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High Energy Diffraction Microscopy at the Advanced Photon Source 1-ID Beamline

by Ulrich Lienert

APS 1-ID Beamline

Advanced Photon Source

1-ID Timeline

96: First operational APS beamline

High energy program started

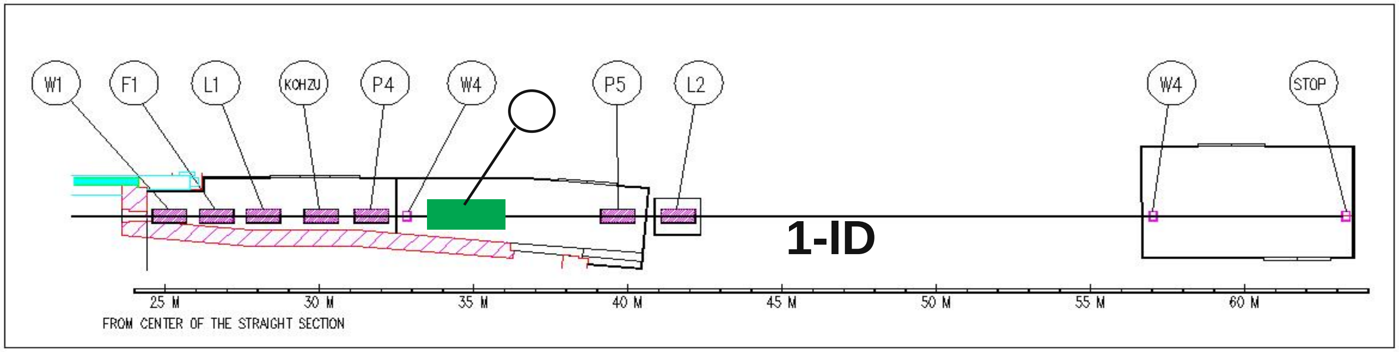
by D. Haeffner & S. Shastri

05: Dedicated high energy beamline

06: Dedicated 3DXRD setup in 1-ID-B

08: Short period undulator

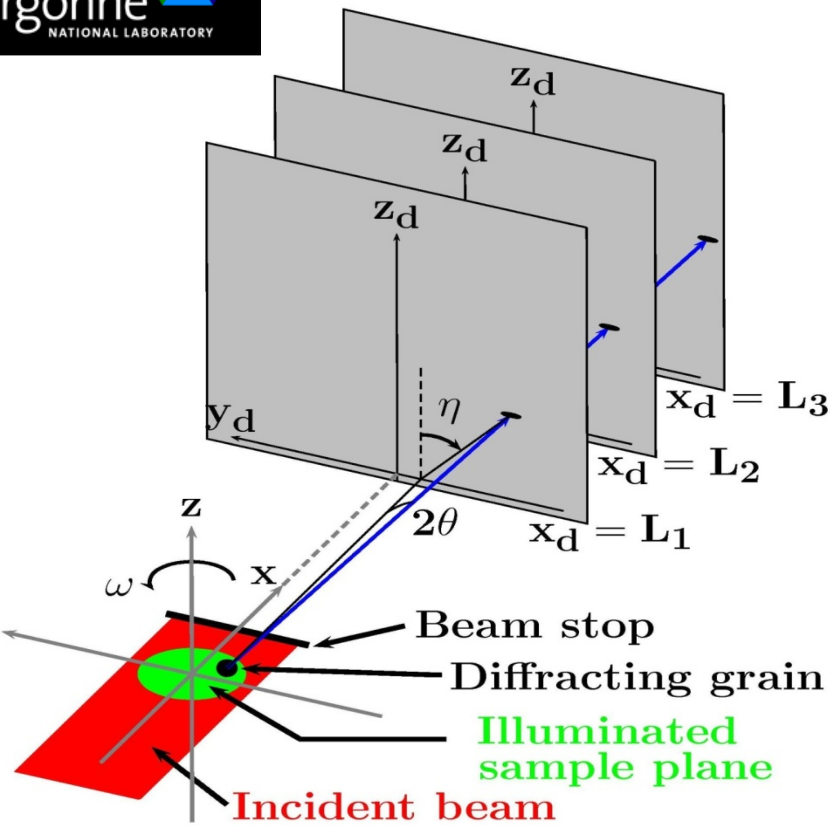
0?: Redesign of hutch layout



Near-field

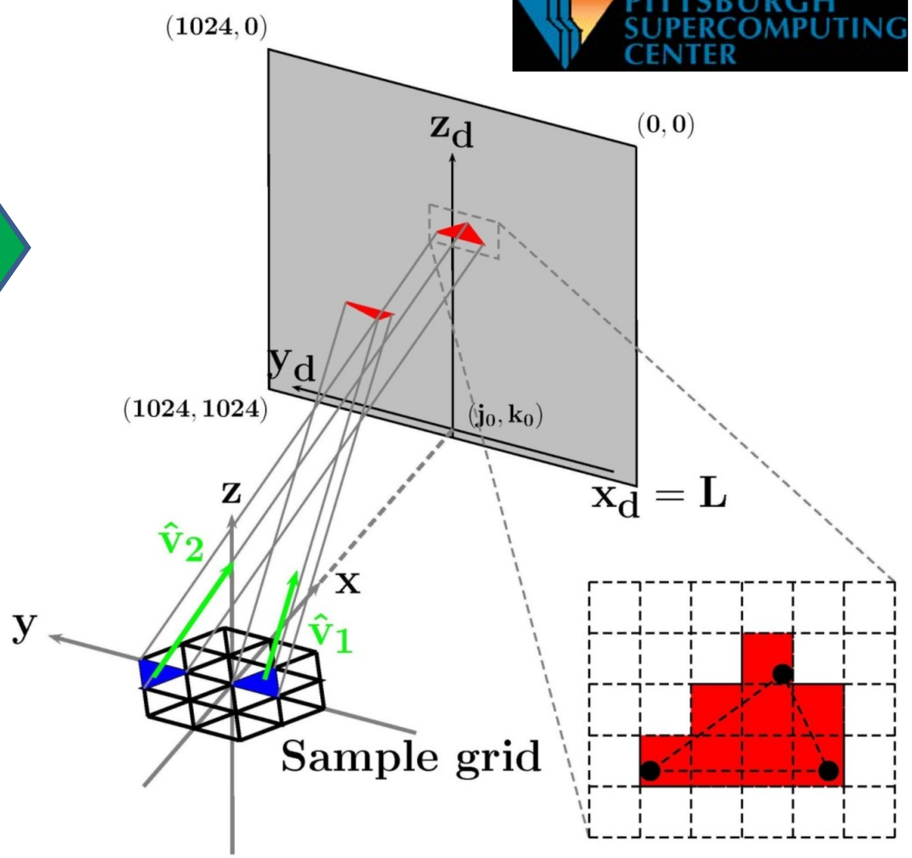
- C. Hefferan, S. Li, R. Suter (CMU)

Experimental Measurement at APS synchrotron 1-ID beamline



H.F. Poulsen, Springer Tracts

Monte Carlo Simulation conducted on BigBen & CMU cluster



Suter, et al, Reviews of Scientific Instruments, 2006 and J. Engineering Materials & Technology, 2008

Analysis: computer simulation of experiment and sample: “forward modeling”

Minimize cost function:

$$\chi = S - 2\,O$$

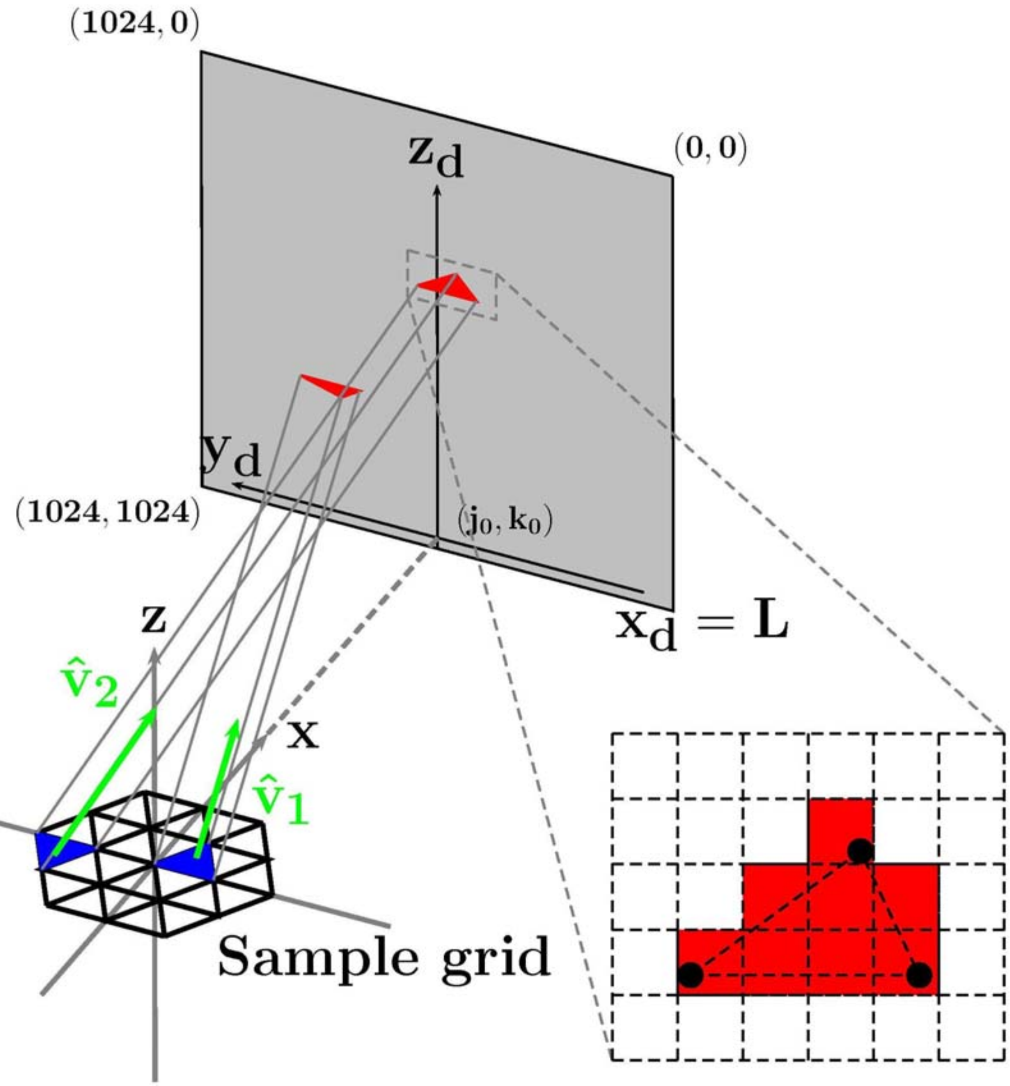
Diffracted beam overlap:

$$C = N_{exp} / N_{sim}$$

$$= \text{“confidence”}$$

Need to know (L, j_0, k_0, …) before this will work

Analysis doesn’t “know” anything

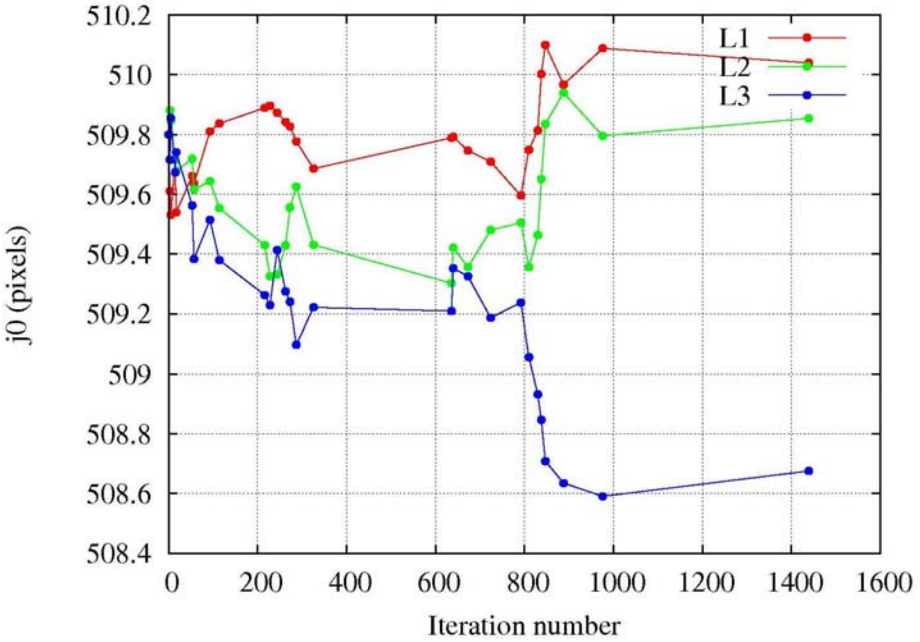


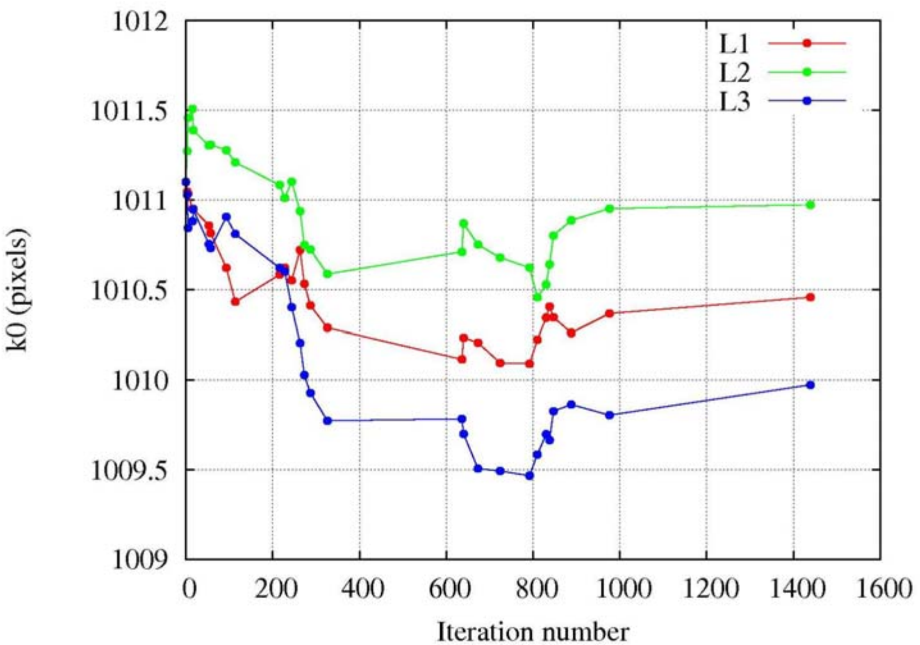
Suter, et al, Reviews of Scientific Instruments, 2006 and J. Engineering Materials & Technology, 2008

Determination of experimental parameters

Based on diffraction images from centered 20 micron dia annealed gold wire

1. Back projections yield overlap point in 3D: detector origin and detector - rotation axis distance
2. Monte Carlo optimization:
   - Fit orientations to ~100 elements
   - Move parameters and re-adjust orientations
   - Cost function maximizes overlap with data

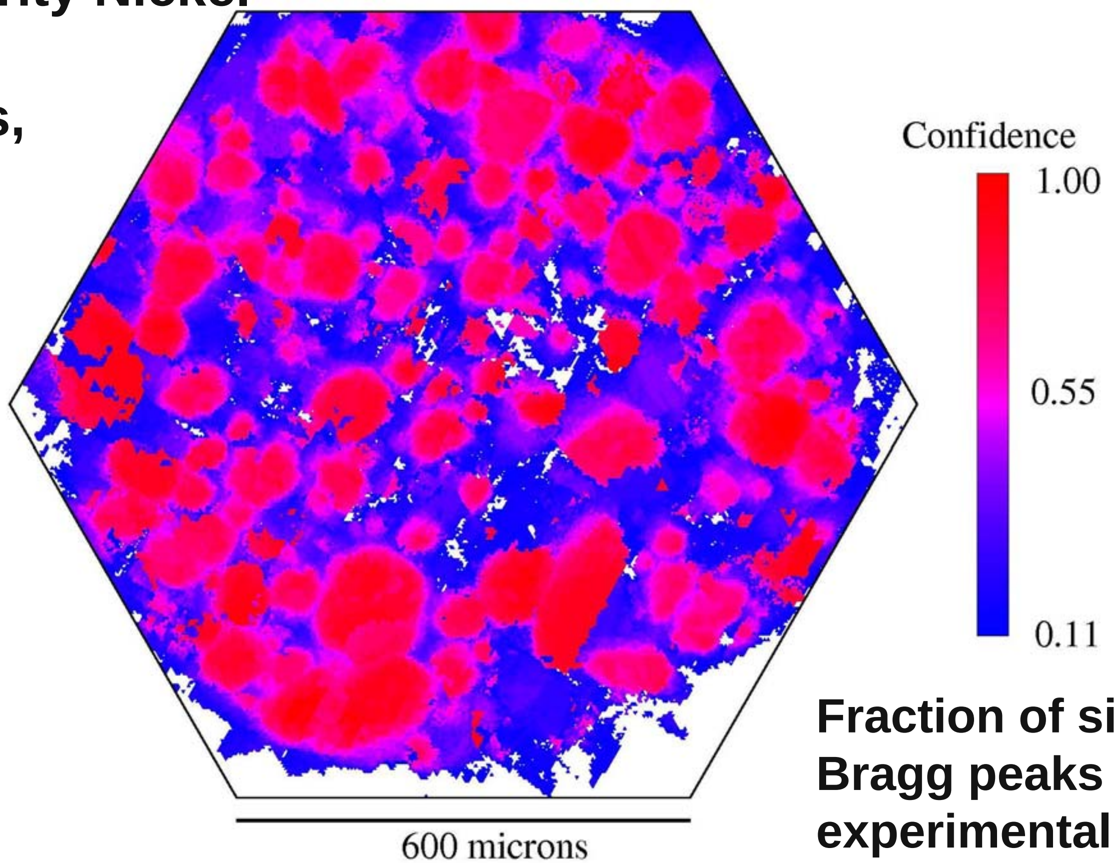




HEDM: Grain Mapping

Annealed high purity Nickel

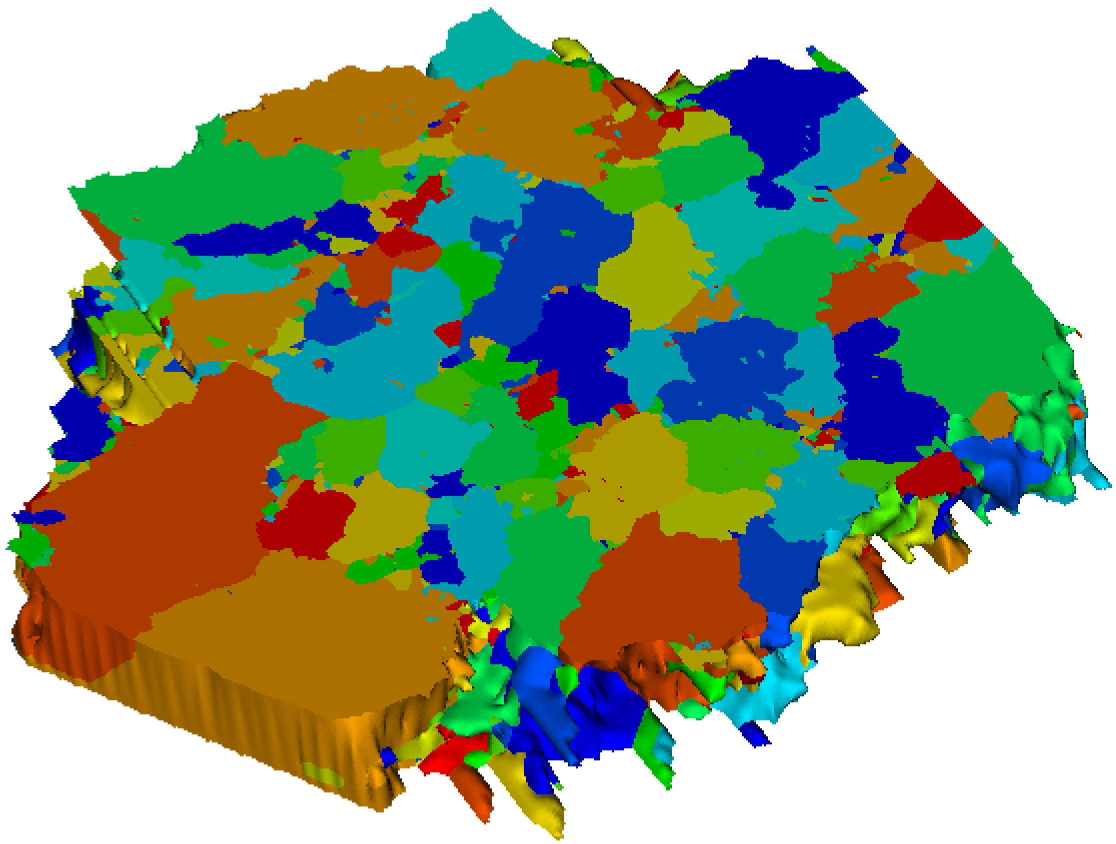
One of nine layers, 20μm separation



Tweaked fitting algorithm yielded greatly improved fits: $L_n - L_1$ free

Fraction of simulated Bragg peaks striking experimental intensity (of ~45 qualified)

3D Reconstructed Microstructure



Triangular grid of boundaries
with Laplacian smoothing

R.H. Moore, et al, Comp. Mat. Sci.

Computations are "embarrassingly parallelizable"

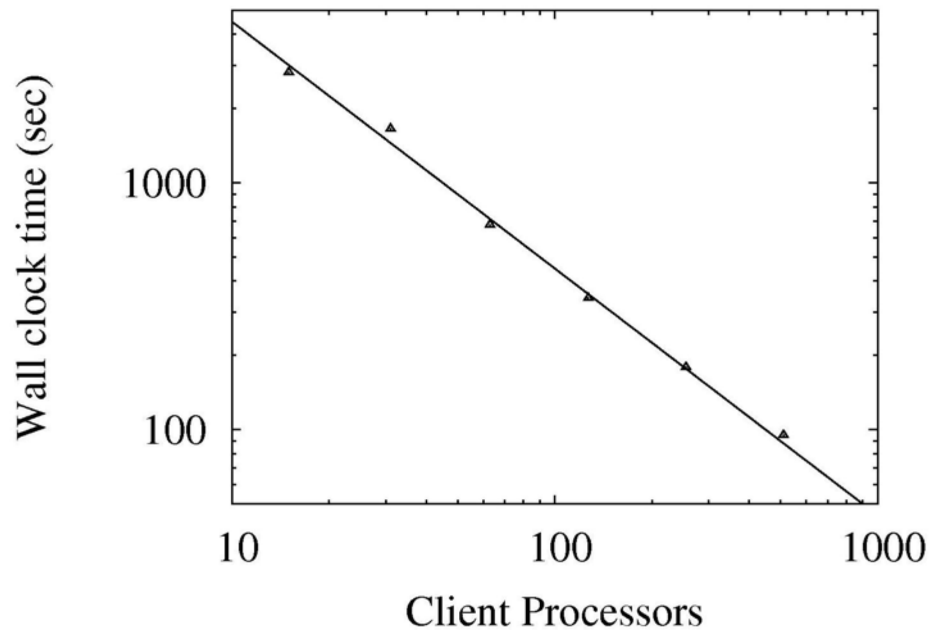
• Each triangular element treated independently

