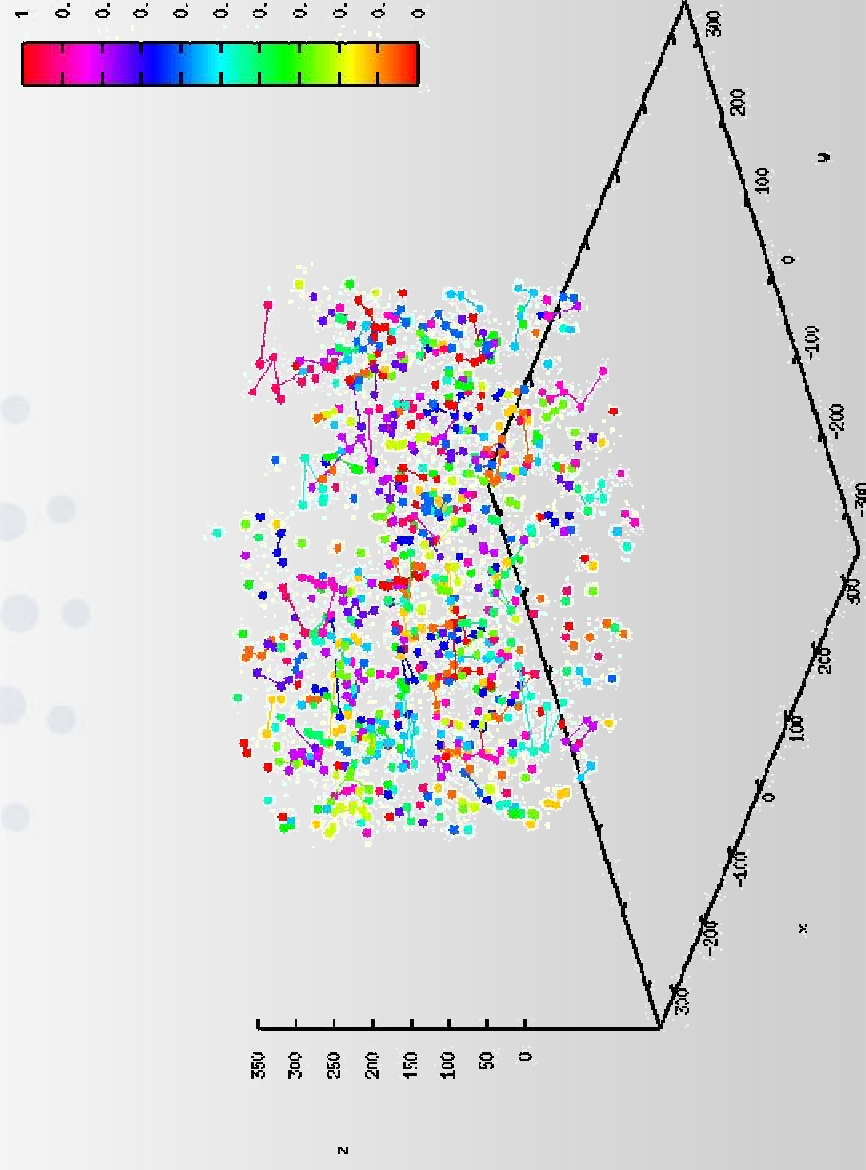
- Depicted are the Rodrigues vectors of subsequent rotations after straining a sample



