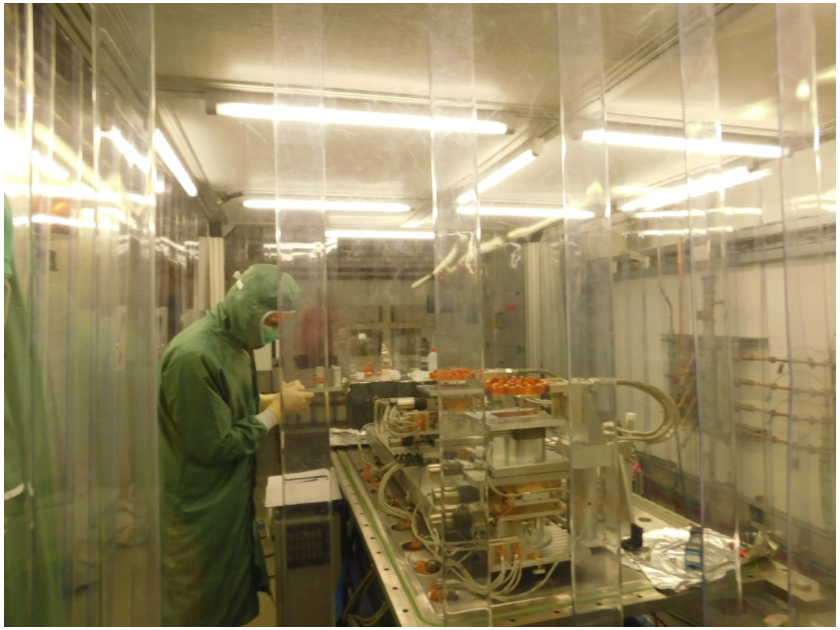
Single-grain diffraction data analysis needs

Ulrich Lienert, Thomas Bäcker

P21.2 Swedish Materials Science beamline
First beam anticipated late May 2018



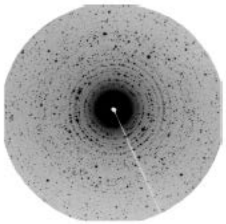
HELMHOLTZ | ASSOCIATION

DESY

Single grain diffraction: Motivation

- Grains are fundamental units of microstructure
  - Crystallographic phase
  - Shape
  - Orientation
  - Stress/strain partitioning
- Anisotropic elastic & plastic properties
- Grain – grain interactions
- Beyond fiber-averaging
  - Non-linear quantities (peak profiles)
- Non-destructive, bulk penetration
- *In situ* 3D observations (4D)

Multicrystal



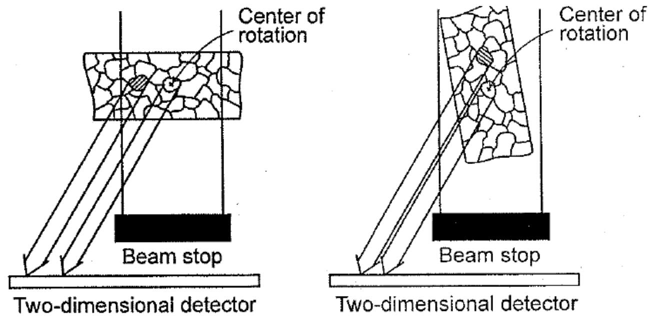
Powder

Real & orientation space

> Position space $\mathcal{R}^3$

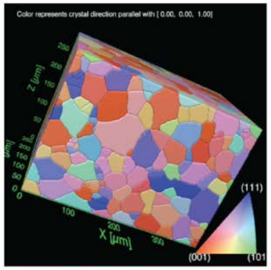
- Precession around rotation axis
- Shift of detector position

(Margulies *et al*., Acta Mater., 2001)

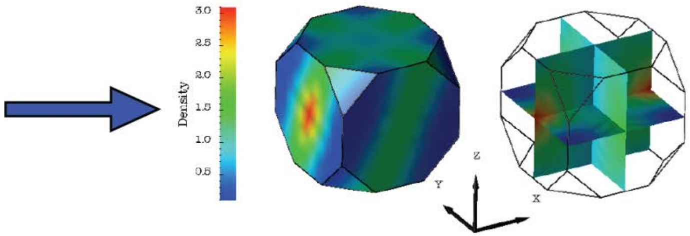


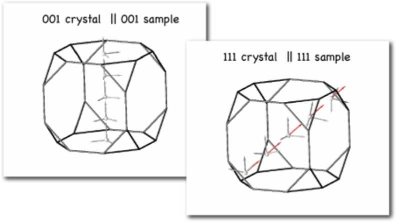
> Orientation space SO(3)