• Using PSC, CMU and APS clusters: time scales linearly with processor number

• But no intensity matching

• More complex parallel schemes ⟶ intra-grain orientation gradients

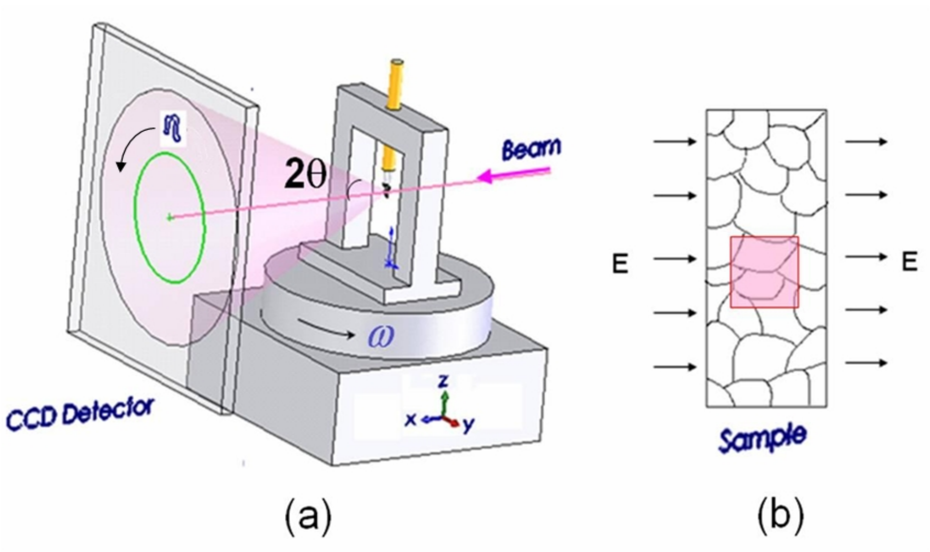
Total fitting time for test data set



Far-field: Ferroelectrics $BaTiO_3$

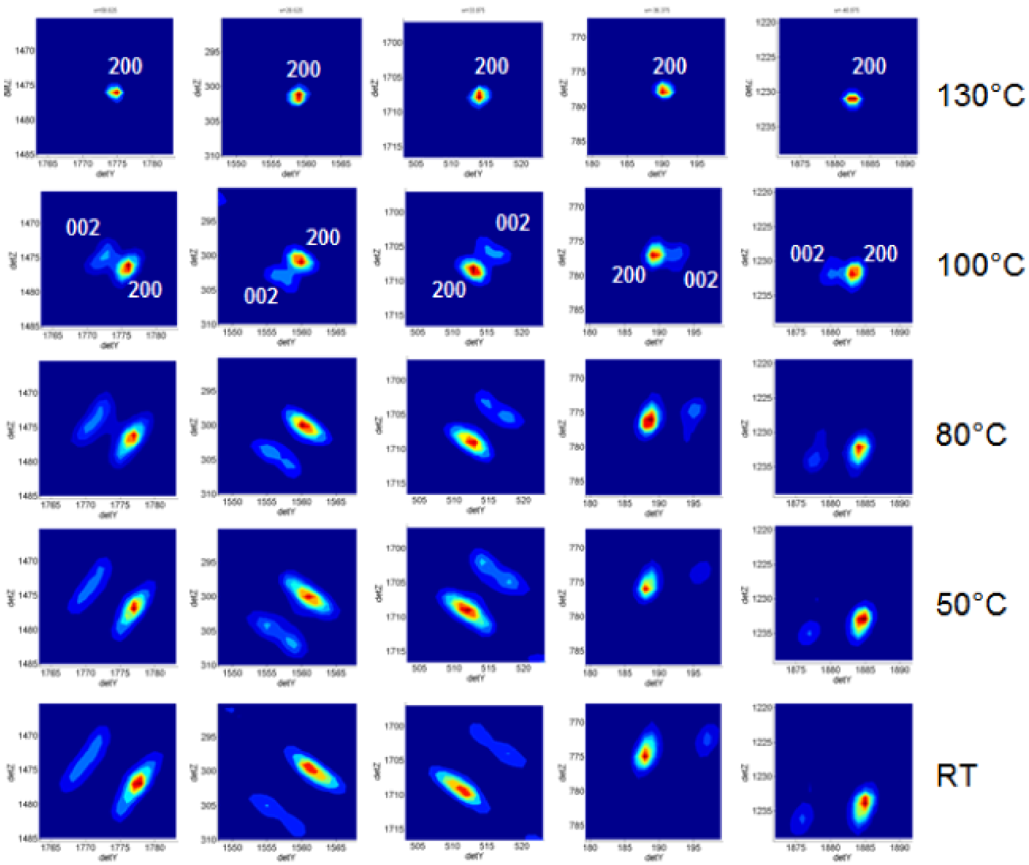
- Mesut Varlioglu, Ersan Ustundag (Iowa State University)
- Tetragonal, c/a ≈ 1.01
- Domain crystallography: twinning on {110} planes
- Mismatch angle: 2 atan(a/c) ≈ 89.4 deg

- E = 80.73 keV
- Sample 1x1x5 mm3
- Grainsize 20-30 μm
- Beamsize 30x30 μm
- $\Delta\omega = 0.2^o$, $+-65^o$
- E-field up to +-20 kV/cm
- Sample pre-poled
- Indexed tetragonal grains



Cubic – tetragonal phase transition

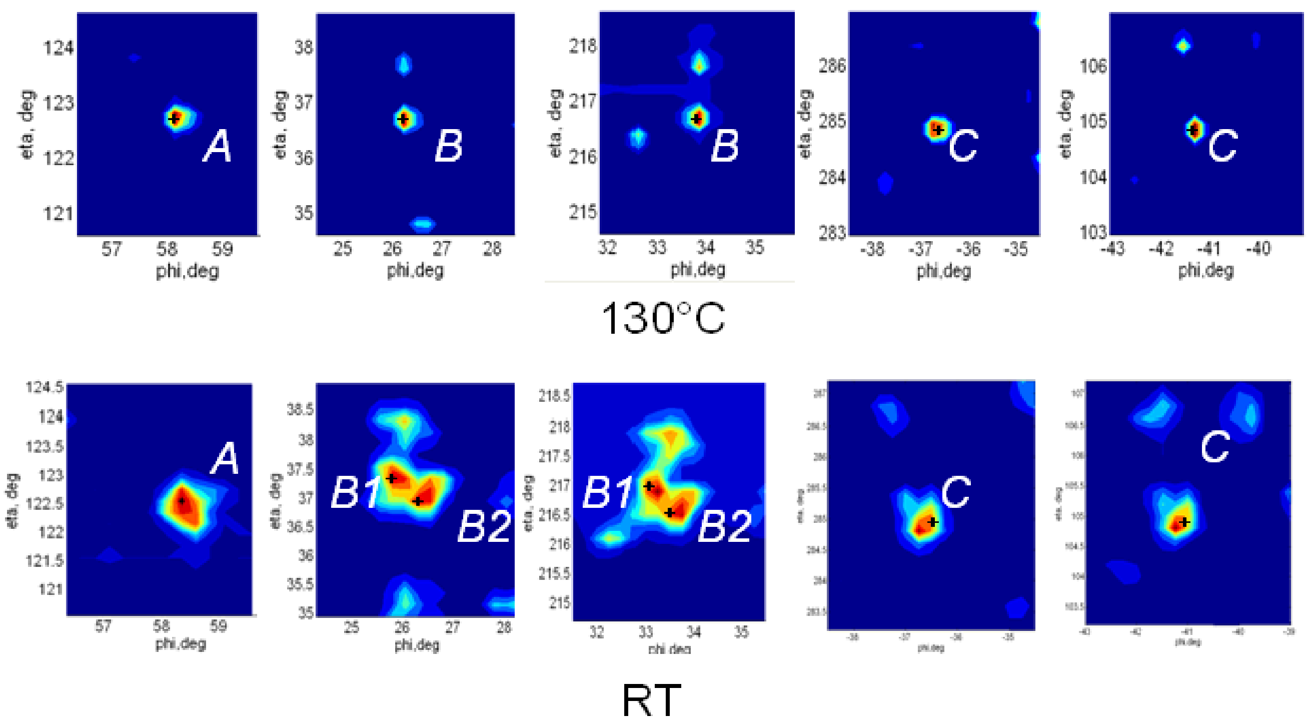
- ω-integrated raw data



- Significant broadening
- Azimuthal & radial domain separation difficult

Domain indexing

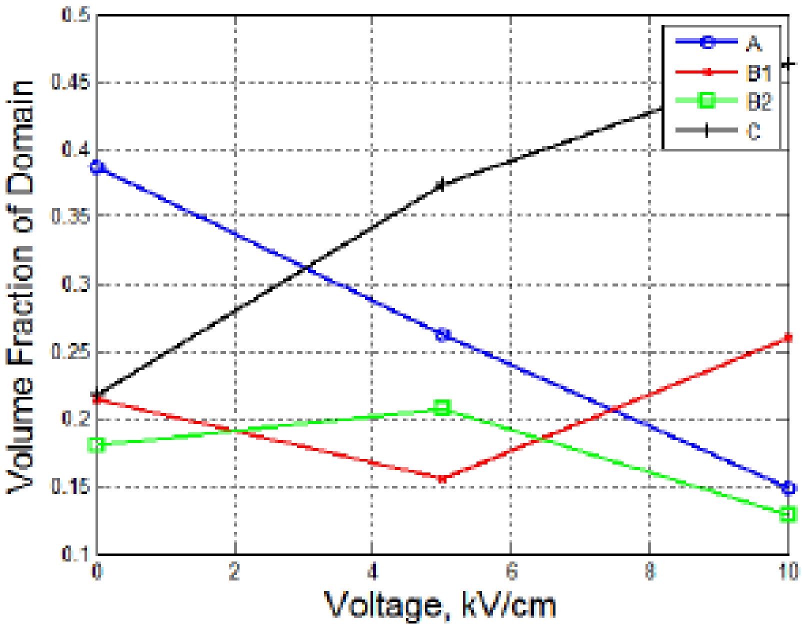
- (002) orientation map (η, ω)



- automated indexing of cubic phase
- manual refinement of tetragonal domains

Applied electric field

- Domain volume fractions vs. applied field

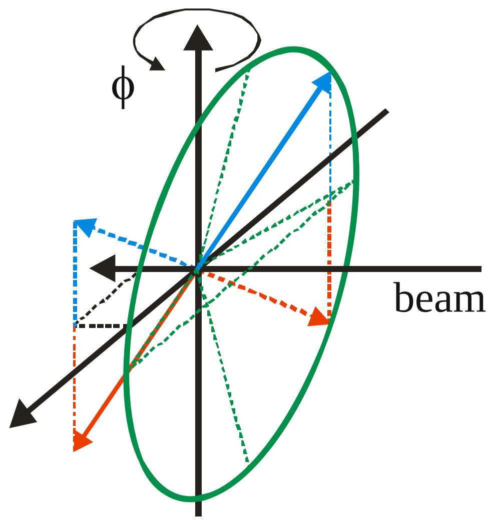


- 90 deg domain switching

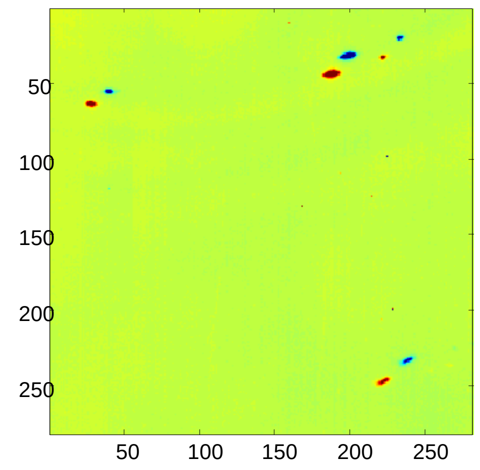
Friedel pair alignment

- Take images rotated 180 deg around vertical axis
- Flip rotated image around center row
- Friedel pairs should line up

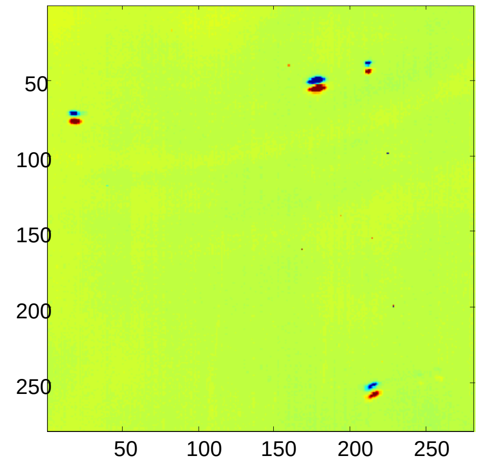
- Alignment of:
  - rotation axis parallel to detector columns
  - vertical beam center on detector
  - beam over rotation axis
- uses mounted sample
- no grain indexing required