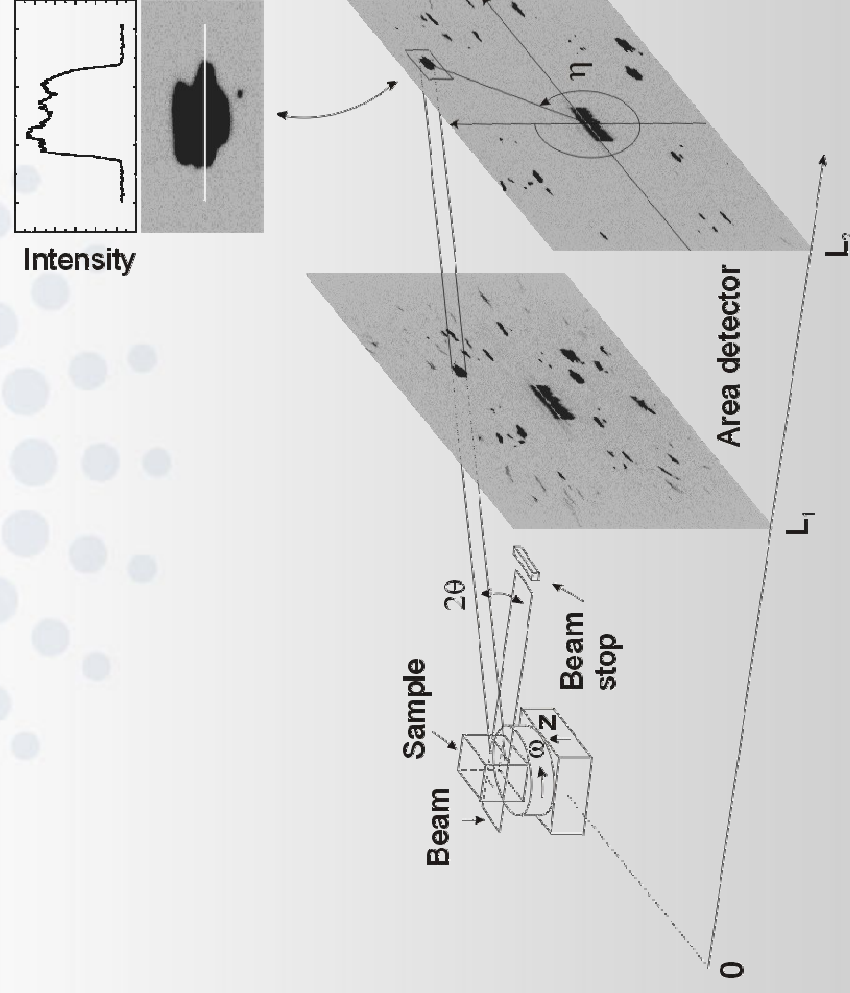
Project with Toyota

Center of mass grain map

Combine layers to 3D center of mass grain map



Reconstruction/Tracking

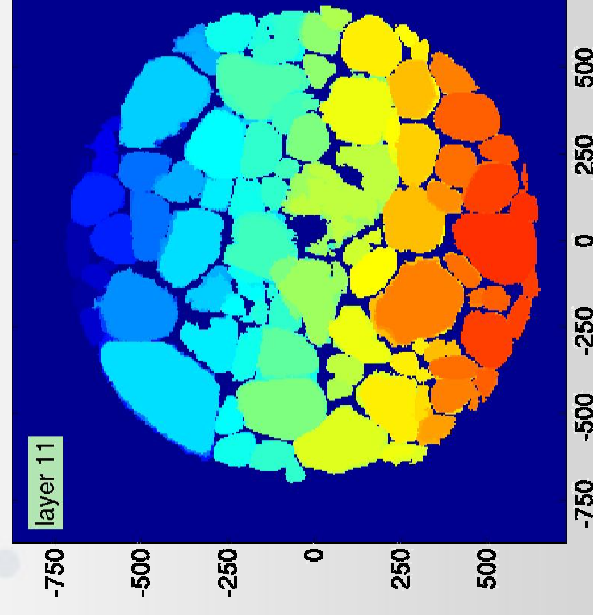
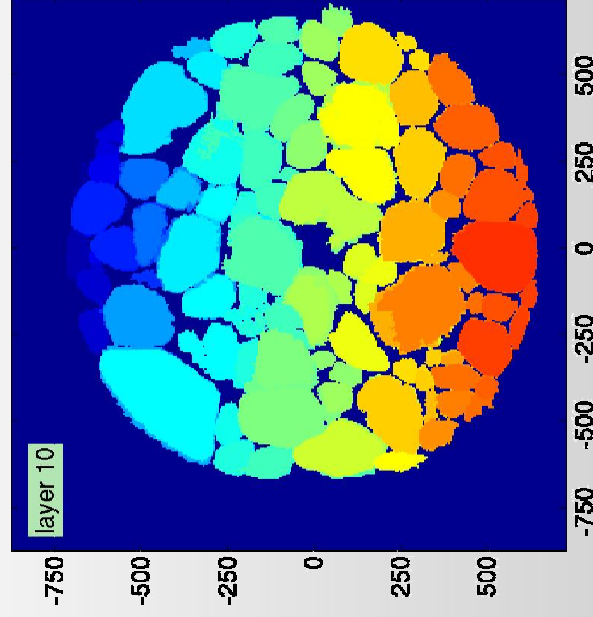


