- Total Cryst -

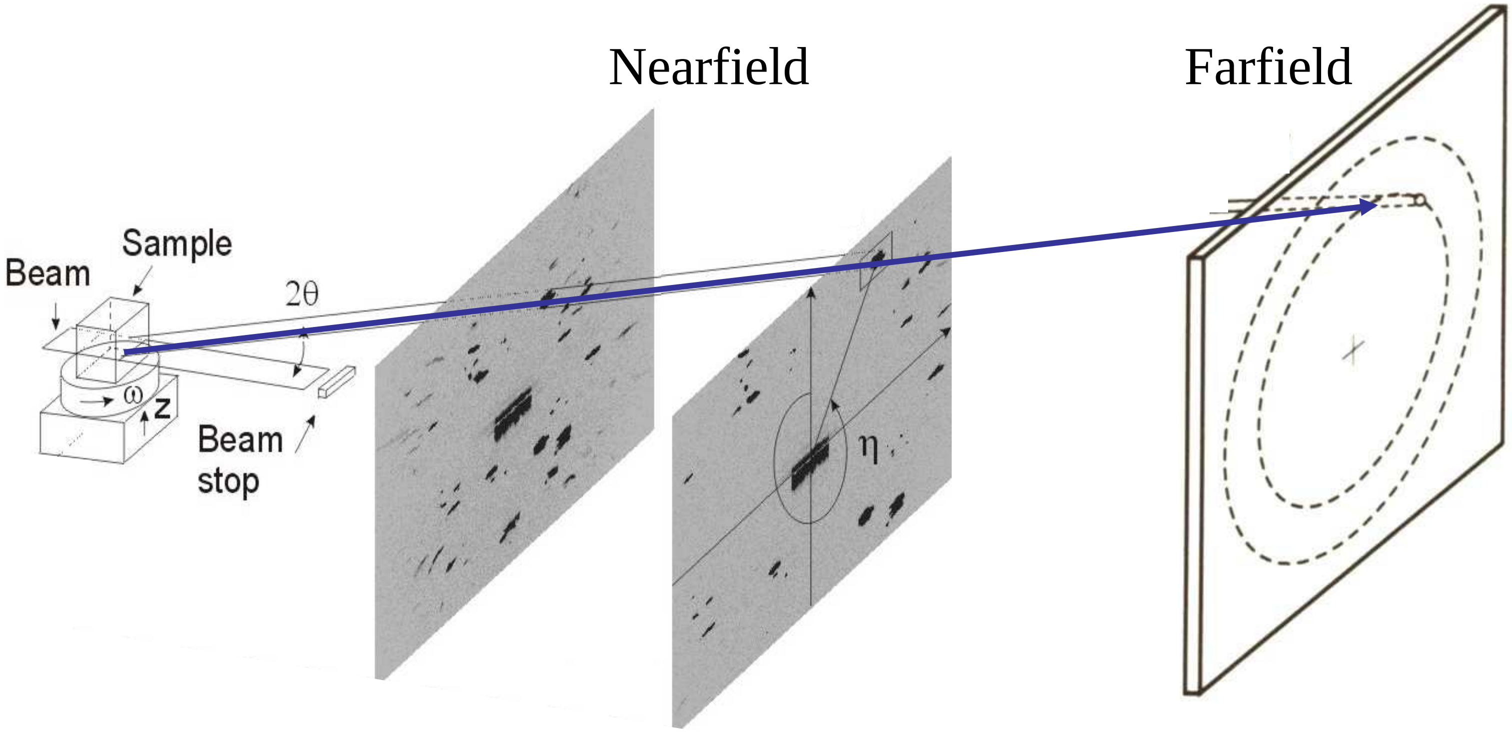
EU NEST/ADVENTURE program: 1/2 2006 – 1/8 2009
www.totalcryst.dk

Multi-grain crystallography:
structure solution and refinement of each crystallite

3D Grain mapping:
position, morphology, orientation & stress-state of each grain

FABLE: Framework for software + GUIs

TotalCryst set-up

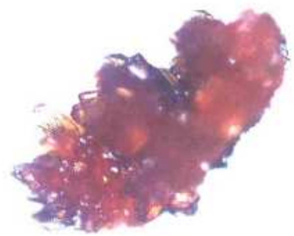


Third road in Crystallograpy

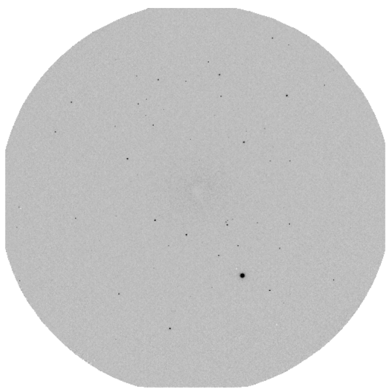
Single Crystal

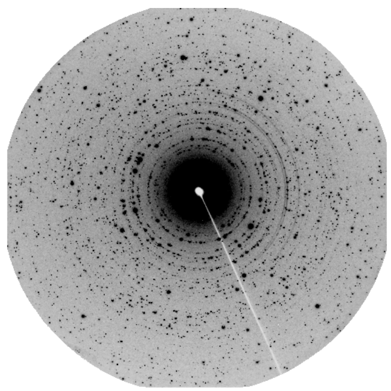
Multicrystal

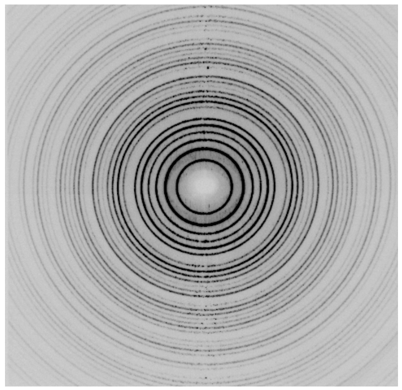
Powder



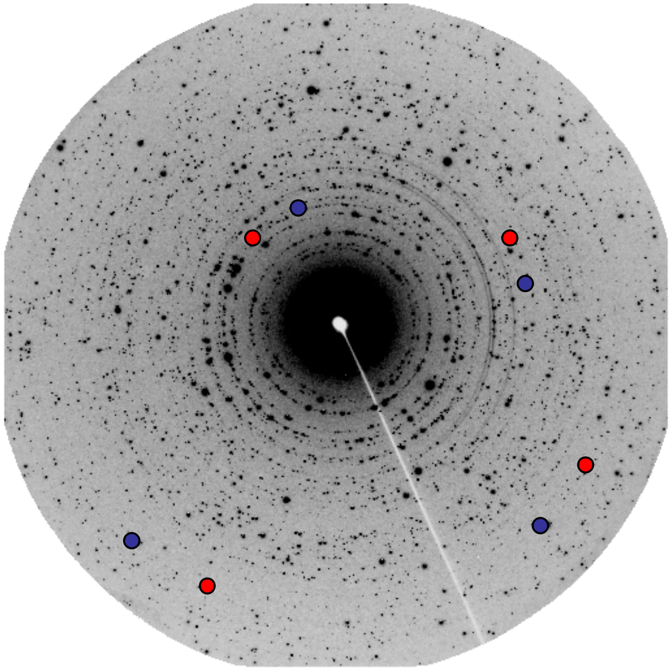
X-ray data:







Multi-grain Crystallography



1. Find spots
2. Indexing based on orientation relations
3. Intensity harvesting
4. Structural solution and refinement using JANA, MOSFLM, …

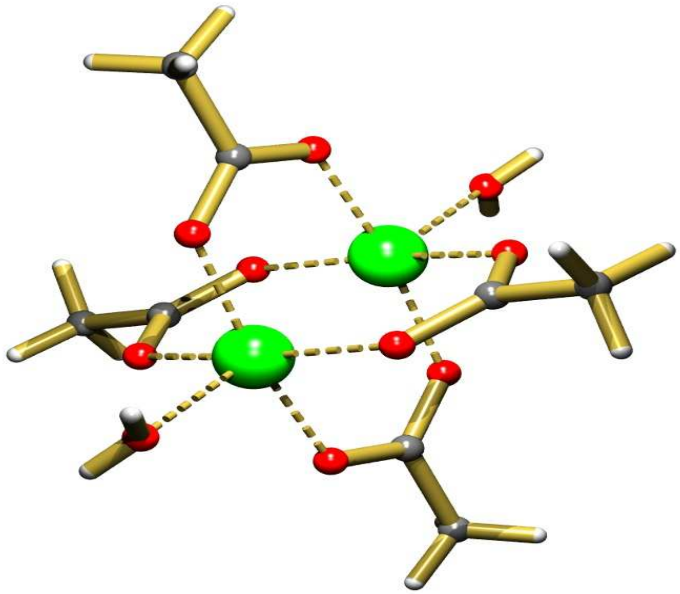
First study

Validation:

$Cu(C_2O_2H_3)_2 \cdot H_2O$.

70 grains of size < 1 micron

Cell ~1400 Å^3 (C2/c)



Result:

Single crystal quality refinement!

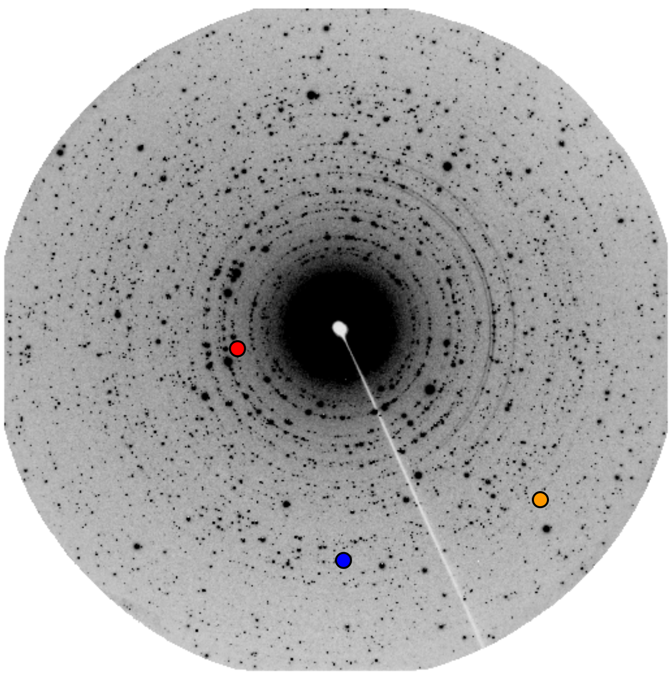
Much better than powder diffraction

S. Schmidt, H.F. Poulsen, G.B.M. Vaughan. *J. Appl. Cryst.* (2003) **36**, 326-332

G.B.M. Vaughan, S. Schmidt, H.F. Poulsen. *Z. Krist.* (2004) **219**, 813-825

Approach I: Small spot overlap

J. Wright, S. Schmidt, H.O. Sørensen, G. Vaughan



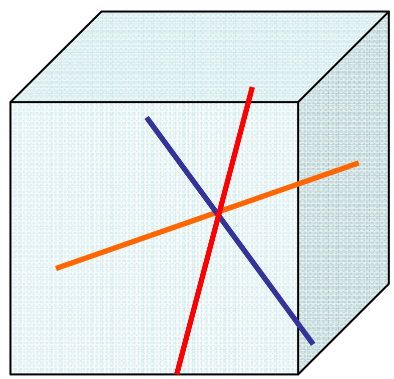
Spots:

- Filter bad spots away

Indexing:

- compare angles

or - resonances in orientation space



Søren

or - FFT

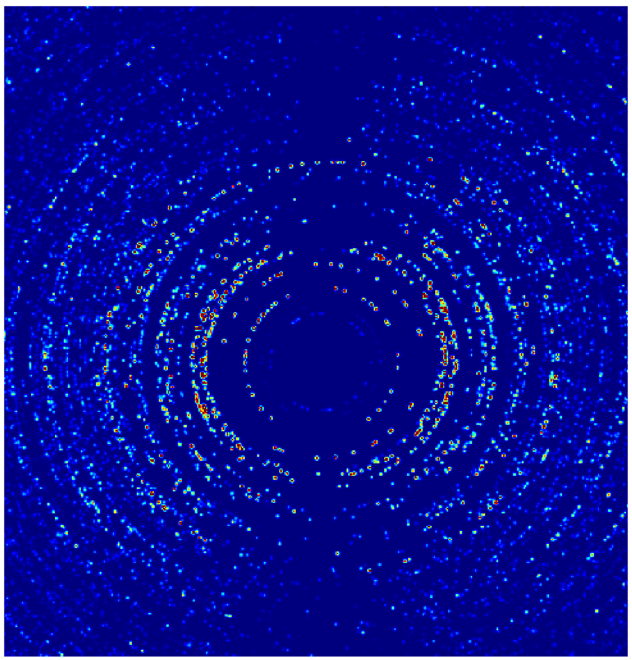
Talk by Jon

Harvesting

- conventional methods

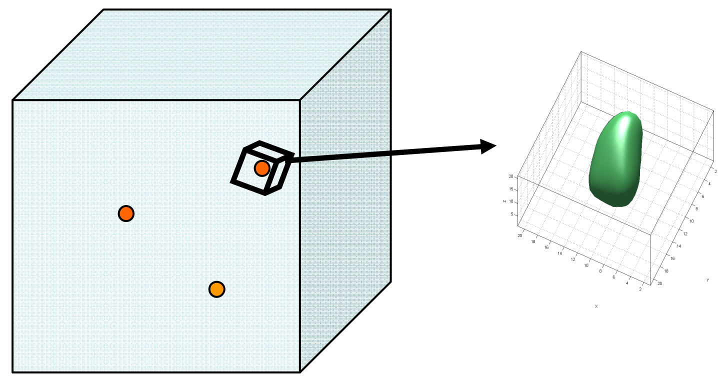
Approach II: Medium spot overlap

H.O. Sørensen, P.C. Hansen



Spot finding and indexing based on inner-most rings

For each grain: determine ODF



Harvesting for each grain: by projection of ODF

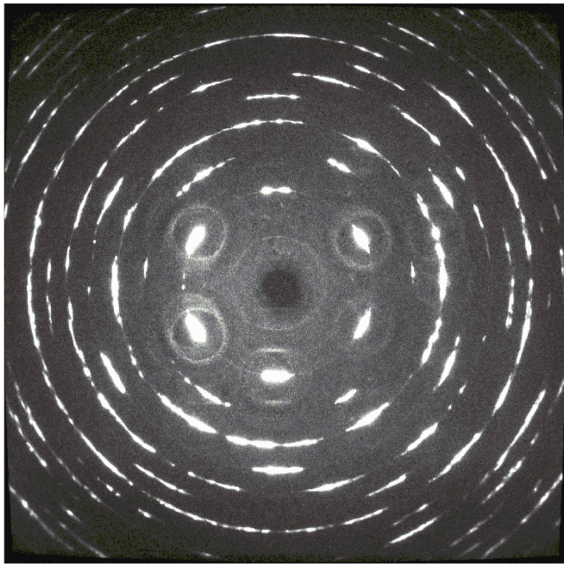
Talk by Henning Osholm

- delete or seperate or link in JANA

P.C. Hansen, H.O. Sørensen, Z. Sükösd, H.F. Poulsen. SIAM J. Imaging Series, in print (2009)

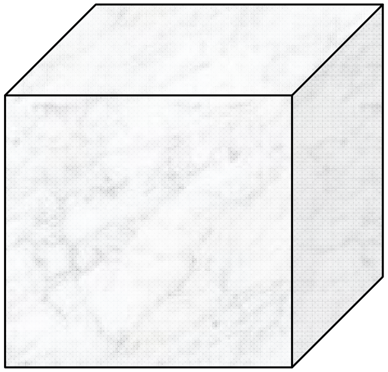
Approach III: Strong spot overlap

I. Kazantsev, S. Schmidt



Think of sample as single crystal with
an enormous mosaic spread.

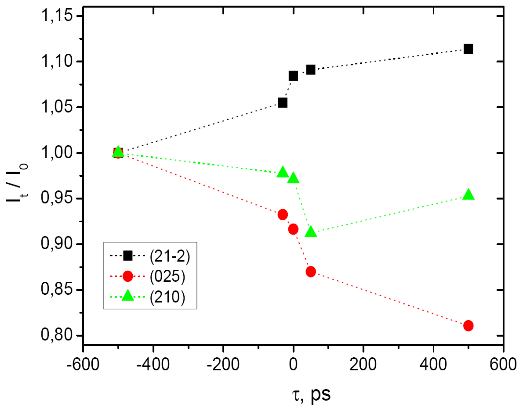
Determine ODF for complete sample
from large d-spacings