- Rodrigues space
- Orientation: $\boldsymbol{r} = \boldsymbol{n}\,tan\frac{\phi}{2}$



β21S lattice orientations from serial section (NRL)





- Fibers are straight lines connecting orientations that differ by rotation about one axis

(Dawson *et al*., Cornell)

> Projection properties of position-orientation space

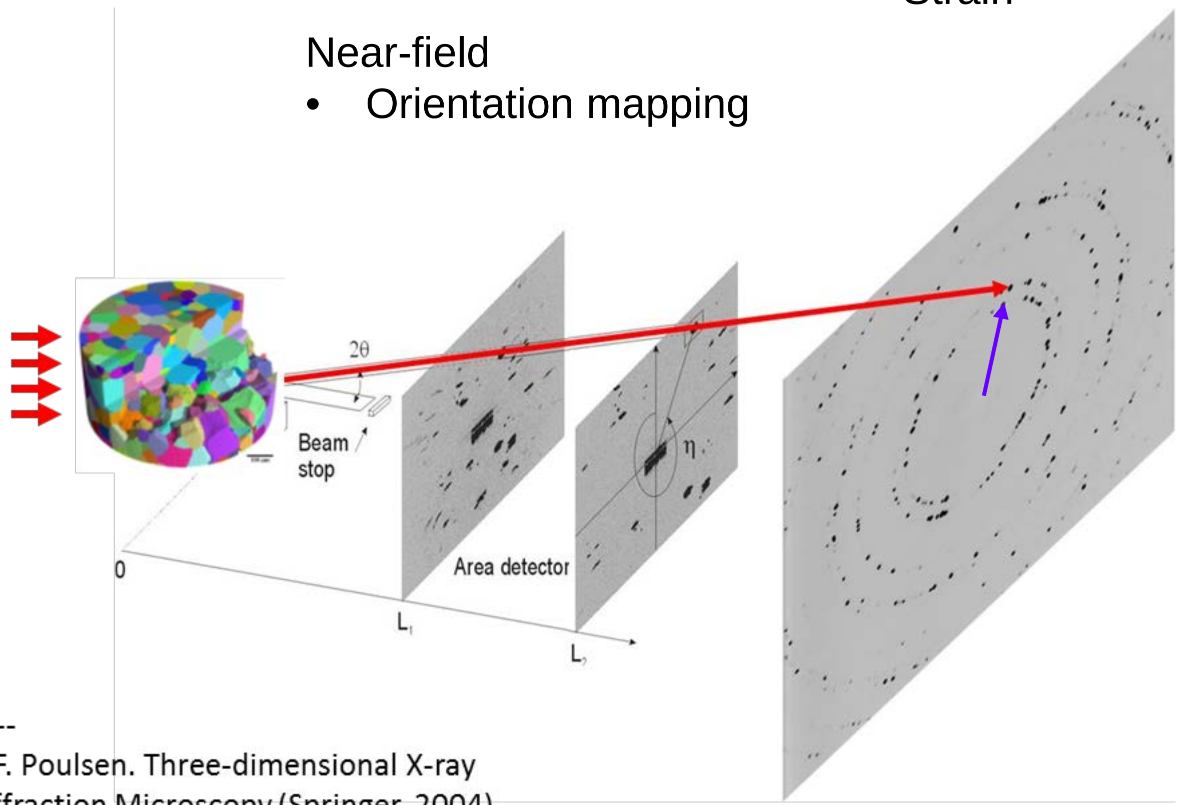
- Indexing & reconstruction strategies (Poulsen, 3DXRD, 2004)