Reconstruction of grain maps by back projection and algebraic reconstruction

GrainSweeper

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

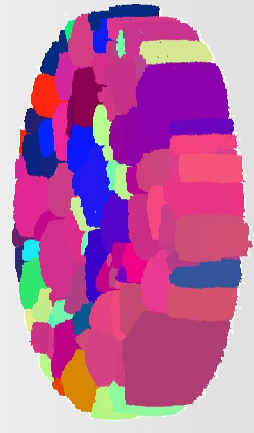
Layer by layer



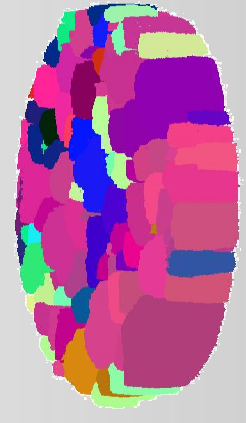
Merge layers

- 3D connectivity search
- Merge on orientation and position

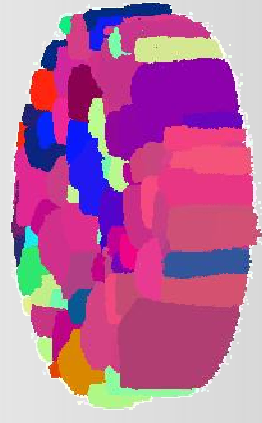
Layer #10



Layer #11



Combined layer #11-12

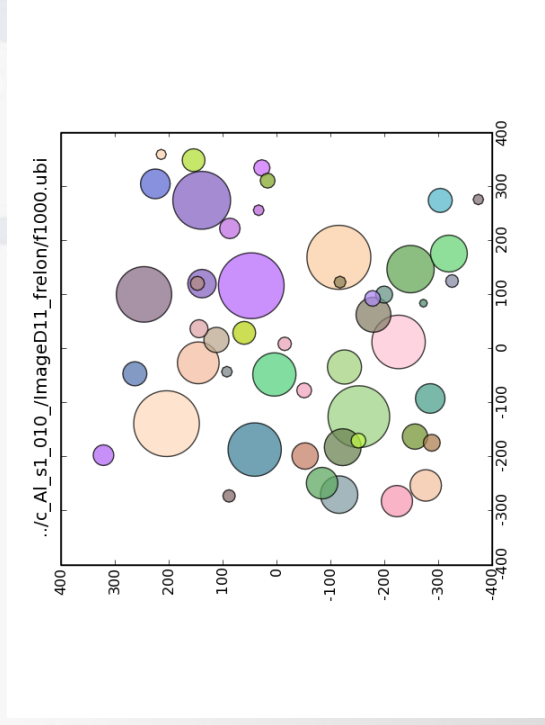