Harvesting by projection of ODF

Applications

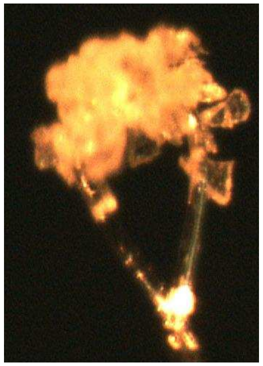
Timeresolved studies
in photochemistry



J. Davaasambuu

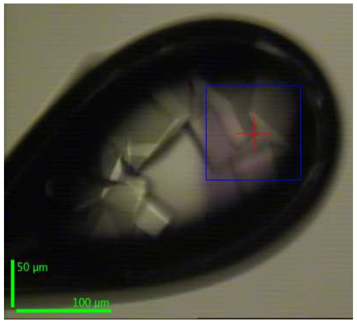
Simone Techert

Small molecule
crystallography



Henning O. Sørensen

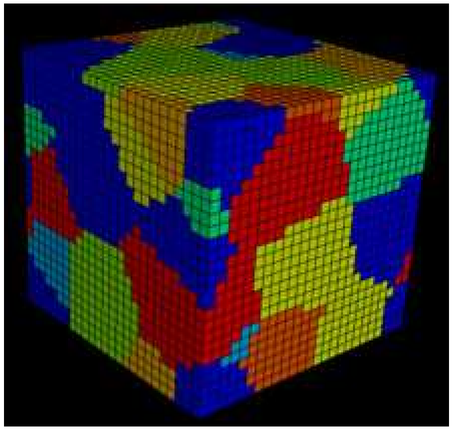
MX



Karthik Paithankar

Elspeth Garman

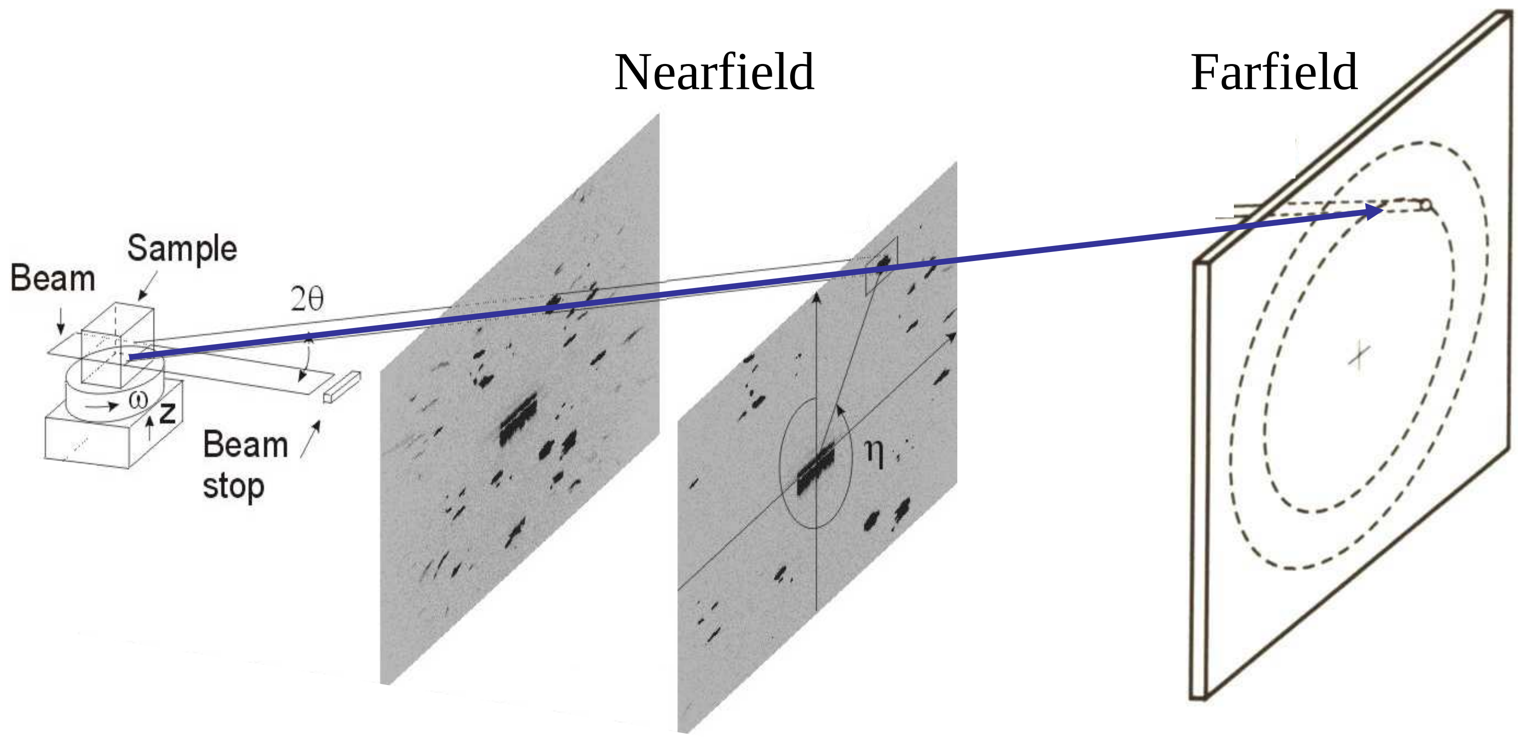
3D grain mapping



- 3D characterisation on a micron scale:
  - position, morphology
  - orientation of lattice
  - plastic and elastic strain
- In-situ studies

H.F. Poulsen: *Three-Dimensional X-ray Diffraction Microscopy* (Springer, 2004).

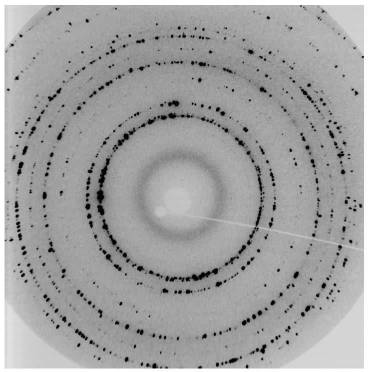
Set-up



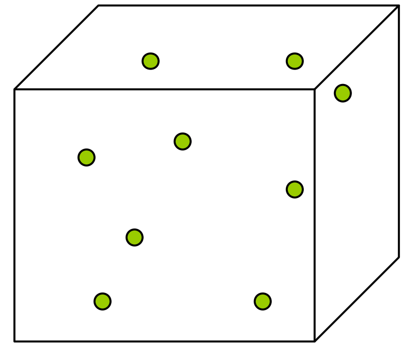
Indexing

Based on orientations

Graindex,
Grainspotter,
ImageD11



100-1000 grains:

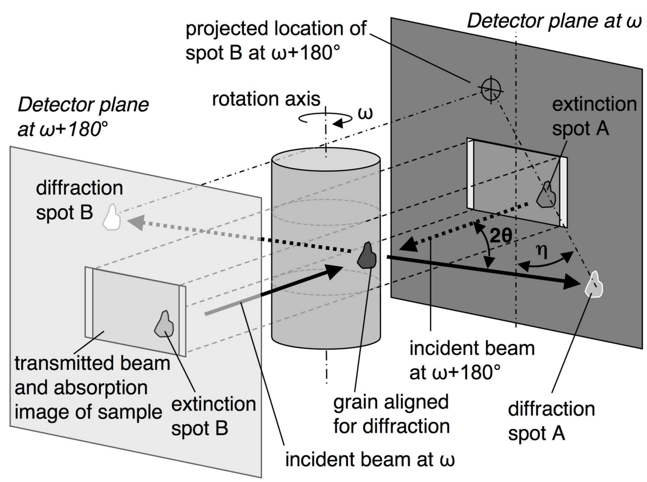


CMS position

volume:	10%
average orientation:	0.2 deg
average elastic strain:	$\Delta\varepsilon = 1*10^{-4}$

Based on positions

Use of Friedel pairs

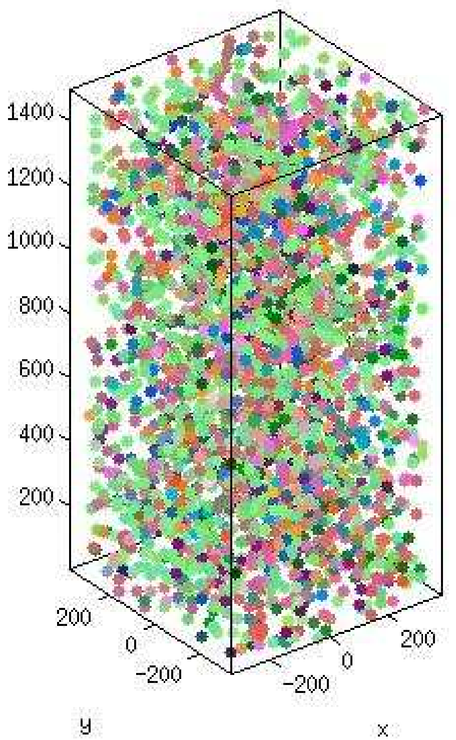


Talks by:
Marcin
Wolfgang

Grain position maps

Ex: IF steel sample with 2842 grains. ID11 work

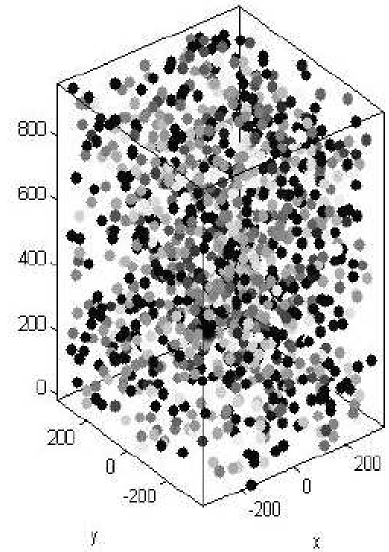
A. Orientation & volume:



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

B. Strain components:

axial strain at
3% tensile load

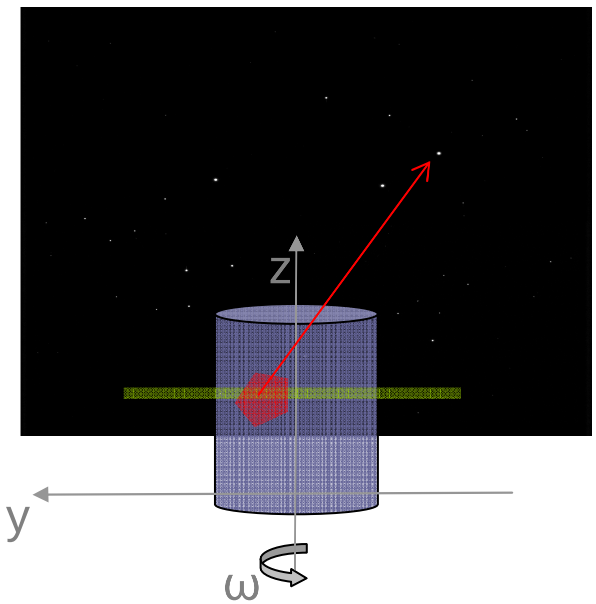


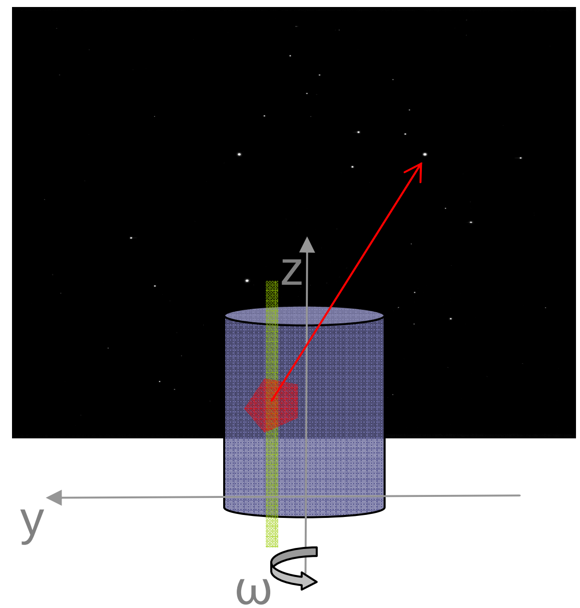
Talks by Jon, Jette, Ulrich, Wolfgang

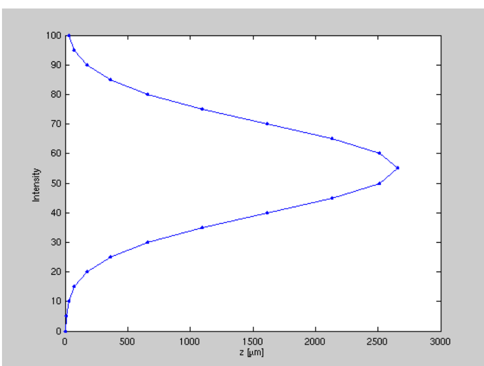
Also work by: C. Aydenier, J. Bernier, M. Miller

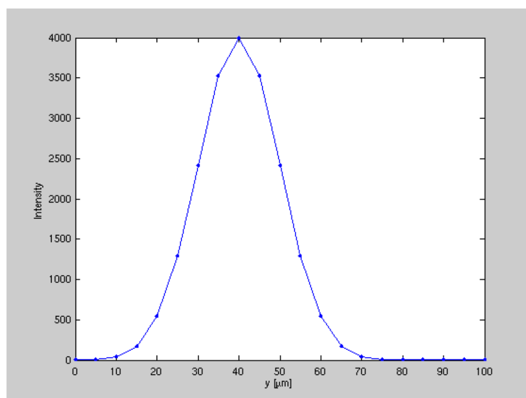
Progres on Boxscan method

Allan Lyckegaards PhD





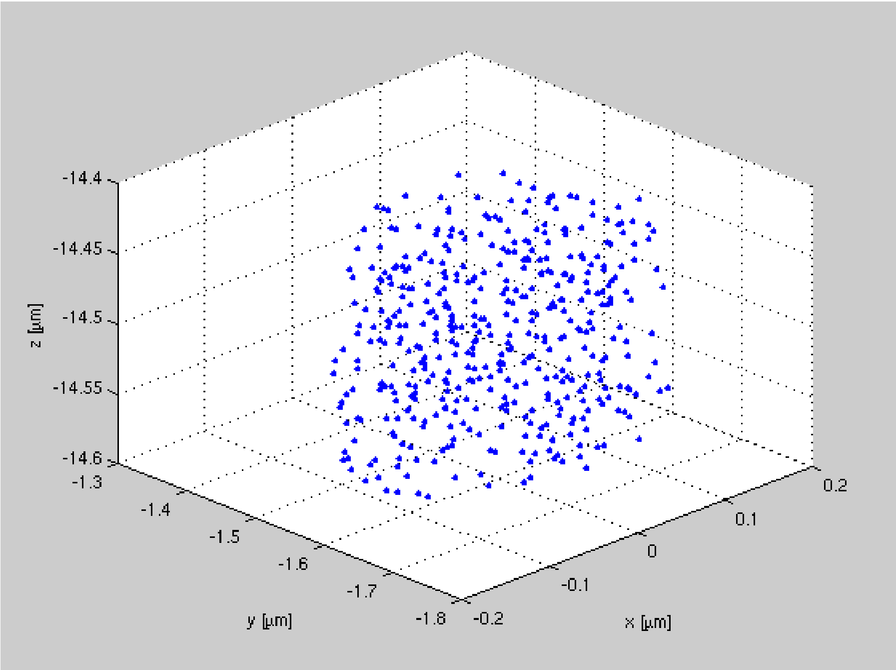




Demonstration of Boxscan

Beta-titanuim cylinder with 430 grains

5 µm steps, 30 deg rotation at ID11.

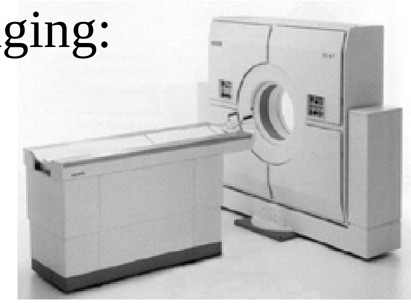


92% completeness
2 µm accuracy

A. Lyckegaard, E.M. Lauridsen, L. Margulies. Work in progress

Orientation mapping - the Math

Medical imaging:



Complications:

Curved space
Discrete events

Methods:

Direct projection: Grainsweeper by Søren & Carsten

Monte Carlo methods: Work by CUNY + Risø (A. Alpers, L. Rodek,…)
Work by R. Suter, Ulrich, …

Algebraic methods: ART, SIRT: Erik Knudsen, …..
Wolfgang, A. King, ….
CGLS: Henning Osholm, P.C. Hansen,…