Single grain diffraction: Experimental geometry

> P21.2, P07(HZG), P02.2, P03(HZG), P06

Far-field: grain averaged
- Position
- Orientation
- Strain

Near-field
- Orientation mapping



Strain
- radial peak shift
- Sensitivity 10^{-4}

$$\boldsymbol{n}_{hkl} \cdot \boldsymbol{\epsilon} \cdot \boldsymbol{n}_{hkl} =$$

$$\frac{\Delta d}{d} = -\frac{\Delta\theta}{tan\theta} = -\frac{\Delta G_{hkl}}{G_{hkl}}$$

H.F. Poulsen. Three-dimensional X-ray Diffraction Microscopy (Springer, 2004)

Detectors: features & data rates

Varex XRD 4343 CT
250 MB/s

- Cheap
- 8 M pixel
- Rolling shutter
- Low dynamic range
- High peak intensity
- Lagging
- Special real time trigger mode
- Contiguous area coverage

2M Pilatus CdTe
1000 MB/s

- Expensive
- 2 M pixel
- Readout time
- High dynamic range
- Low peak intensity
- No lagging
- Real time triggering
- Dead space between modules

specific detectors

\+

Large & focused beams

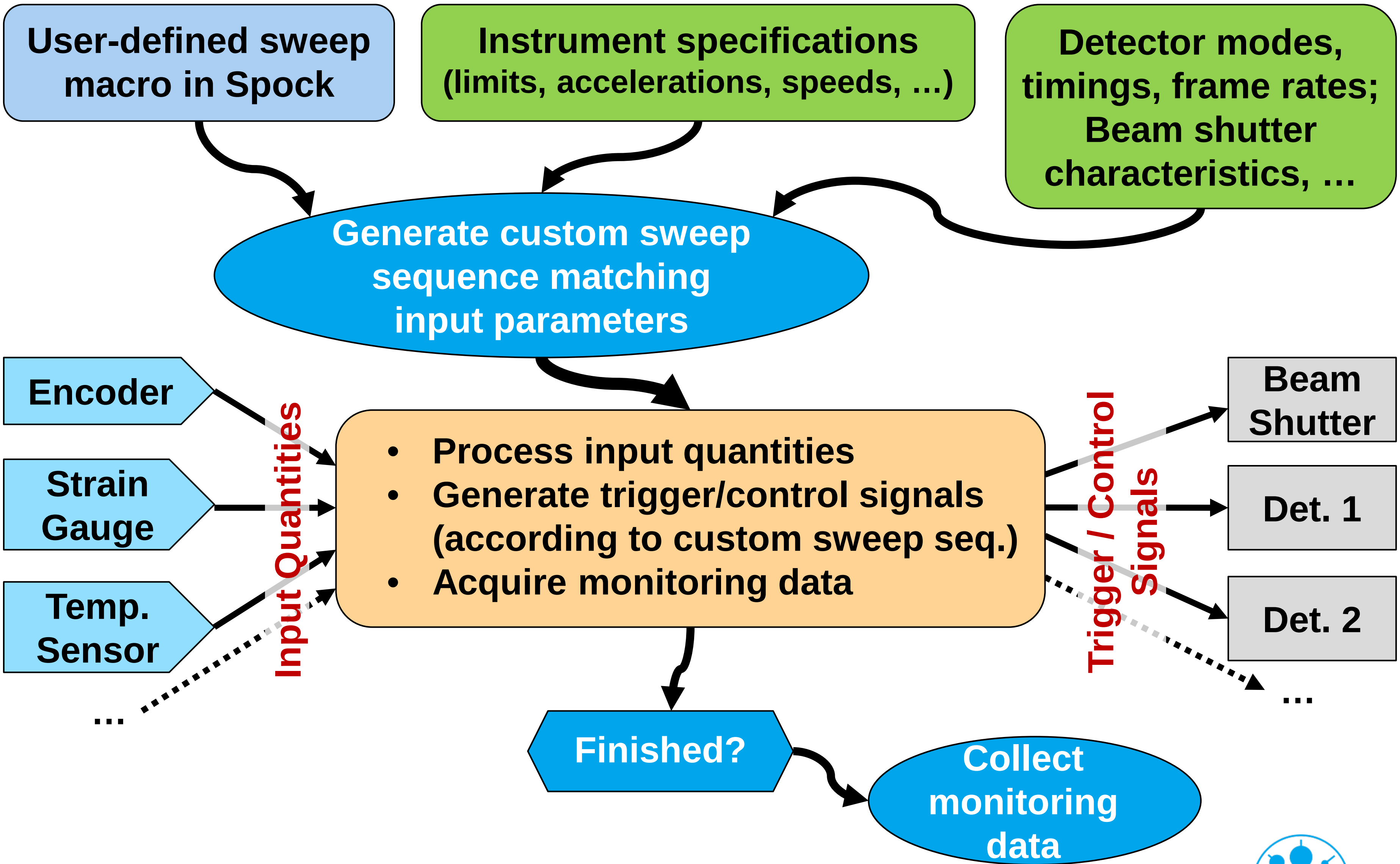
\+

In situ processing

↓

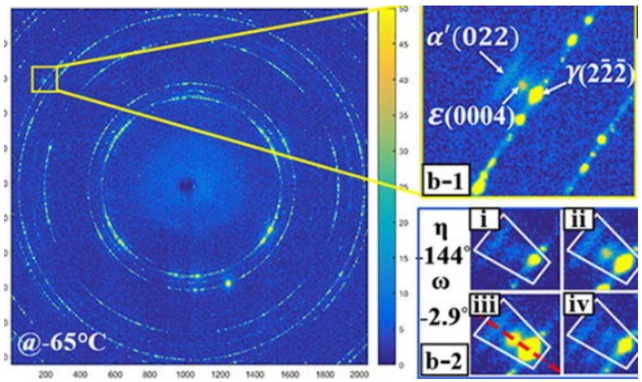
Flexible real time synchronization of
- detectors
- fast shutter
- sample positioning
- Processing parameters