Standards

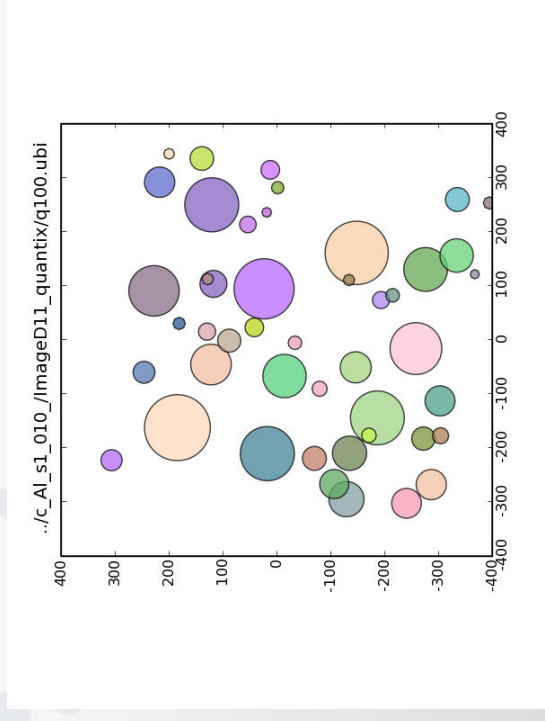
- Within 24 hours of starting the setup it was possible to map 600 μm in height of a sample.
- Started setup at ~16.00
- 4 hours after at ~22.30 the first data was taken
- ~9.00 next morning data measurement was stopped
- This gives a center of mass grain map for each layer
- 3D grain reconstruction

Center of mass

Frelon4m

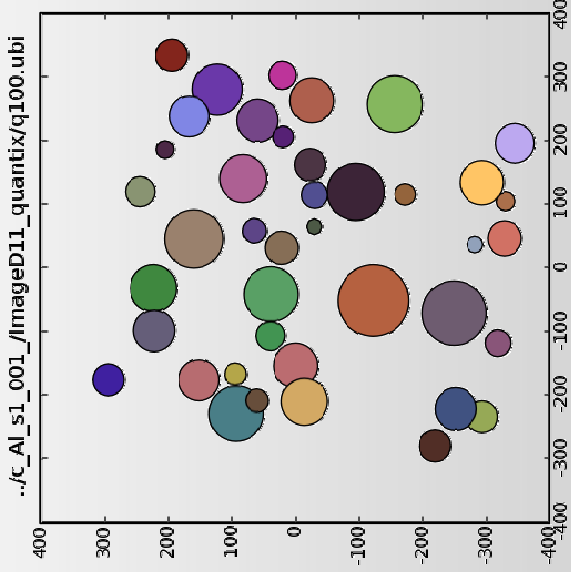
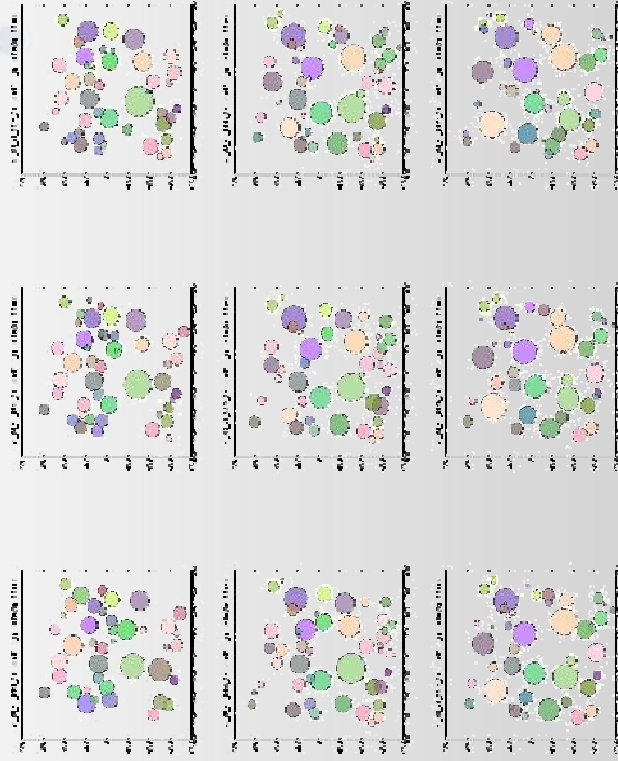