Discrete tomography: Gibbs priors: Work by CUNY + Risø
DART: J. Batenburg, …

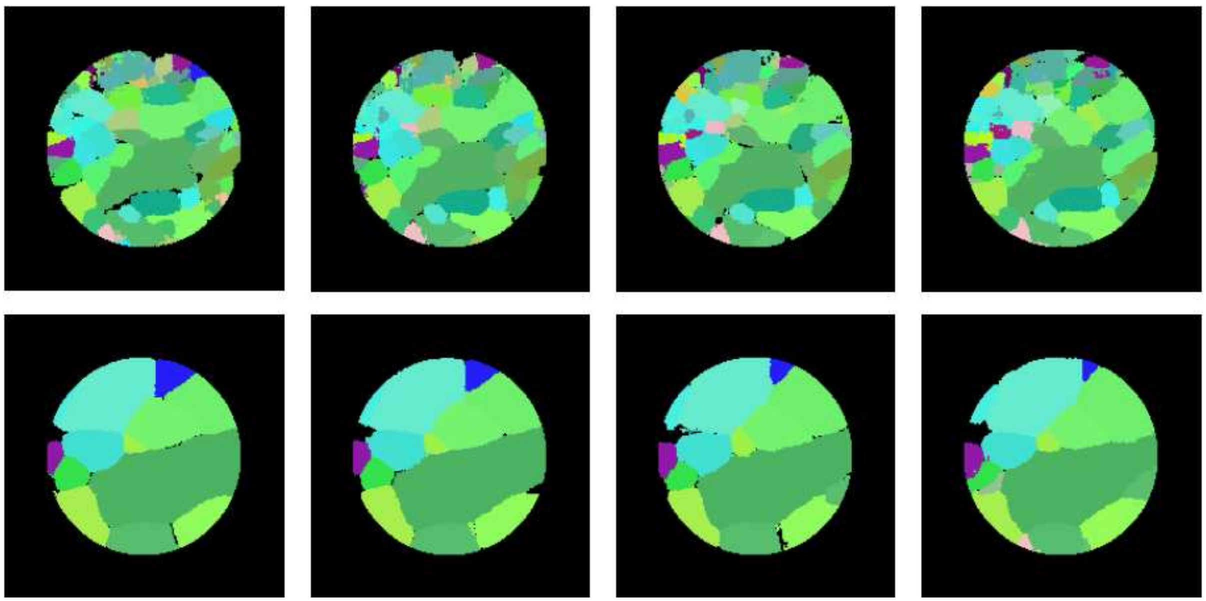
Grainsweeper

Grain growth experiment @ ID11

Layer 1 2 3 4 . . . .

Initial

After heating

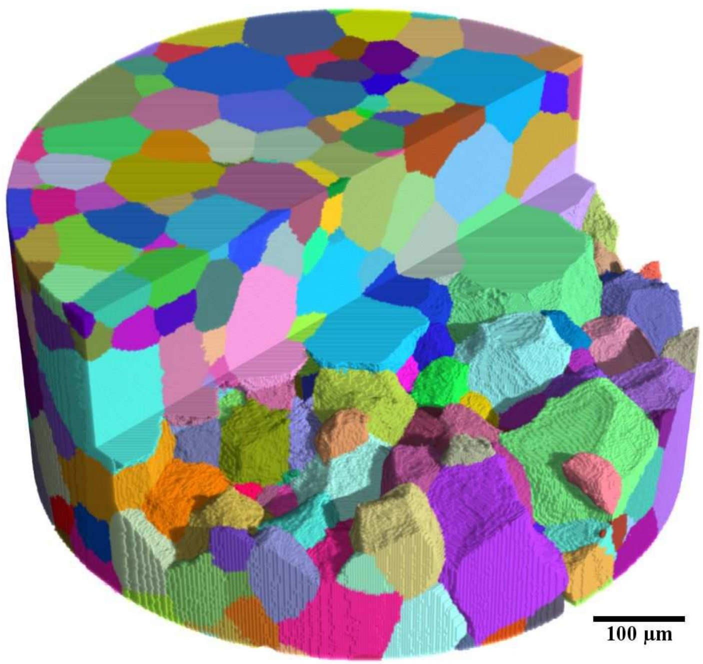


Spatial resolution: 5 μm

Risø: S. Schmidt, U.L. Olsen, H.O. Sørensen, E.M. Lauridsen, L. Margulies, D. Juul Jensen
Ecole des Mines, St. Etienne: C. Maurice, Naval Res. Office, Washington: R.W. Fonda

Diffraction contrast tomography (DCT)

Ex: 3D map of β-Ti made at ID11



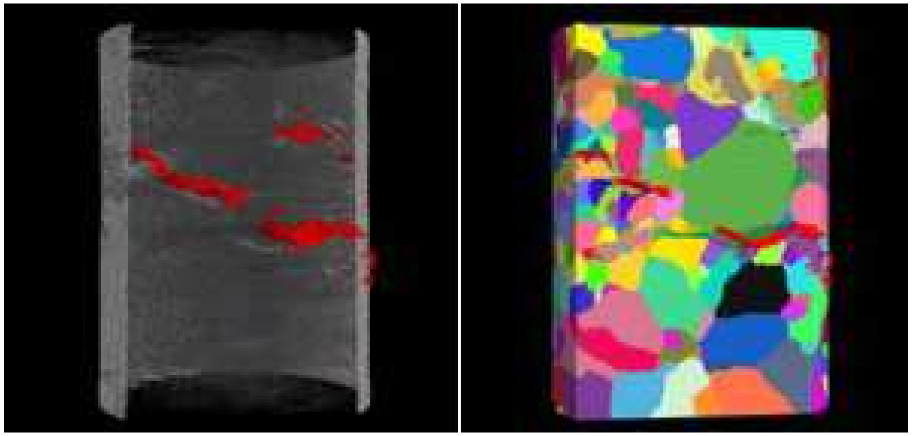
Talk by Wolfgang

Spatial resolution: 2.5 µm

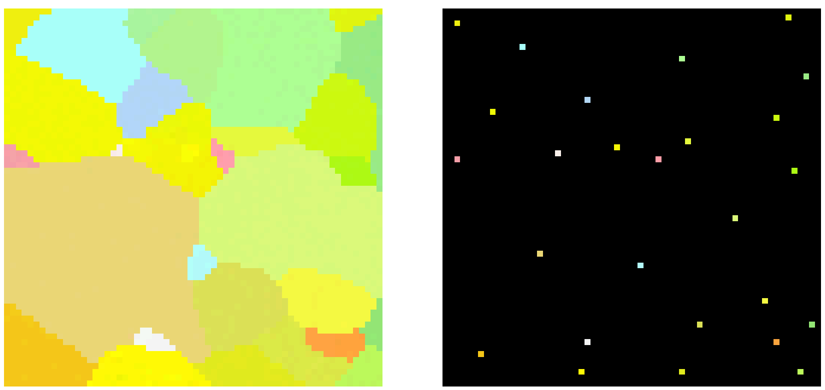
W. Ludwig, P. Reischig, A. King, M. Herbig, E.M. Lauridsen,
T. Marrow, J.Y. Buffière, submitted

Challenges in grain mapping

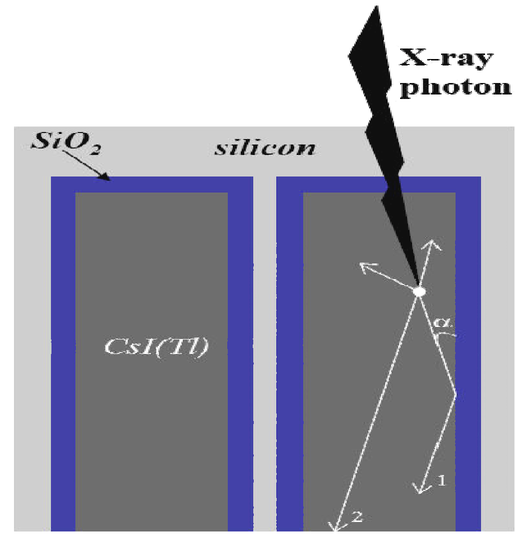
Combination with tomography*:



Mapping deformed materials^x:



New detectors:



U.L. Olsen, S. Schmidt, H.F. Poulsen, J. Linros. M. diMichiel, V. Honkimäki, T. Martin, J. Wright, G. Vaughan.