Synchronous Sweep Controller

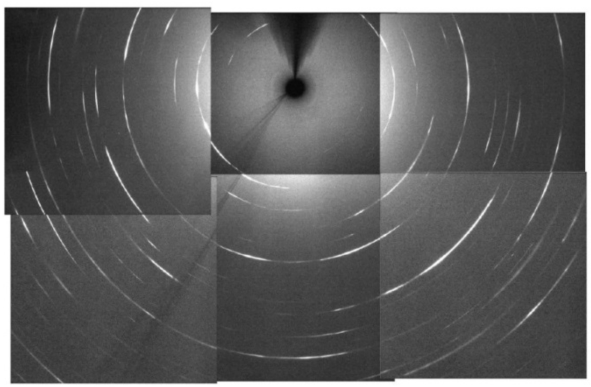


Segmentation

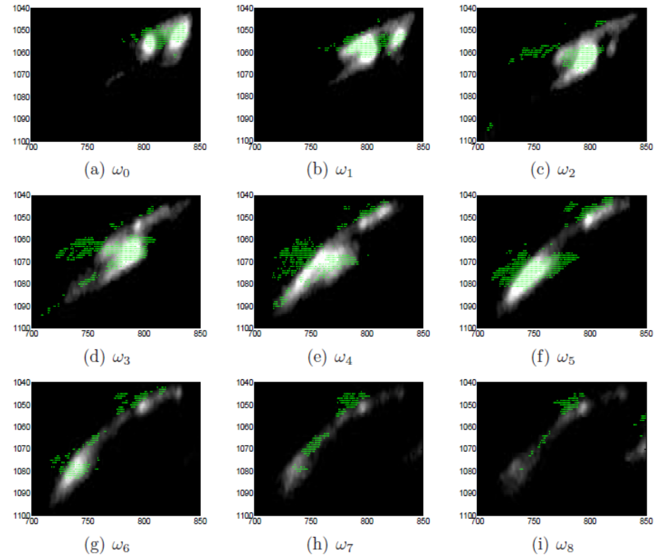
- > Epitaxial growth



- > Azimuthal spot overlap