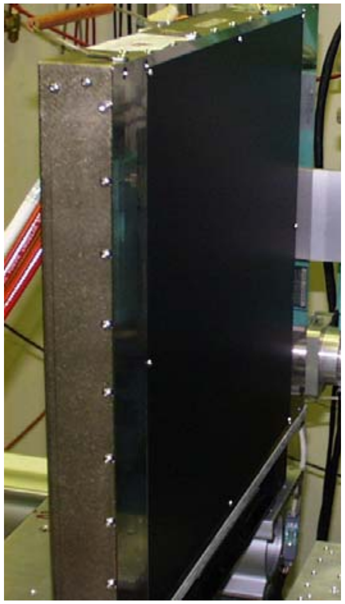
$\Delta\chi = 0.5$ deg



$\Delta\chi = 0$ deg



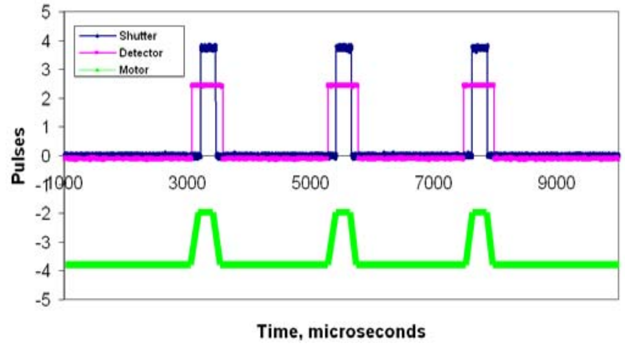
Fast scanning



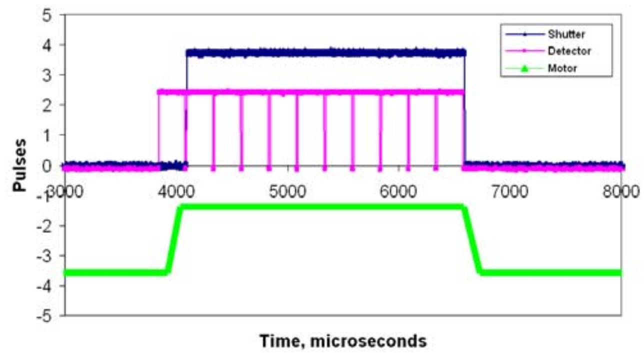
GE Rad detector

- 40x40 cm^2 area
- 2kx2k (1kx1k) pixels
- 7 (30) fps
- low frequency spat. Distortion
- up to 300 frames buffer
- ‘lagging’ (3%)

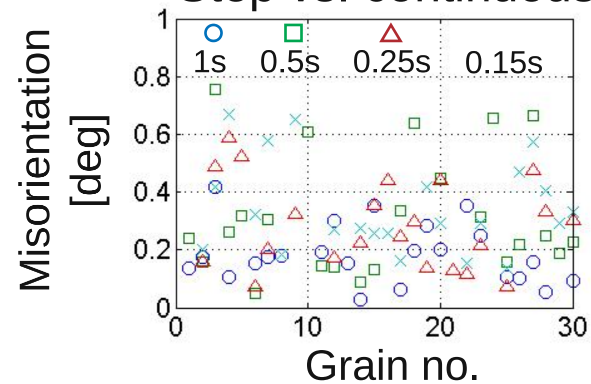
Single frame scanning

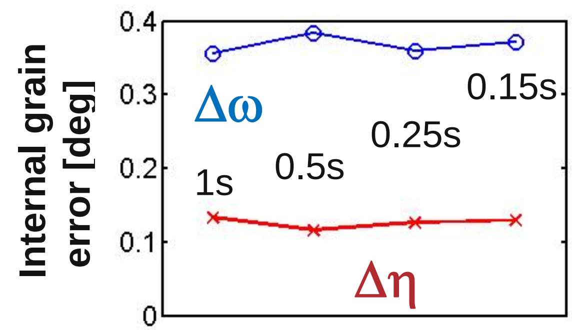


Continuous scanning



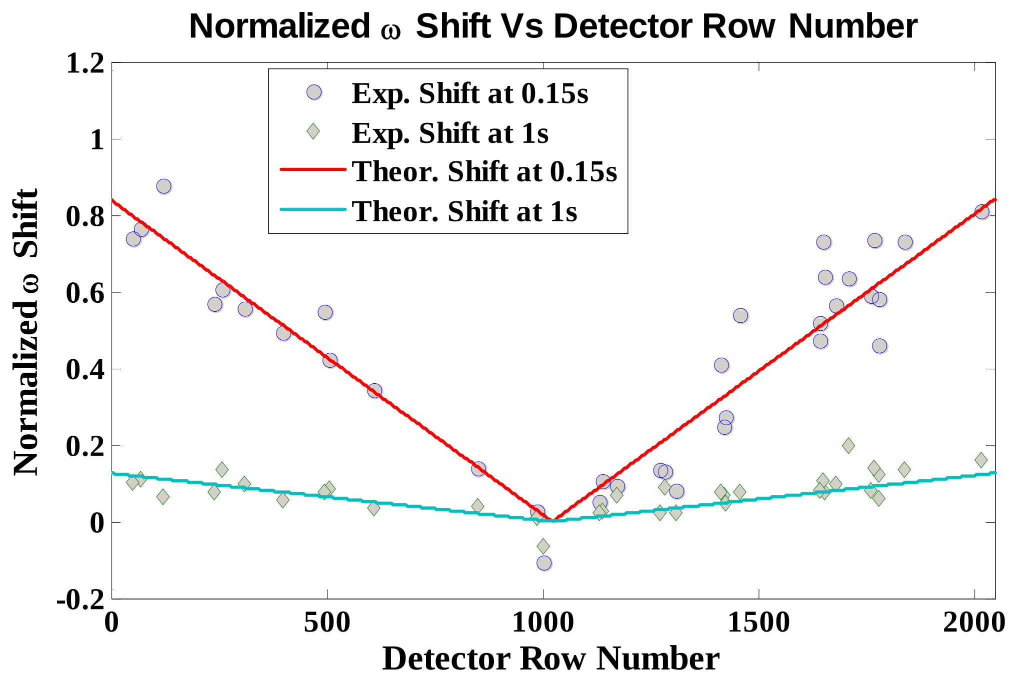
Step vs. continuous





Continuous readout ω correction

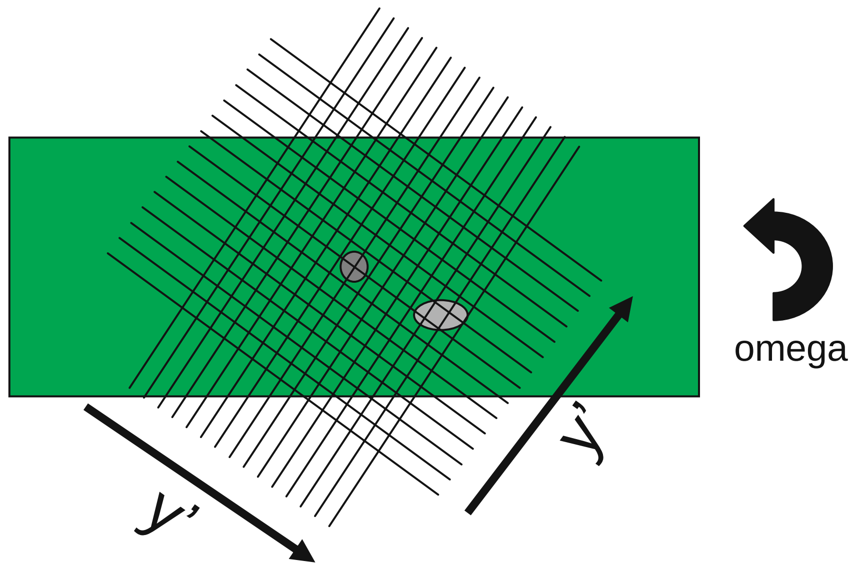
- Readout of rows starting at center
- Compare ω positions of continuous and step scans (Δω = 0.5 deg)

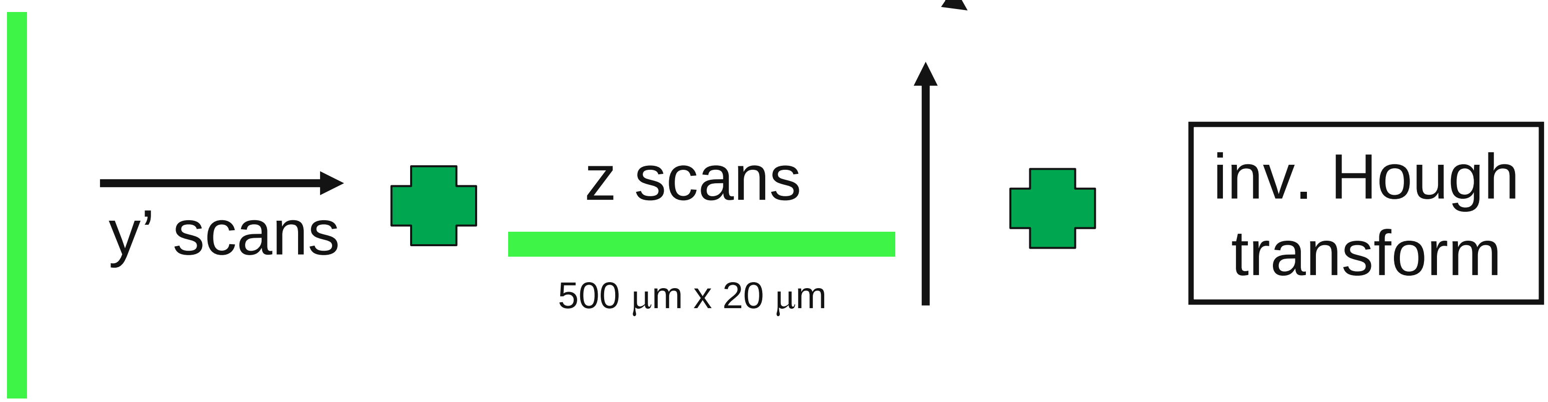


- ω position can be corrected by row number and exposure time
- No information loss by continuous scanning

”Box Scan” Technique for 3-D Center-of-Mass Determination

- A. Lyckegaard, E. Lauridsen (Risø)



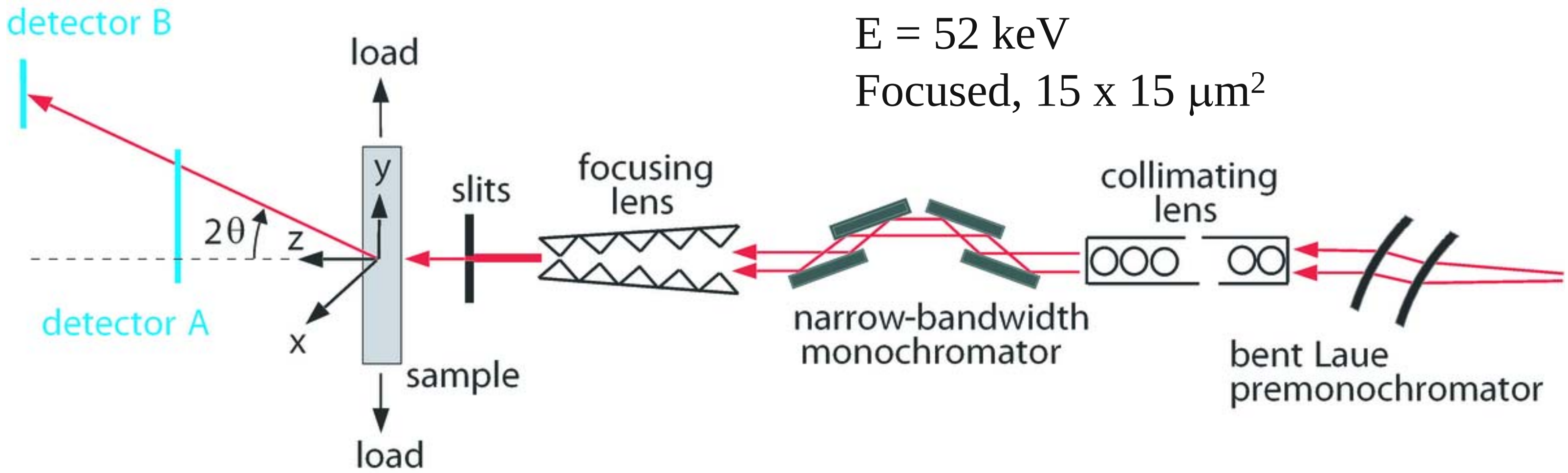


20 μm x 500 μm

- orientation, COM, volume, strain

High resolution reciprocal space mapping

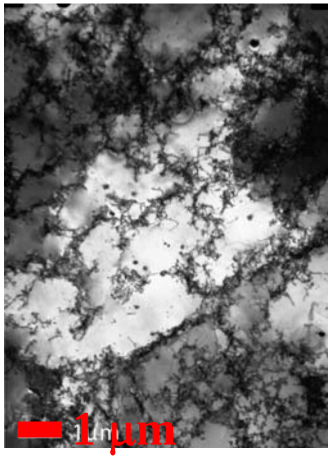
- C. Wejdemann, W. Pantleon, H. Poulsen (Risø)
- S. Shastri (APS)