quantix



- + 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

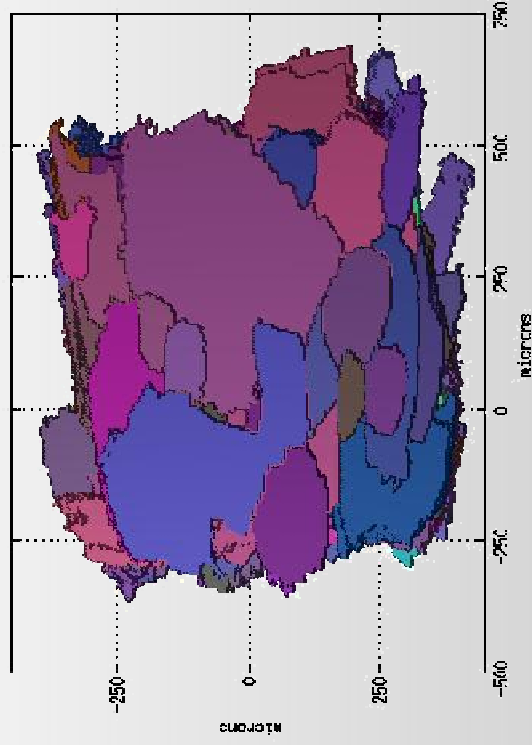
Center of mass



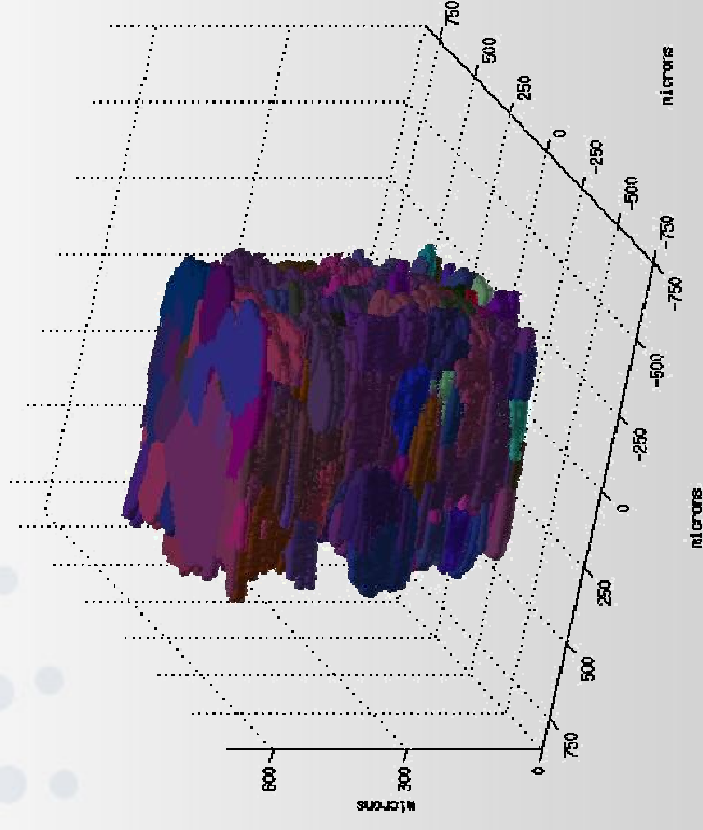
Layer 1 to 9 of AI (AA1050)