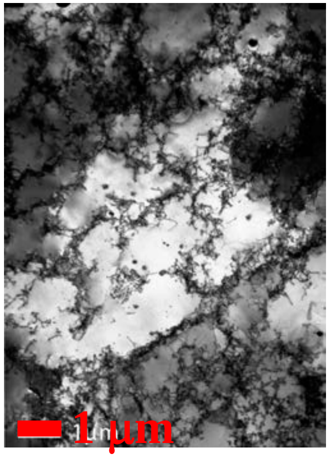


- > Diffuse intensity distribution

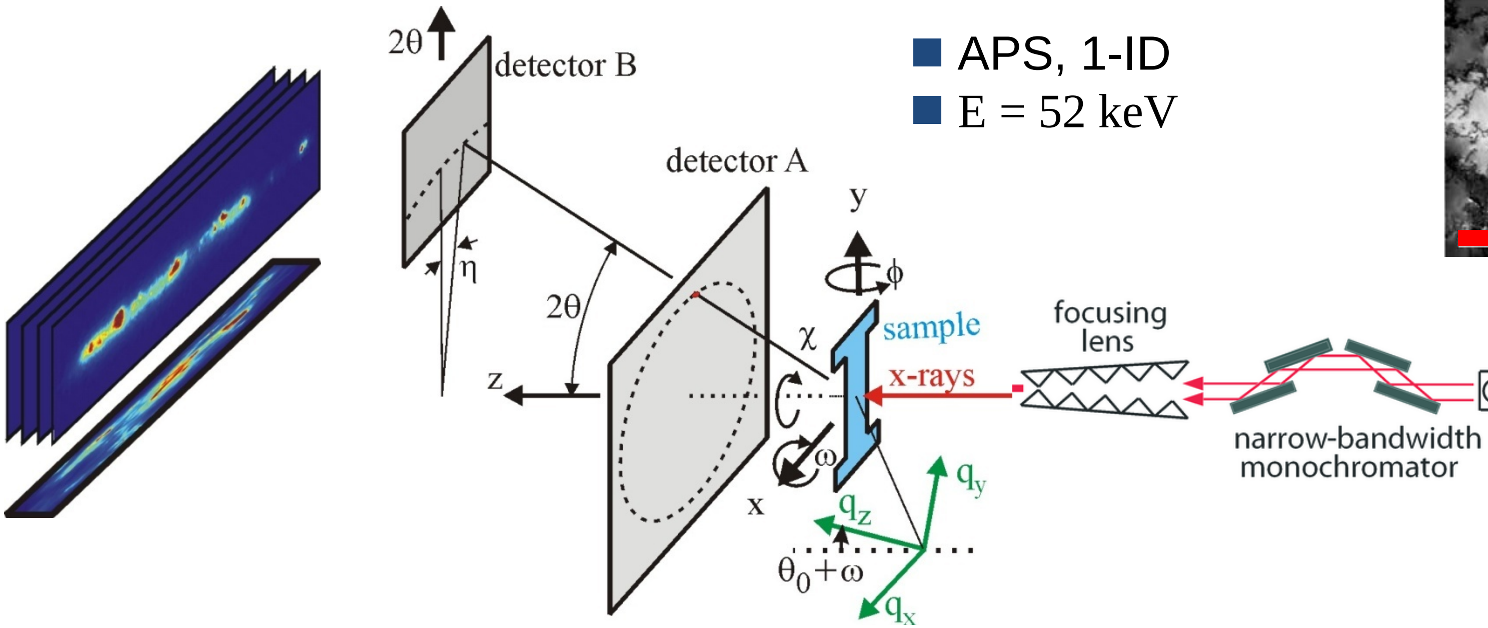


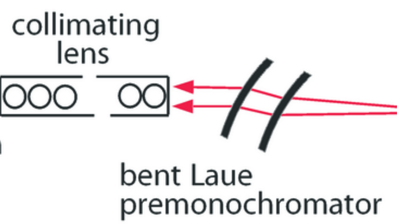
- > Segmentation: automated, 3D
- > Forward modeling