E = 52 keV
Focused, 15 x 15 μm²

Samples

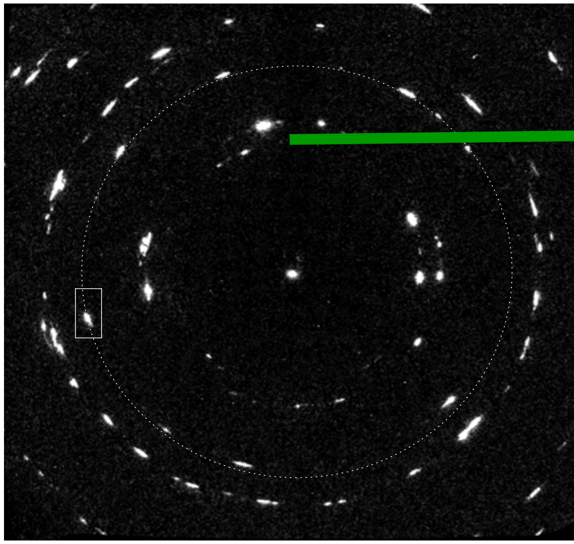
- Cu (99.99% OFHC)
- Cold rolled to 80% reduction, fully recrystallized
- Thickness 300 μm, 30 μm grain size (EBSP)
- Displacement controlled tension rig
- [400] || load axis



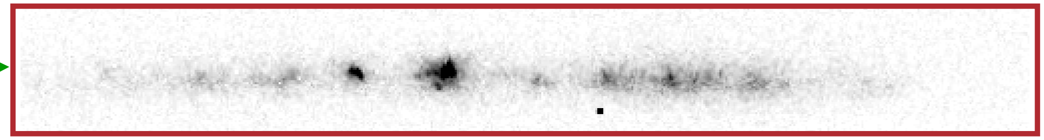
Raw data

Detector A
40 cm from sample

Detector B
4 m from sample



Zoom



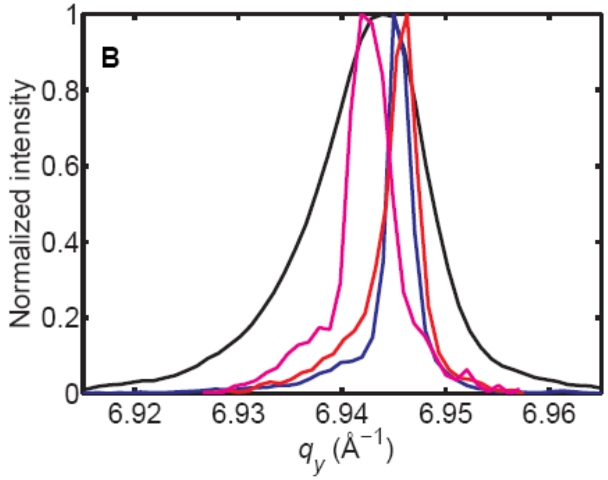
$\Delta\omega = 0.001$ deg

Grain-scale

Subgrain-scale

Cell & wall identification

Strain:

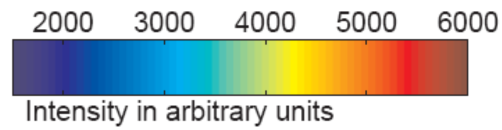


3D reciprocal space map

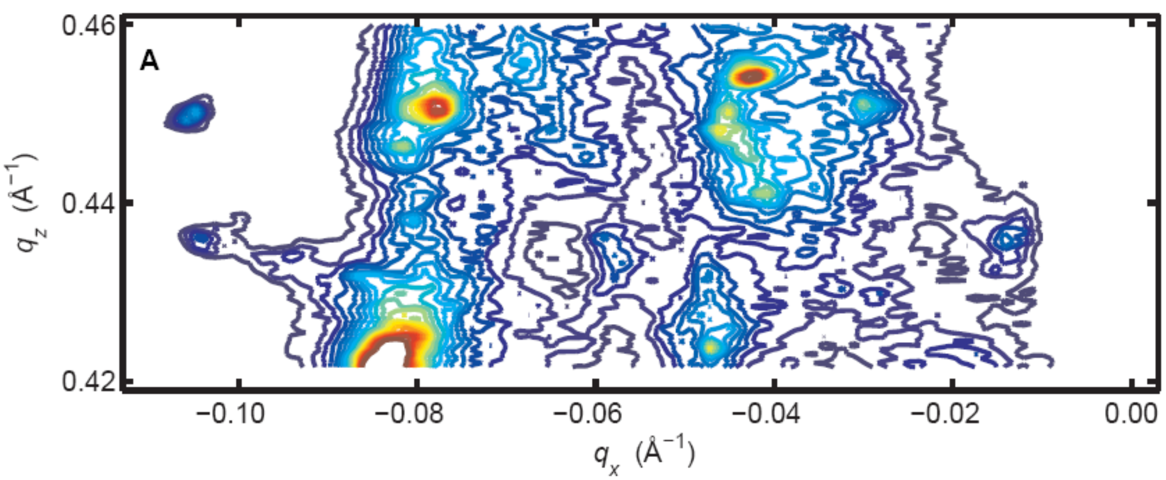
- 400 Cu at 3.5% strain
- Few minutes acquisition

Subgrains

- Size 1-3 μm
- Sharp peaks
- From unique sample position



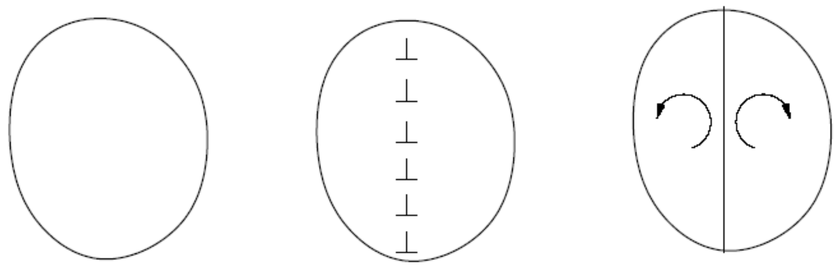
Mosaic spread:



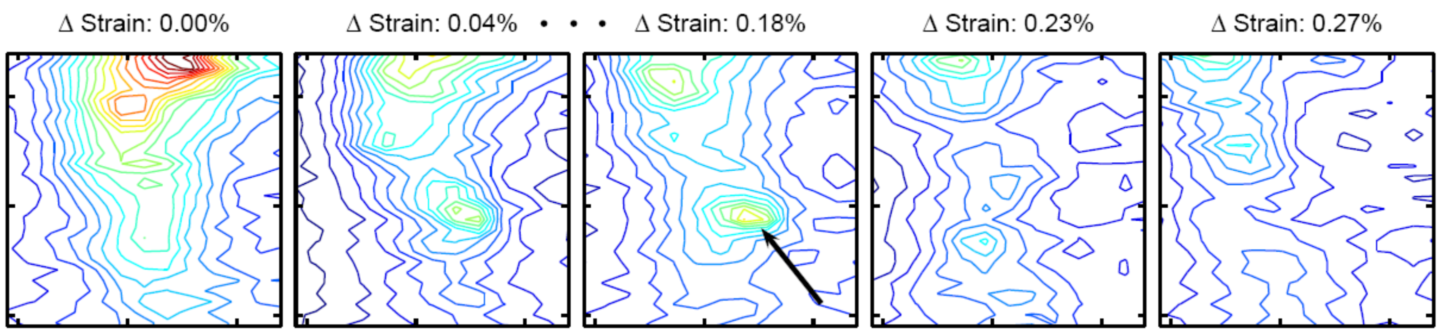
B. Jakobsen et al., *Science* **312** (2006) 889-892

Subdivision

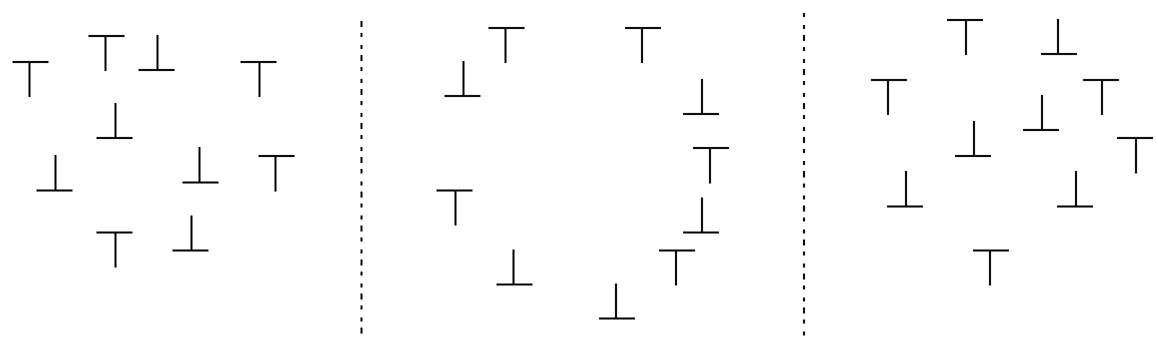
Old model:



Data:



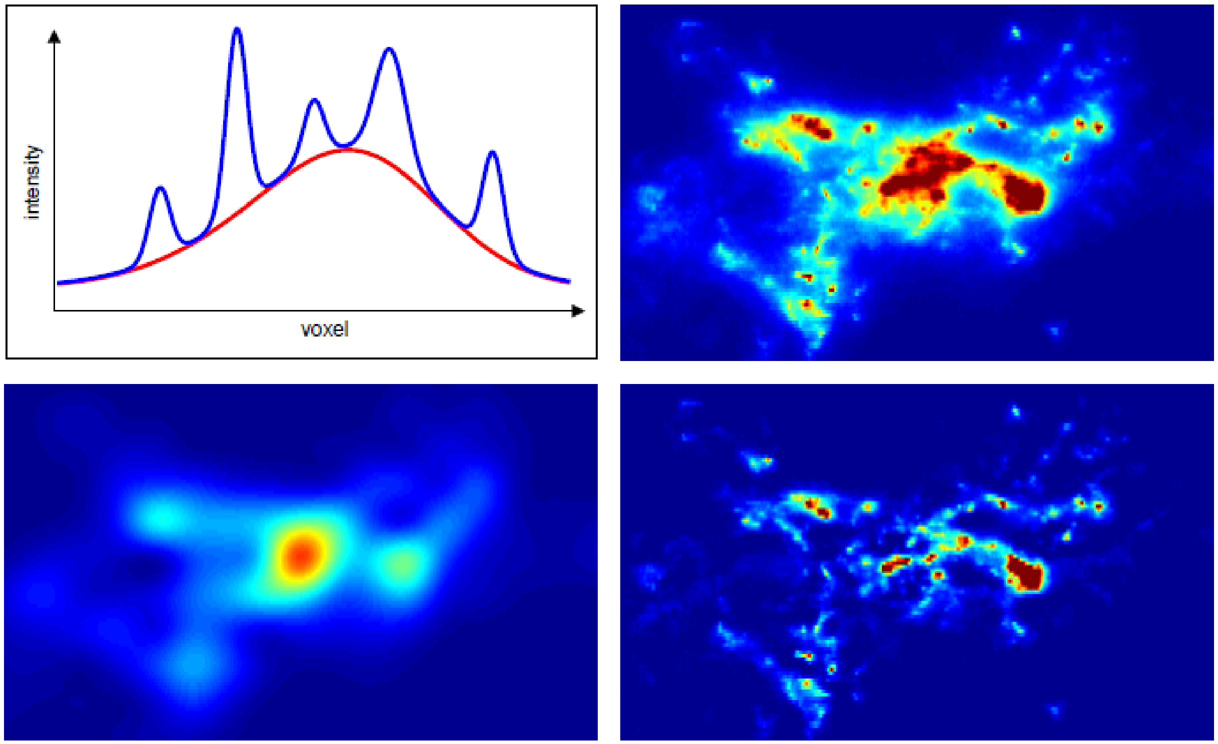
New model:



B. Jakobsen, H.F. Poulsen, U. Lienert, J. Almer, S.D. Shastri, H.O. Sørensen, C. Gundlach, W. Pantleon
Science **312** (2006) 889-892