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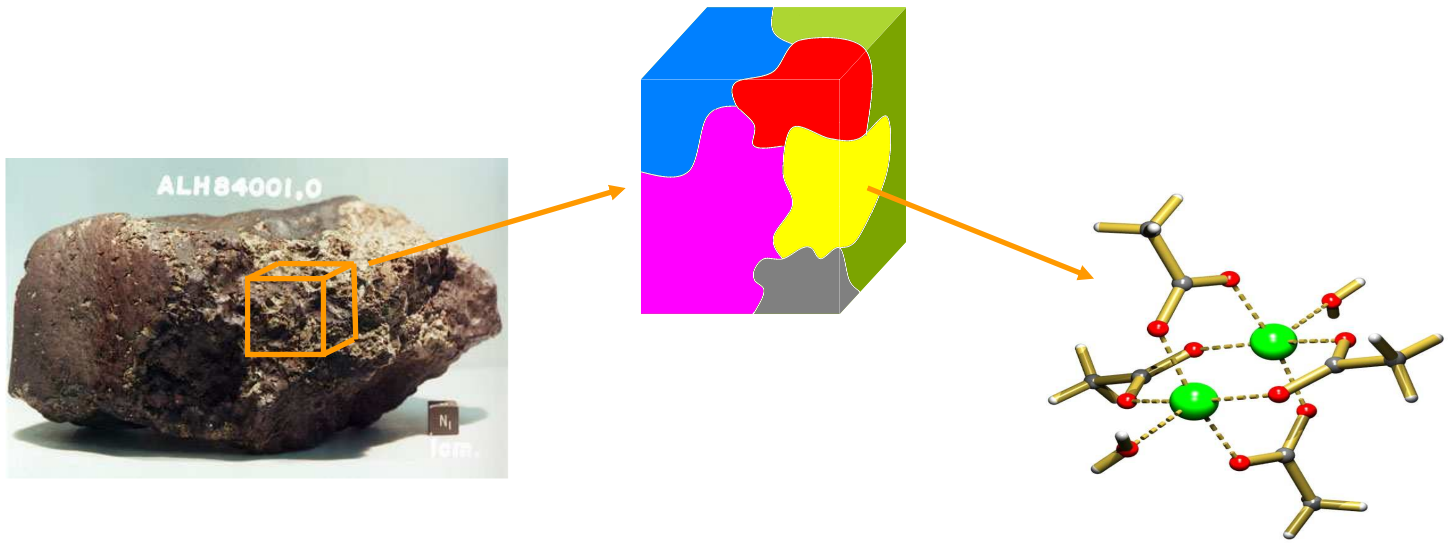
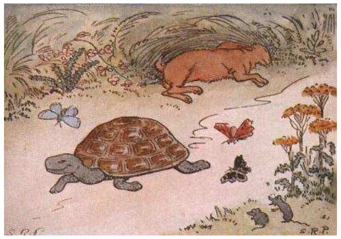
*: ID19 work: A. King, G. Johnson, D. Engelberg, W. Ludwig, and J. Marrow, *Science* (2008) **321**, 382 – 385

x: L. Rodek, H.F. Poulsen, E. Knudsen, G.T. Herman. J. Appl. Cryst. (2007) **40**, 313-321

TotalCrystallography



Phase, grain maps and dynamics of unknown multi-phase polycrystals



Talk by Andy

FABLE

Fully Automatic BeamLines and Experiments

Old school: GRAINDEX

- Linked to ImagePro
- Windows only
- Non modular

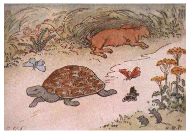
- No parallel computing
- Property of Risø

1 hour running time
Known space groups

New school: GrainSpotter

- No commercial programs
- Windows & Linux & Mac
- Modular
- Standalone or GUI
- Runs on clusters
- Sourceforge

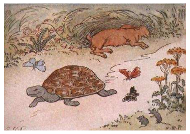
1 minute running time
Unknown space groups



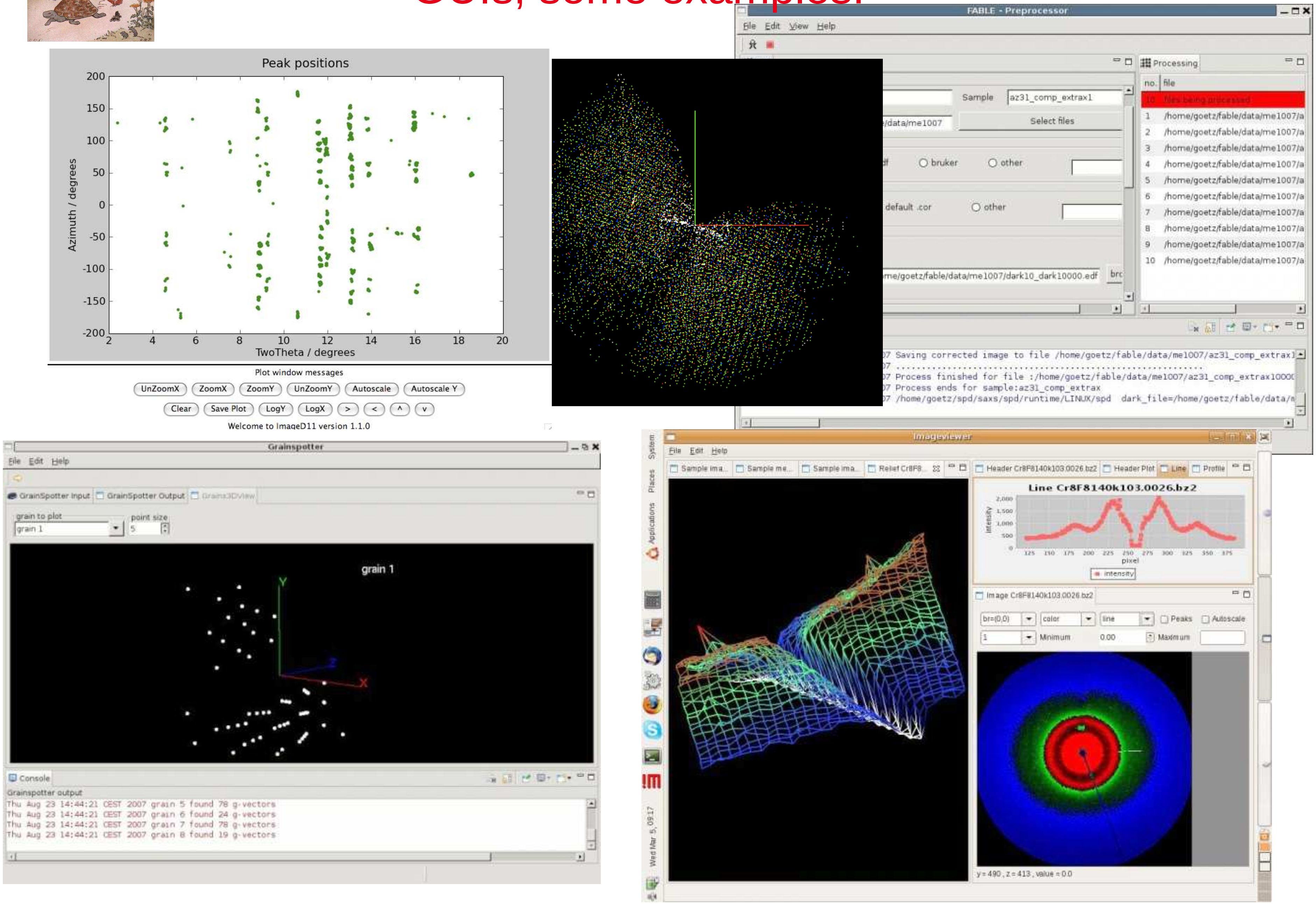
Simulators

For developers:
- Debugging
- Benchmarking
- Parameterstudies

For users:
- Training
- Optimisation of samples
- Optimisation of beamtime
- Future: *Optimisation of optics*



