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

Nearfield

Farfield
Third road in Crystallography

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:

\[ \text{Cu(C}_2\text{O}_2\text{H}_3\text{)}_2\text{H}_2\text{O}. \]

70 grains of size < 1 micron
Cell \( \sim 1400 \text{ Å}^3 \) (C2/c)

Result:

Single crystal quality refinement!
Much better than powder diffraction

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
  or  - FFT

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

- delete or separate or link in JANA

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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

Small molecule crystallography

MX

J. Davaasambuu
Simone Techert

Henning O. Sørensen

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**

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Set-up
Indexing

Based on orientations

Graindex,
Grainspotter,
ImageD11

100-1000 grains:
CMS position volume: 10%
average orientation: 0.2 deg
average elastic strain: $\Delta \varepsilon = 1 \times 10^{-4}$

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:

B. Strain components:

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

Talks by Jon, Jette, Ulrich, Wolfgang

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

Allan Lykkegaard's PhD
Demonstration of Boxscan

Beta-titanium 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

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Diffraction contrast tomography (DCT)

Ex: 3D map of β-Ti made at ID11

Spatial resolution: 2.5 µm

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Challenges in grain mapping

Combination with tomography*:

Mapping deformed materials\textsuperscript{x}:

New detectors:


\textbf{New detectors:}

\textbf{X-ray photon}

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

Talk by Andy
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?
Better detector

Conventional

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

Structured scintillator

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

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ESRF: M. diMichiel, V. Honkimäki, T. Martin, J. Wright, G. Vaughan.
Even better detector

Principle:

Simulations:

Aim: maps with resolution of 100 nm