Automated partitioning

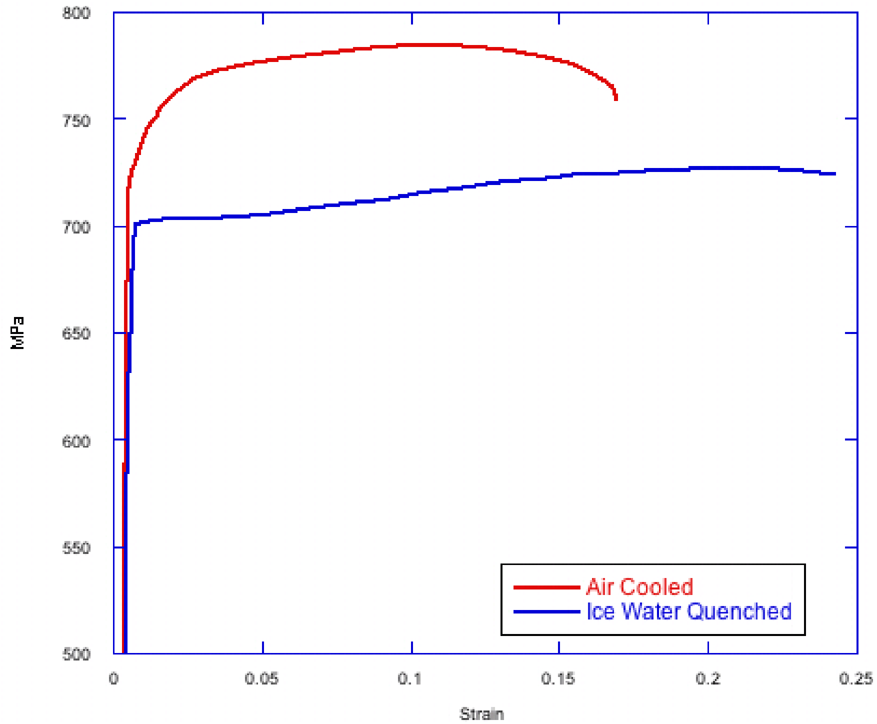
- Azimuthal intensity projection
- Cloud: bi-cubic spline with asymmetric cost function



Medium & high resolution far-field: Ti-7Al

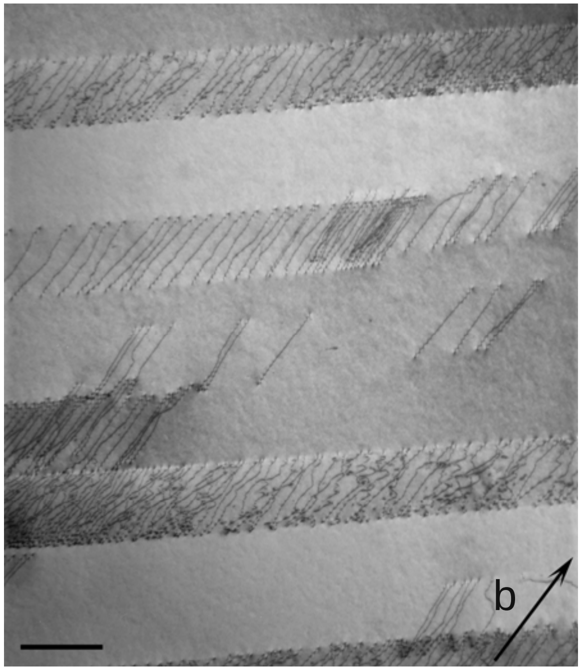
- M. Miller (Cornell), M. Brandes, M.J. Mills (Ohio State), J. Bernier (LLNL)

- High strength & low density
- Large strength to stiffness ratio
- Elastically fairly isotropic
- Al alloying (7 wt%):
  - Suppresses deformation twinning
  - Promotes <a> slip (b = <1 1 -2 0>) on basal, prism, pyramidal planes.
  - Short Range Order (SRO) domains => planar shear band formation
- Air Cooling **AC**: SRO
- Ice Water Quenching **IWQ**: SRO suppressed



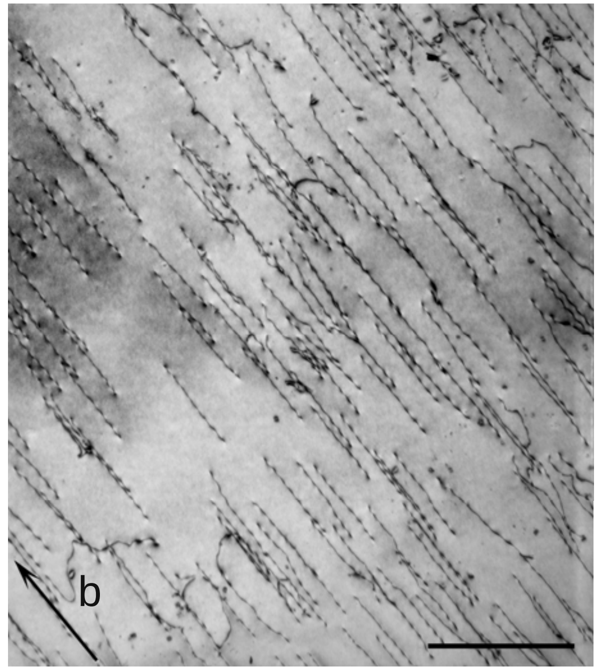
TEM

AC



1 μm

IWQ

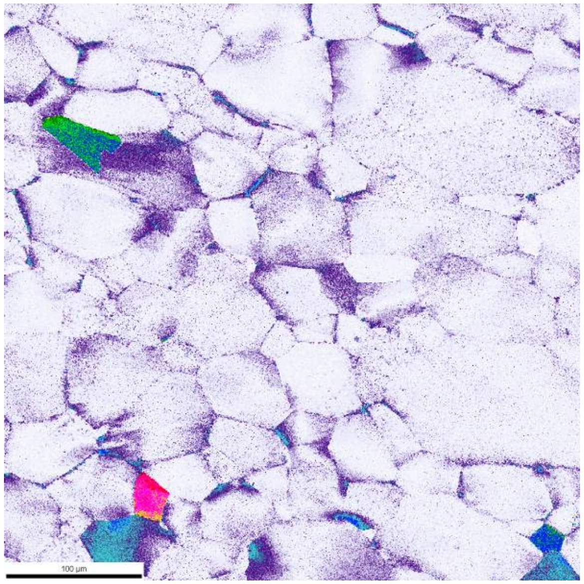


1 μm

- Post-mortem TEM on internal ND, TD plane. $\vec{g} = \{1\bar{1}01\}$
- AC: shear bands of planar <a> screw dislocation pile ups. Contrast variation across shear bands.
- IWQ: homogeneously distributed <a> screw dislocations.

EM: Grain average misorientation

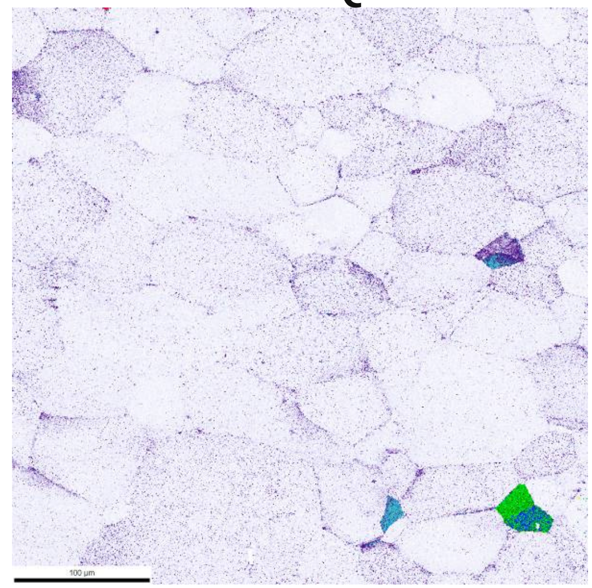
AC



100 μm

Fraction		Fraction
0.845	0 – 0.636	0.691
0.143	0.636 – 1.273	0.260
0.006	1.273 – 1.909	0.032
0.001	1.909 – 2.546	0.006
0.002	2.546 – 3.182	0.006
0.002	3.182 – 3.819	0.001
0.000	3.819 – 4.455	0.000
0.000	4.455 – 5.092	0.002

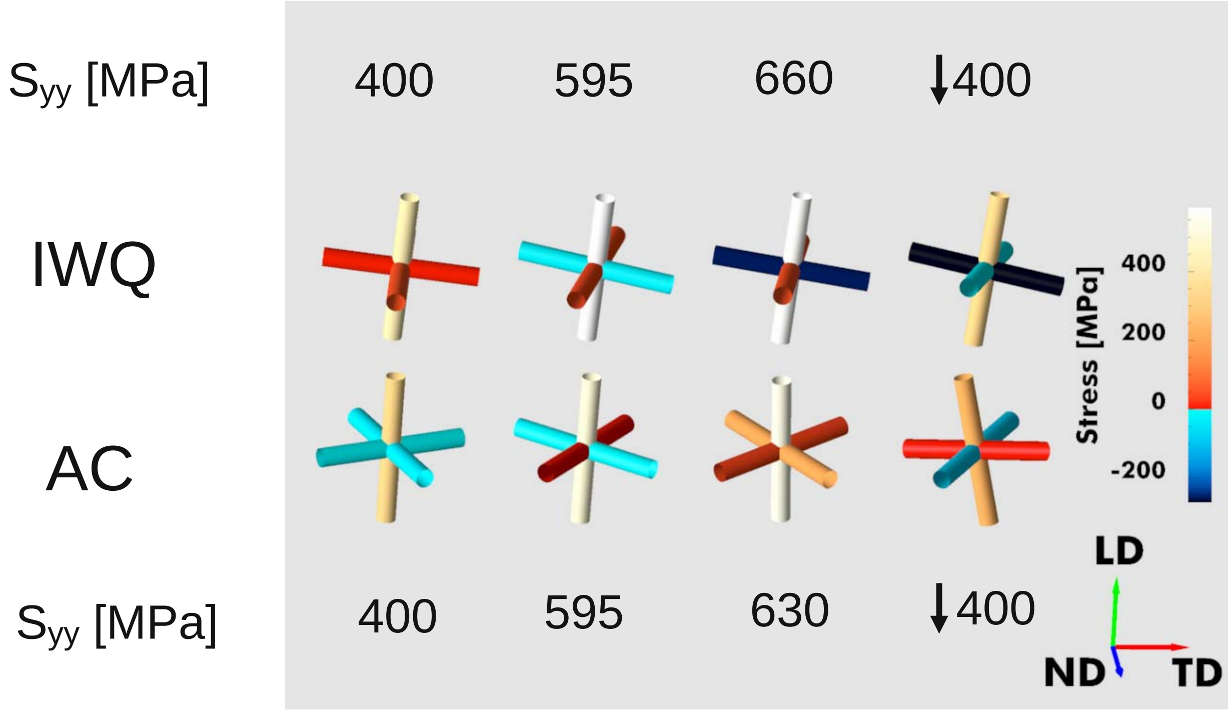
IWQ



100 μm

- Tensile deformation
- Post-mortem SEM EBSD on internal LD, ND plane.
- AC: orientation gradients at grain boundaries of up to 1°.
- No twinning. Grain size about 75 μm equiaxed.