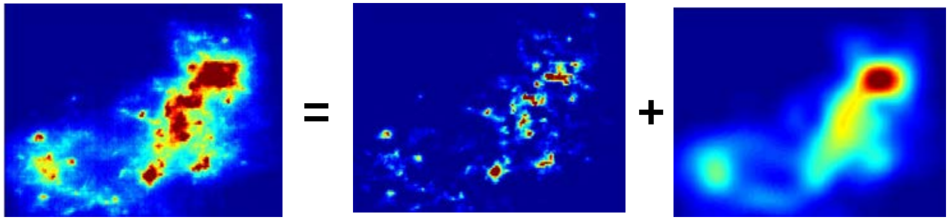
Subgrain - cellwalls



- APS, 1-ID
- E = 52 keV







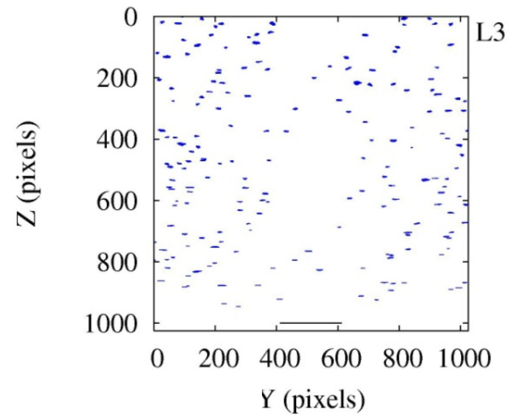
- orientation contrast
- 1D scan maps 3D reciprocal space
- subgrain volume, strain component
- subgrain evolution
  during tensile deformation

DTU

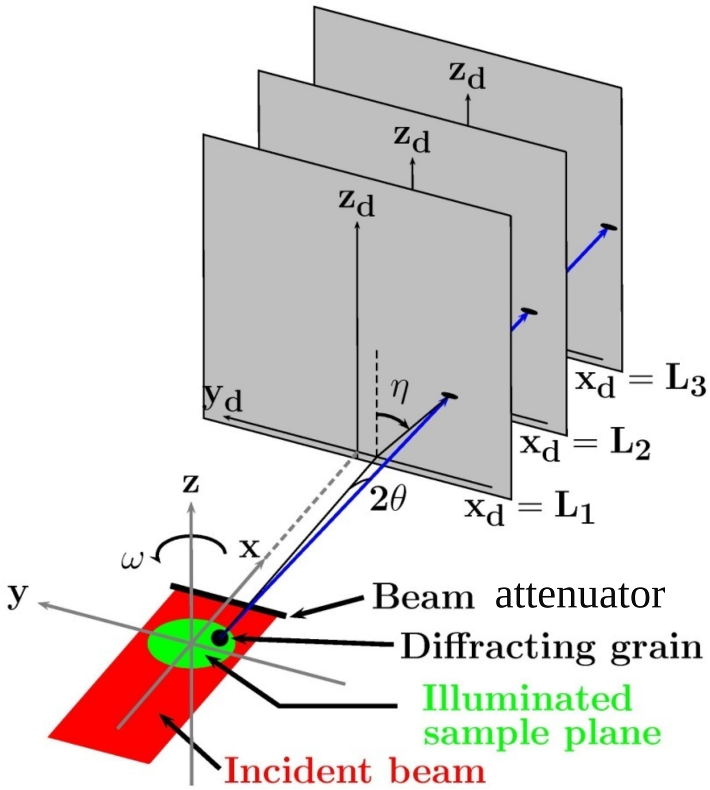
Argonne NATIONAL LABORATORY

Near-field orientation mapping: experimental

- C. Hefferan, S.-F. Li, J. Lind, R. Pokharel, R.M. Suter (CMU)
- 1-ID-B station
- E = 65.4 keV, 2.5 μm high line focus
- Typically 1 mm diameter samples
- ω-range ±90 °, ω-interval 1°
- 0.1 ° orientation resolution
- Integration time 1-3 s
- 2-3 L distances (5-9 mm)
- Detector
  - 2k x 2k pixels, 12 bit
  - 1.48 μm pixel size
  - 3 x 3 mm field of view
  - 25 μm LuAG:Ce
  - 4 μm resolution
- Annealing, tensile deformation