GUIs, some examples:



This workshop

Demonstration of FABLE software

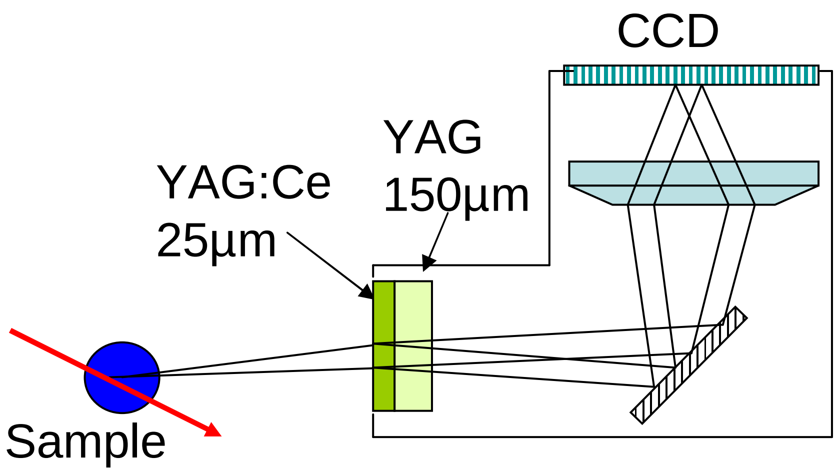
Workshop on polycrystal methods

How to continue?

- Total Cryst -

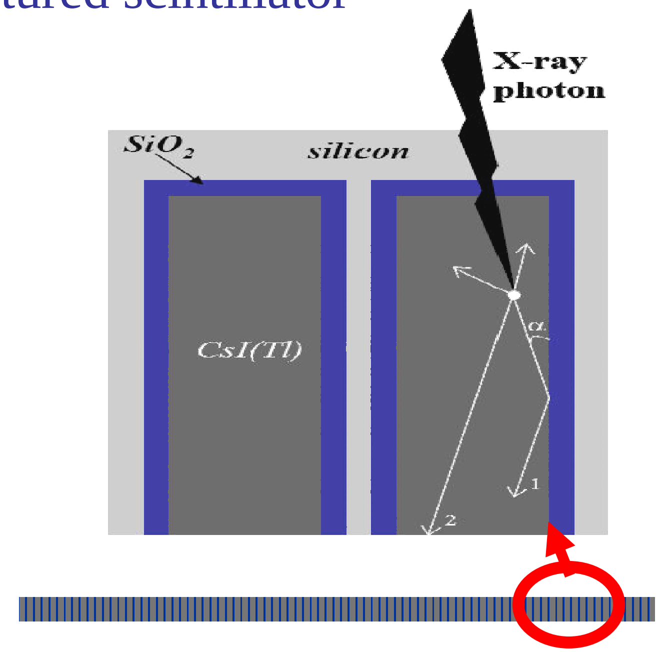
Better detector

Conventional

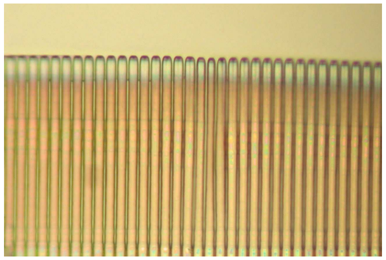


- Resolution: ~ 3 µm
- Efficiency: ~ 1%

Structured scintillator



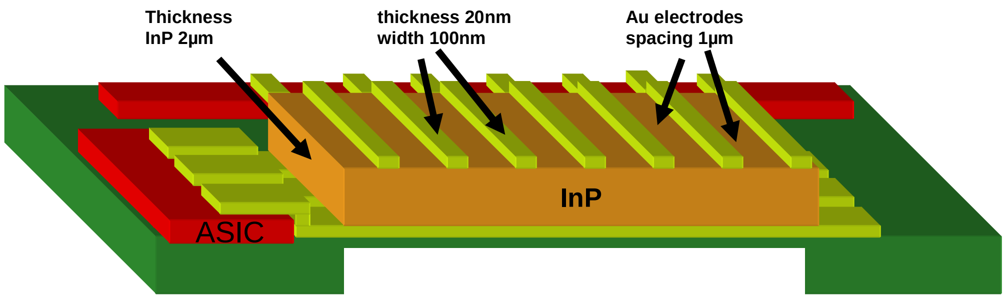
- Resolution: ~1 µm
- Efficiency: ~10%

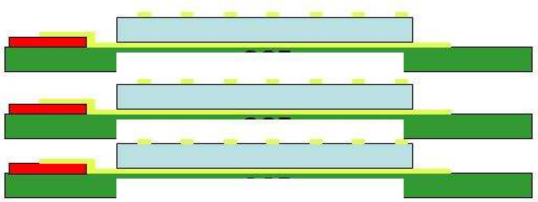


Risø: U.L. Olsen, S. Schmidt, H.F. Poulsen, KTH , Sweden: J. Linros.
ESRF: M. diMichiel, V. Honkimäki, T. Martin, J. Wright, G. Vaughan.

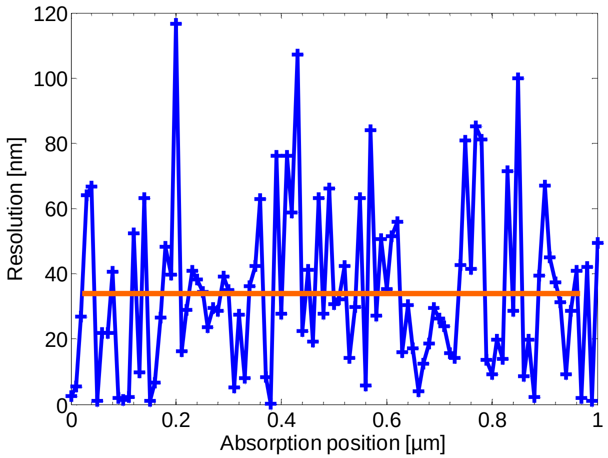
Even better detector

Principle:





Simulations:



Aim:
maps with resolution of 100 nm

Risø DTU: U.L. Olsen, S. Schmidt, H.F. Poulsen; DTU Photonics : K. Yvind, DESY: H. Graafsma