Single grain stresses



- AC & IWQ: Significant tri-axial components
- AC stress rotates more than IWQ
- AC & IWQ: Significant tri-axial residual stress after deformation

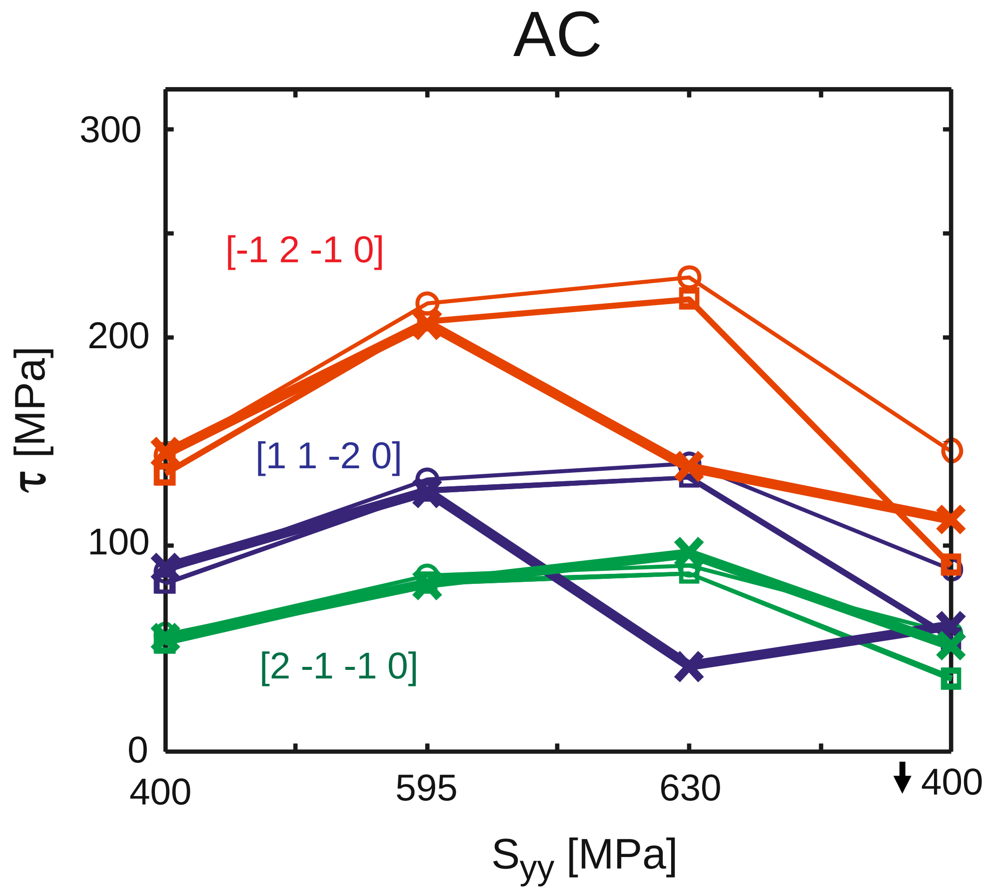
AC Resolved Shear Stress

- Basal <a> slip

$$\tau = S_{yy} \cos\phi \cos\lambda$$ (open circle marker)

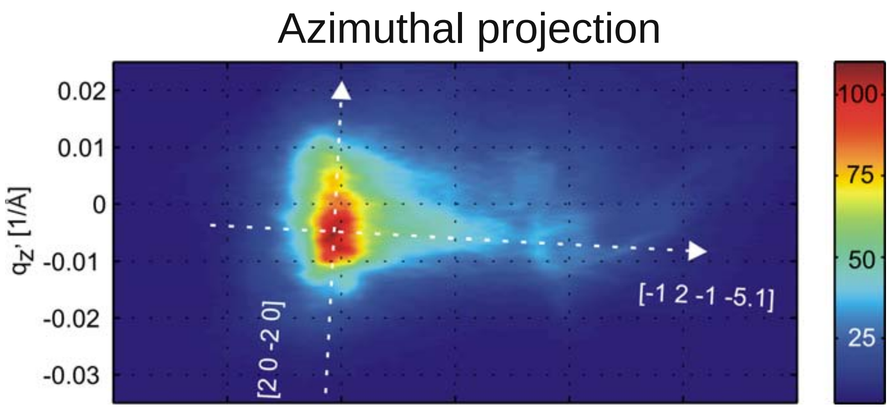
$$\tau = \sigma_{yy} \cos\phi \cos\lambda$$ (open square marker)

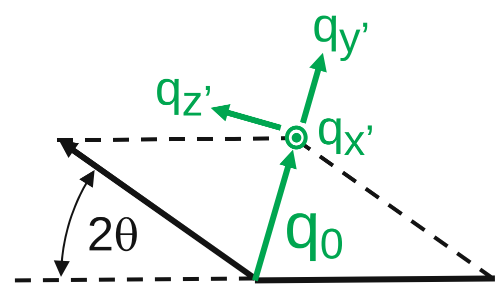
$$\tau = s_i \cdot \sigma_{ij} \cdot n_j$$ (cross marker)



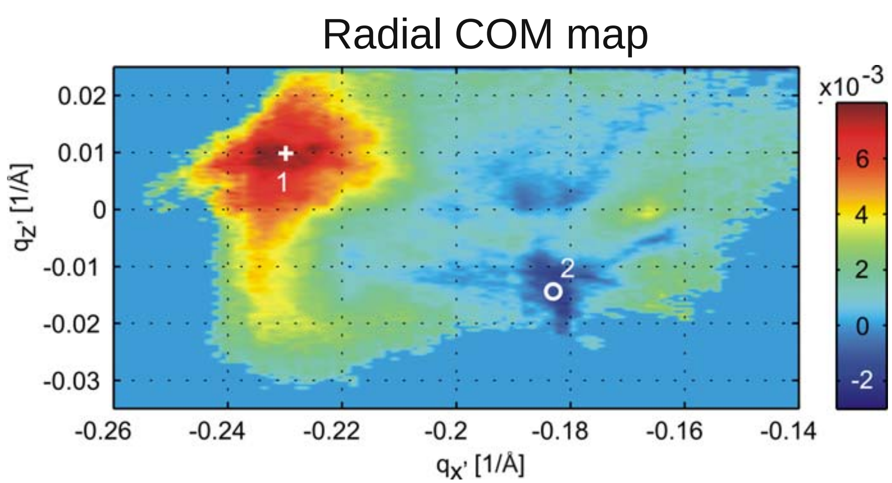
- Residual stresses upon unloading
- Drop in RSS on most stressed slip systems upon plasticity

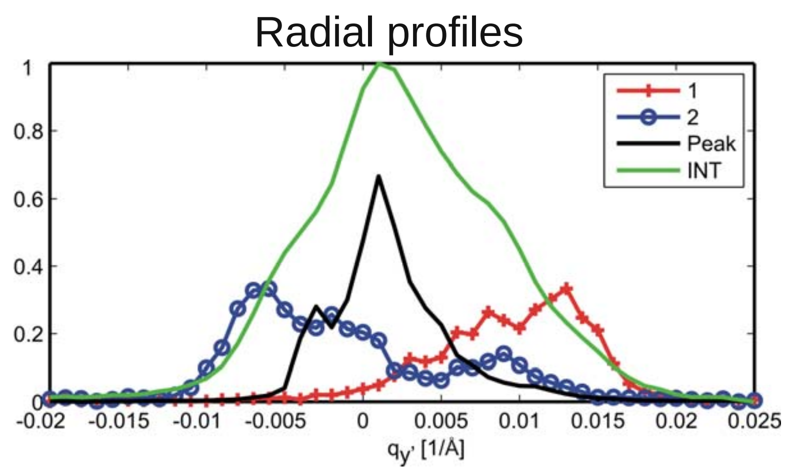
Reciprocal space map AC, (-1 2 -1 2), 400 MPa (unloaded)





$$\varepsilon_{yy'} = -\frac{q_{y'}}{q_0}$$

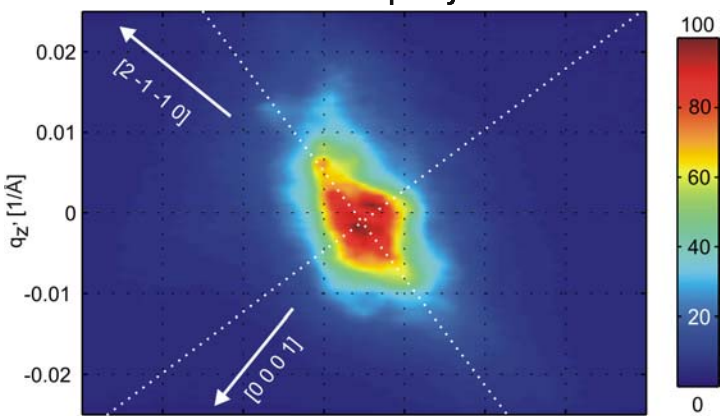


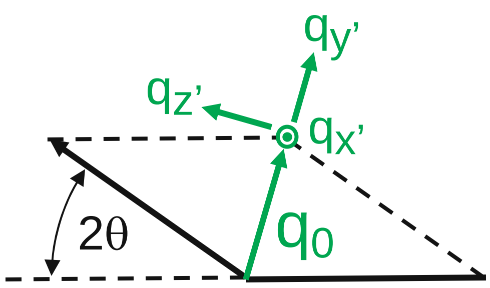


- large azimuthal tail perpendicular to rotation axis
- large strain gradient perpendicular to rotation axis

Reciprocal space map IWQ, (0 2 -2 0), 660 MPa

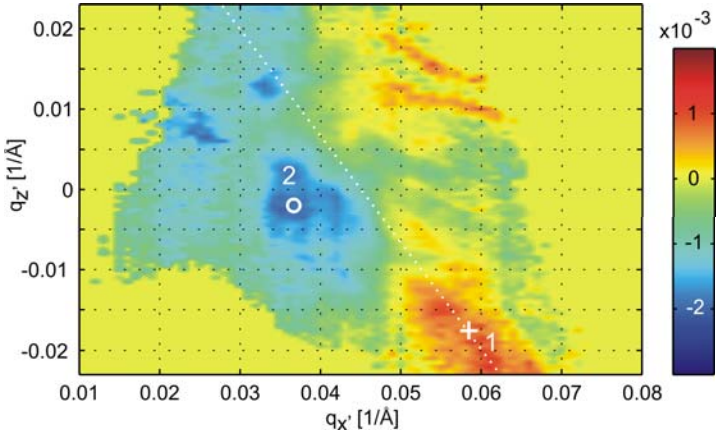
Azimuthal projection



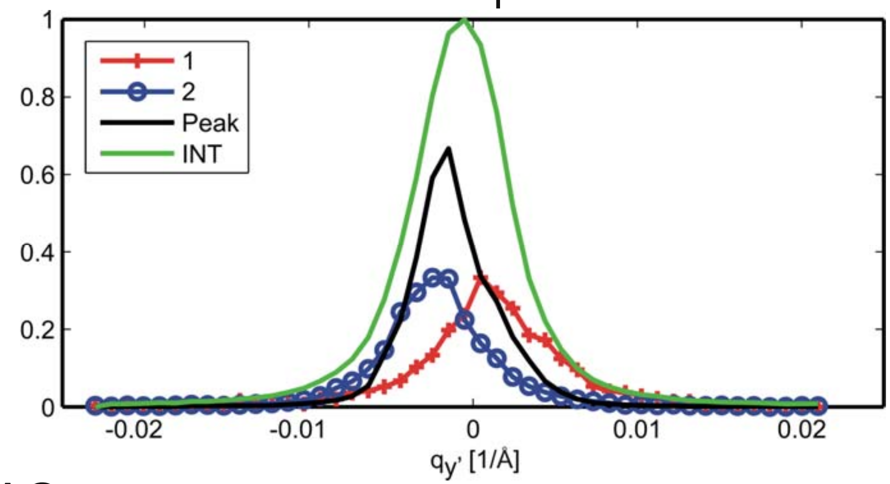


$$\varepsilon_{yy'} = -\frac{q_{y'}}{q_0}$$

Radial COM map



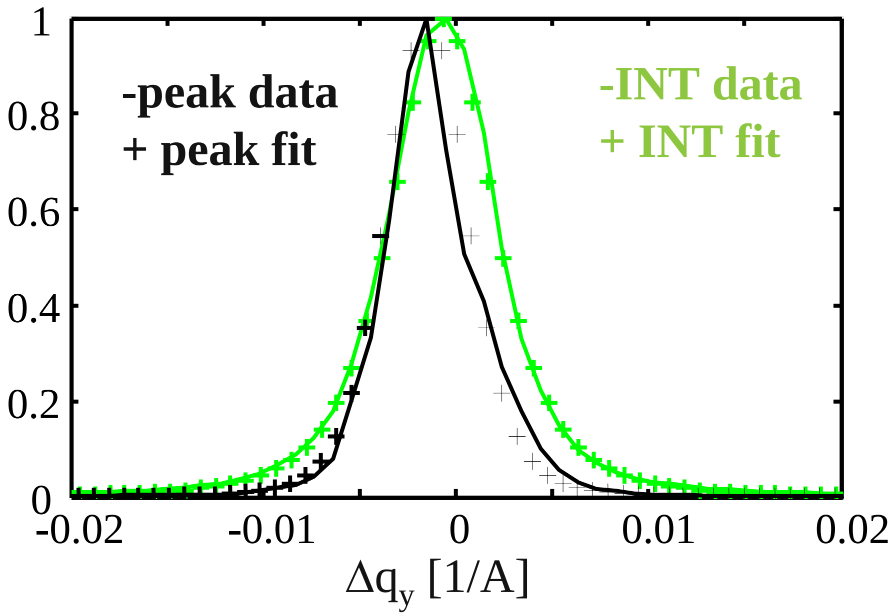
Radial profiles



- IWQ strain gradient smaller than AC
- IWQ & AC: Azimuthal widths larger than radial
- IWQ & AC: Comparable azimuthal FWHM and anisotropy

IWQ Radial peak profile fitting

- Restricted random dislocation distribution (Wilkens, 1970)
  - Negligible size broadening
  - Elastic isotropy
- **b** = [1 -2 1 0]/3 screw dislocations, **g** = [0 2 -2 0]