Reconstruction

Top view



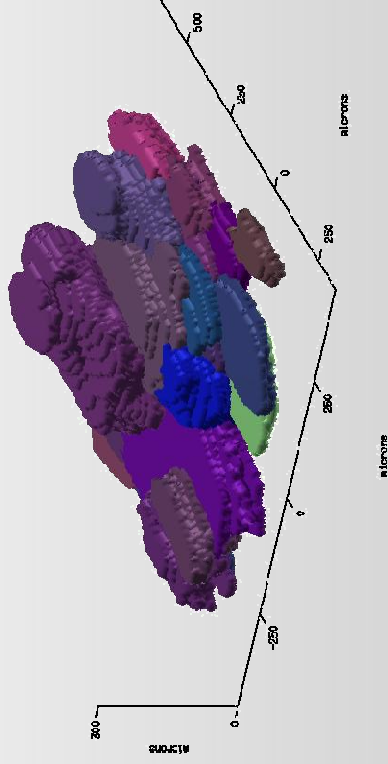
Side view



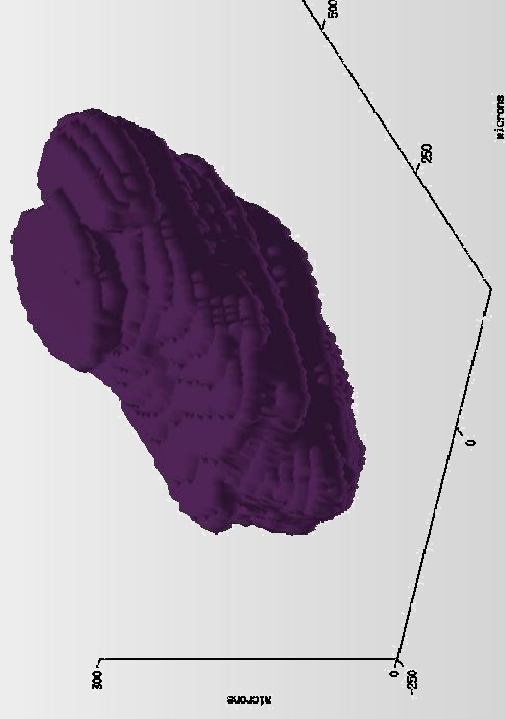
The grains for the outline
of the sample

Reconstruction

40 grains



Grain #32



Euler angles: 4.1974 85.1124 100.1058
Size: 61900 voxels (61900x5x10 μm^3)
Height 31 layers

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 Piazzolo, Stockholm University

Computer power

- Image based
 - Image processing (peaksearching, corrections)
 - 3D Reconstruction
 - Normally 180 images with 0.5 degree rotation
 - ~ 250 Mb in images
 - The minimum is 90 images ~125 Mb
 - Ideally 360 images ~ 500 Mb
- Text based
 - CMS mapping
 - Grain refinement
 - Parameters refinement

Computer power

\$!d condor_status_submitters_vrview.xzit 26 2007-06-07 10:37:32z klotz \$ WDK
 Last Update: 17:17:37

Name	Scheduler	Running Jobs	Flocked Jobs	Idle Jobs	Held Jobs	Max Jobs Running
ma505@esrf.fr	coral1.esrf.fr	0	0	2	0	500
collivier@esrf.fr	coral21.esrf.fr	0	0	0	85	500
collivier@esrf.fr	coral24.esrf.fr	0	0	0	27	500
ma508@esrf.fr	coral25.esrf.fr	38	0	0	0	500
collivier@esrf.fr	coral28.esrf.fr	0	0	0	1	500
collivier@esrf.fr	coral29.esrf.fr	0	0	0	2	500
jaferman@esrf.fr	coral29.esrf.fr	0	0	4	0	500
jaferman@esrf.fr	coral30.esrf.fr	0	0	20	0	500
md360@esrf.fr	coral31.esrf.fr	0	0	0	20	500
collivier@esrf.fr	coral32.esrf.fr	0	0	0	2	500
md360@esrf.fr	coral32.esrf.fr	0	0	0	20	500
gundlach@esrf.fr	coral34.esrf.fr	211	0	2246	0	500
tafforea@esrf.fr	coral36.esrf.fr	0	0	1	0	500
lauridse@esrf.fr	coral37.esrf.fr	0	0	1	0	500
jaferman@esrf.fr	coral41.esrf.fr	1	0	0	0	500
coan@esrf.fr	coral43.esrf.fr	0	0	1	0	500
jaferman@esrf.fr	coral43.esrf.fr	0	0	3	52	500
lauridse@esrf.fr	coral44.esrf.fr	3	0	0	0	500
collivier@esrf.fr	coral47.esrf.fr	0	0	0	90	500
jaferman@esrf.fr	coral47.esrf.fr	0	0	0	60	500
collivier@esrf.fr	coral53.esrf.fr	0	0	0	1	500
collivier@esrf.fr	coral54.esrf.fr	0	0	0	1	500

26 layers

Computer power

\$fd condor_status_submitters_view.xmlt.26.2007-06-07 10:37:32Z klorz.\$ WDK

Last Update: 17:21:23

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ma595@esrf.fr	coral1.esrf.fr	0	0	2	0	500
colivier@esrf.fr	coral21.esrf.fr	0	0	0	85	500
colivier@esrf.fr	coral24.esrf.fr	0	0	0	27	500
ma508@esrf.fr	coral25.esrf.fr	38	0	0	0	500
colivier@esrf.fr	coral28.esrf.fr	0	0	0	1	500
colivier@esrf.fr	coral29.esrf.fr	0	0	0	2	500
jaferman@esrf.fr	coral29.esrf.fr	0	0	4	0	500
jaferman@esrf.fr	coral30.esrf.fr	0	0	20	0	500
md360@esrf.fr	coral31.esrf.fr	0	0	0	20	500
colivier@esrf.fr	coral32.esrf.fr	0	0	0	2	500
md360@esrf.fr	coral32.esrf.fr	0	0	0	20	500
gundlach@esrf.fr	coral34.esrf.fr	214	1	5216	0	500
tafforea@esrf.fr	coral36.esrf.fr	0	0	1	0	500
lauridse@esrf.fr	coral37.esrf.fr	0	0	1	0	500
jaferman@esrf.fr	coral41.esrf.fr	1	0	0	0	500
coan@esrf.fr	coral43.esrf.fr	1	0	0	0	500
jaferman@esrf.fr	coral43.esrf.fr	0	0	0	0	500
lauridse@esrf.fr	coral44.esrf.fr	3	0	3	52	500
colivier@esrf.fr	coral47.esrf.fr	0	0	0	0	500
jaferman@esrf.fr	coral47.esrf.fr	0	0	0	90	500
colivier@esrf.fr	coral53.esrf.fr	0	0	0	60	500
colivier@esrf.fr	coral54.esrf.fr	0	0	0	1	500
colivier@esrf.fr	coral54.esrf.fr	0	0	0	1	500

53 layers

Computer power

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 Last Update: 17:30:14

Name	Scheduler	Running Jobs	Flocked Jobs	Idle Jobs	Held Jobs	Max Jobs Running
ma505@esrf.fr	coral1.esrf.fr	0	0	2	0	500
colivier@esrf.fr	coral21.esrf.fr	0	0	0	85	500
colivier@esrf.fr	coral24.esrf.fr	0	0	0	27	500
ma508@esrf.fr	coral25.esrf.fr	38	0	0	0	500
colivier@esrf.fr	coral28.esrf.fr	0	0	0	1	500
colivier@esrf.fr	coral29.esrf.fr	0	0	0	2	500
jafernan@esrf.fr	coral29.esrf.fr	0	0	4	0	500
jafernan@esrf.fr	coral30.esrf.fr	0	0	20	0	500
md360@esrf.fr	coral31.esrf.fr	0	0	0	20	500
colivier@esrf.fr	coral32.esrf.fr	0	0	0	2	500
md360@esrf.fr	coral32.esrf.fr	0	0	0	20	500
gundlach@esrf.fr	coral34.esrf.fr	209	0	10480	0	500
tafforea@esrf.fr	coral36.esrf.fr	0	0	1	0	500
lauridse@esrf.fr	coral37.esrf.fr	0	0	1	0	500
jafernan@esrf.fr	coral41.esrf.fr	0	0	0	0	500
coan@esrf.fr	coral43.esrf.fr	0	0	0	52	500
jafernan@esrf.fr	coral43.esrf.fr	0	0	3	52	500
lauridse@esrf.fr	coral44.esrf.fr	3	0	0	0	500
colivier@esrf.fr	coral47.esrf.fr	0	0	0	90	500
jafernan@esrf.fr	coral47.esrf.fr	0	0	0	60	500
colivier@esrf.fr	coral53.esrf.fr	0	0	0	1	500
colivier@esrf.fr	coral54.esrf.fr	0	0	0	1	500

100 layers

Collaborators

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Gaëlle Suchet

Jean Michel Chaize

Michel Rossat

Denis Van Brussel

- Totalcryst

Søren Schmidt

- Ulm University

Carl E. Krill

- Toyota

Yoshiharu Hiriose

- TUDelft

Sven E. Offerman

- Stockholm University

Sandra Piazzolo