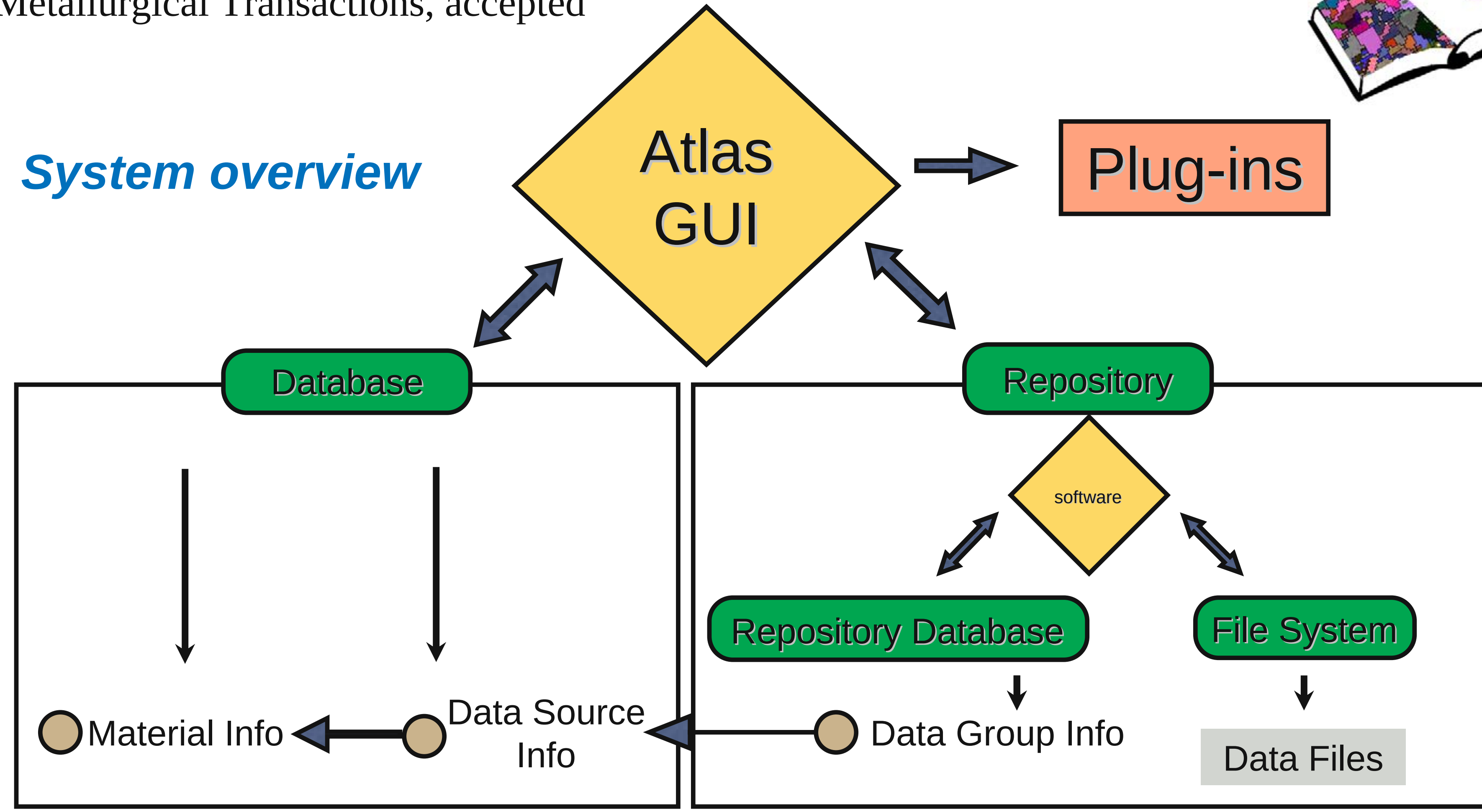
	peak	INT
ρ [m^{-2}]	**$0.85 \cdot 10^{14}$**	**$3.1 \cdot 10^{14}$**
M	**4.13**	**1.149**
Re [nm]	**470**	**65**
Δq_size [1/A]	**0.0006**	**0.004**
Δq_{FWHM} [1/A]	**$5.11 \cdot 10^{-3}$**	**$6.43 \cdot 10^{-3}$**

- ρ(TEM): 10^{14} m^{-2} (Conrad 1981, Ghosh 2008)
- Significant differences between peak & integrated profiles

D.E. Boyce, P.R. Dawson, M.P. Miller, “The Design of a Software Environment for Organizing, Sharing and Archiving Materials Data”, Metallurgical Transactions, accepted

3D Materials Atlas

System overview



The primary database stores information about the materials and the data sources. The exact structure (schema) is flexible. ***The only link between primary database and the repository is through the Data Group/Data Source association.***

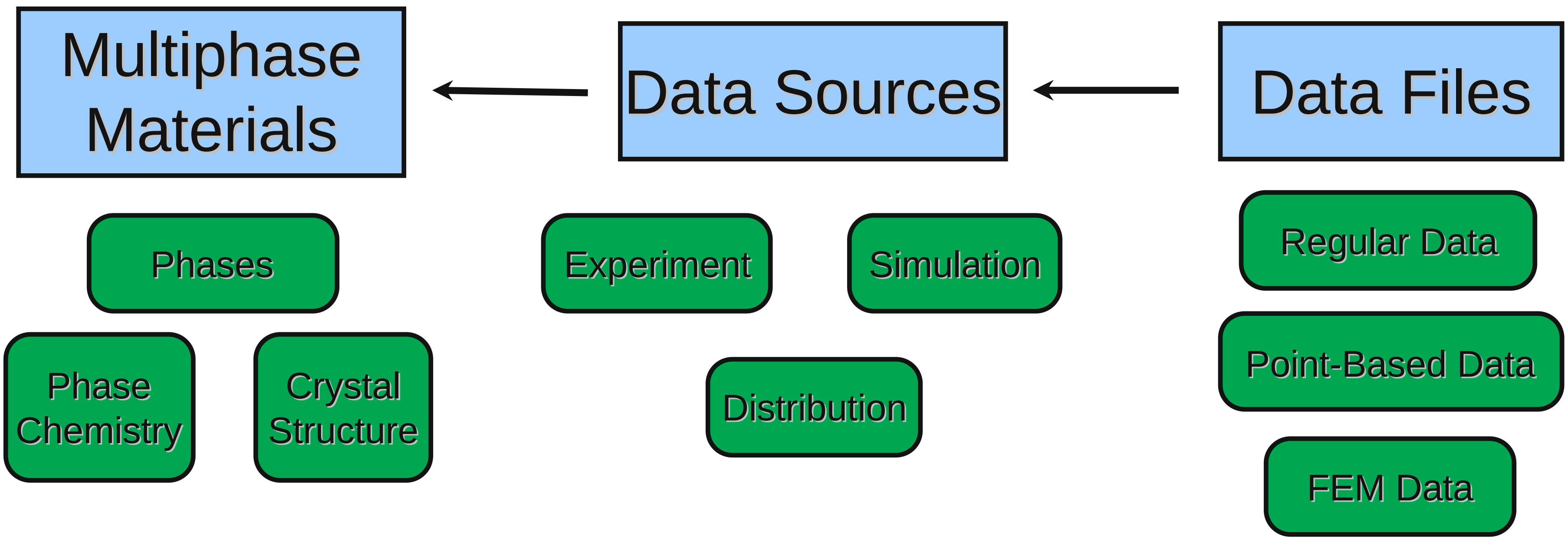
The repository uses its own database to store descriptive information about the actual data sets. It stores the actual data on its file system. The repository need not be on the same machine as the primary database.

metadata

The exact structure of the metadata database is **flexible**. Changes can be easily accommodated through a configuration file.

data

The data file formats are **extensible**. (add new formats, enhance old ones)



Multiphase materials are defined by their phases, which in turn are defined by a chemistry and crystal structure.

Data sources refer to the process which generated the data set. A ***distribution*** is a statistical representation of a material attribute and can be used for generating digital microstructures.

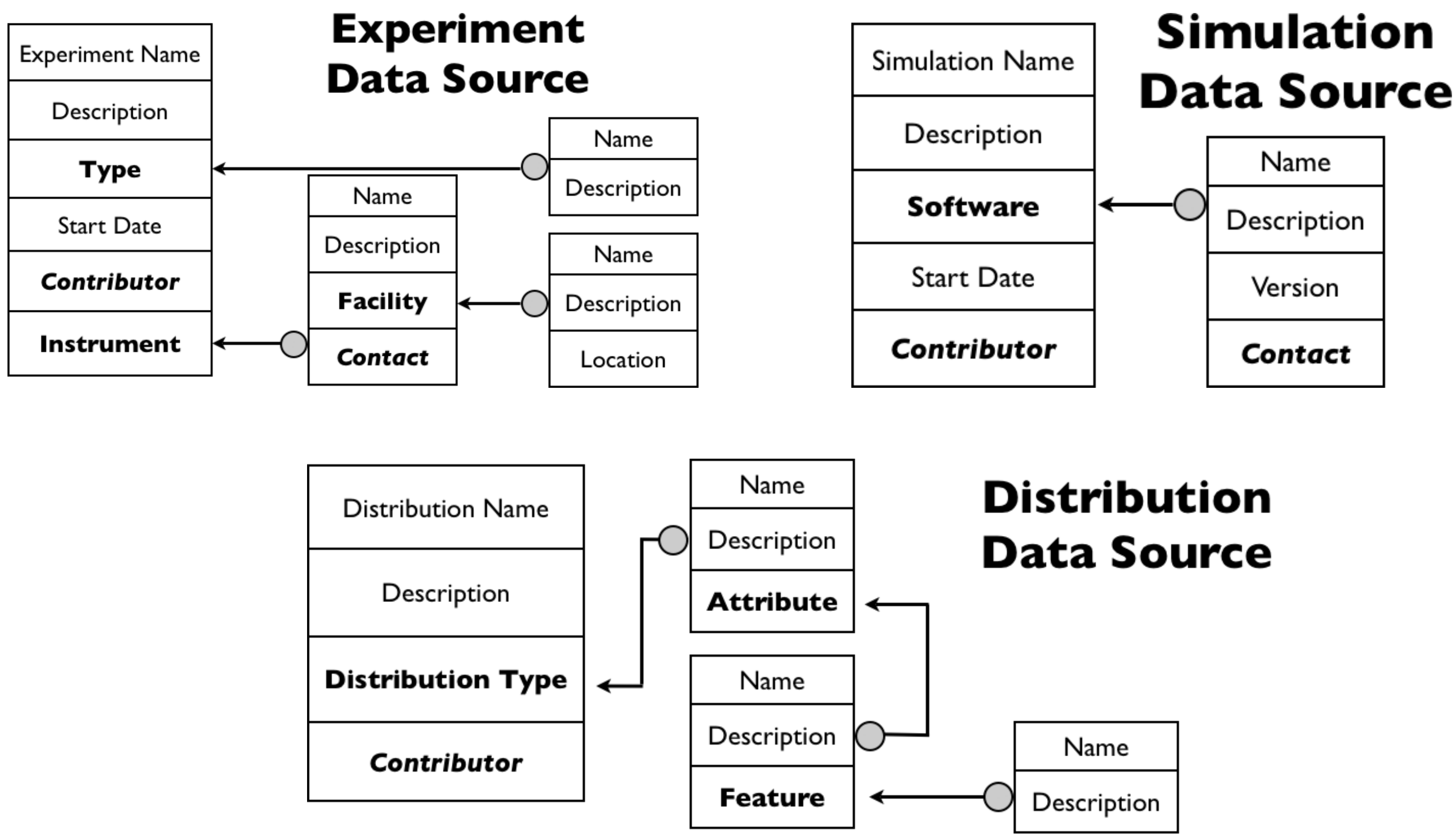
Data is entered in the form of ***Data Groups***, collections of related files. Each of the three data group types has a customized entry form providing detail about the data file contents.

3D Materials Atlas System Components

Data Source Information

Key to the design is the Data Source Information:

- links the subject information to the primary data files
- archives critical 'pedigree' data
- facilitates additional types of searches regarding data source attributes



Summary

- Near field
  - Forward modeling Monte Carlo algorithm
  - Highly parallelizable for binary images
  - Use of intensity, deformed materials
- Far field
  - Increase number of grains
  - Box scan & strain scans
  - Strain/stress evaluation software
  - Using Fable & Matlab
  - Started using GUI (Ken Evans)
- High resolution reciprocal space mapping
  - Automate grain alignment
  - Automated partitioning
  - ODF reconstruction
- Combine techniques