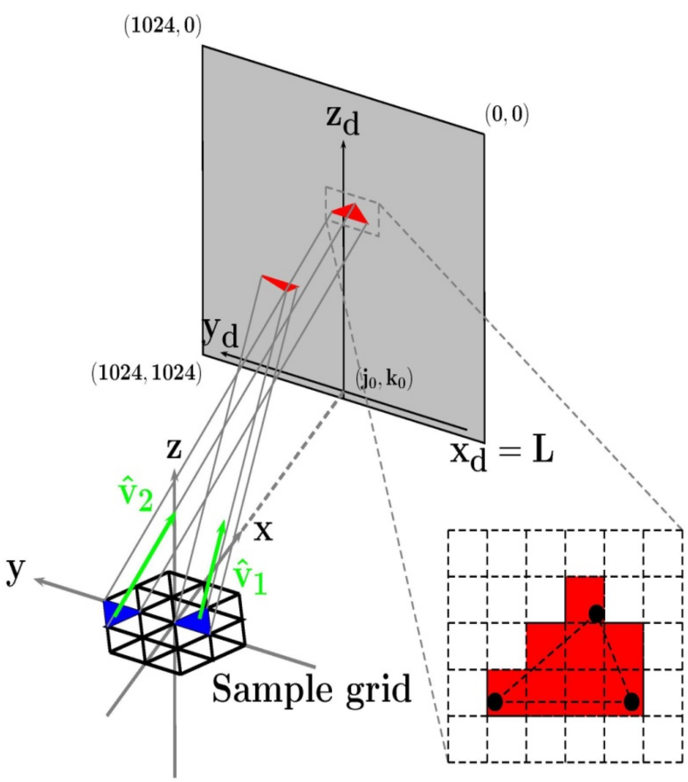


500 grains



Suter et al, RSI 2006
Lauridsen et al, Appl Cryst 2001

Near-field orientation mapping: reconstruction



Suter et al, Eng Mat & Tech 2007
S.F. Li et al., J. Appl. Cryst., 2013

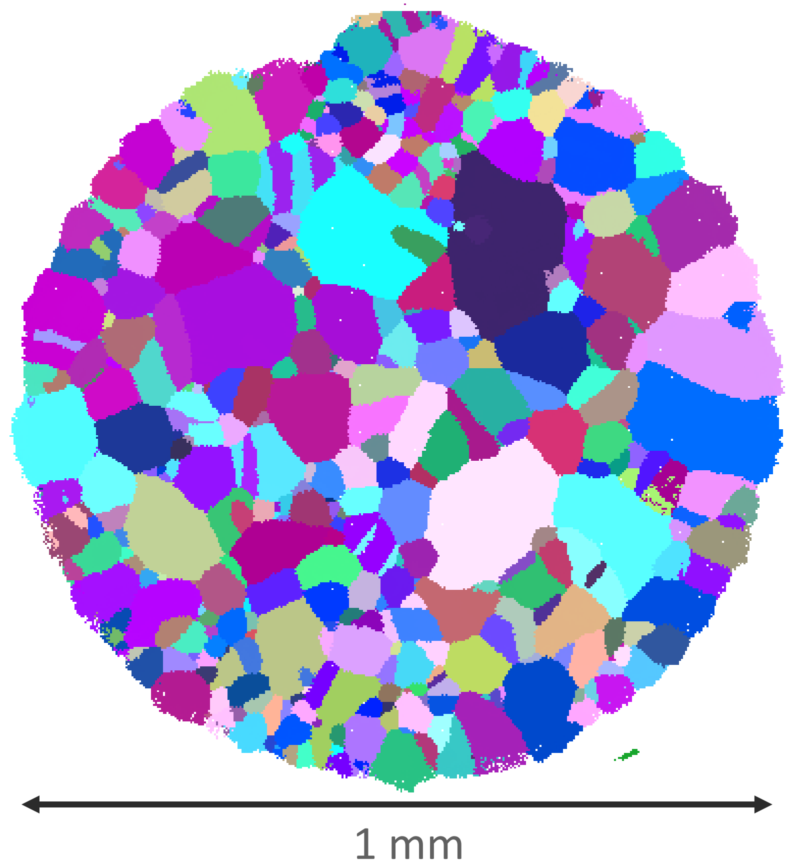
- Preprocessing & thresholding (APS cluster)
- Geometry determination from fitting Au-wire calibration sample
- GridFTP to CMU
- “brute force” orientation search
  - Test grid element orientation to maximize diffraction with binary data
  - Monte Carlo optimization
  - 4 µm **independent** grid elements
  - 1 min/element/core
  - TeraGrid: 1000 cores
- “space filling” algorithm
  - Scales with number of grains not elements
- Preliminary output during run
- General user implementation
- Intensity fitting

Near-field: High purity nickel

One of 42 layers,
156μm^3 volume

- ~400 grains/layer
- ~45 μm ave diameter

Black lines: mesh element neighbors with > 0.1 deg misorientation

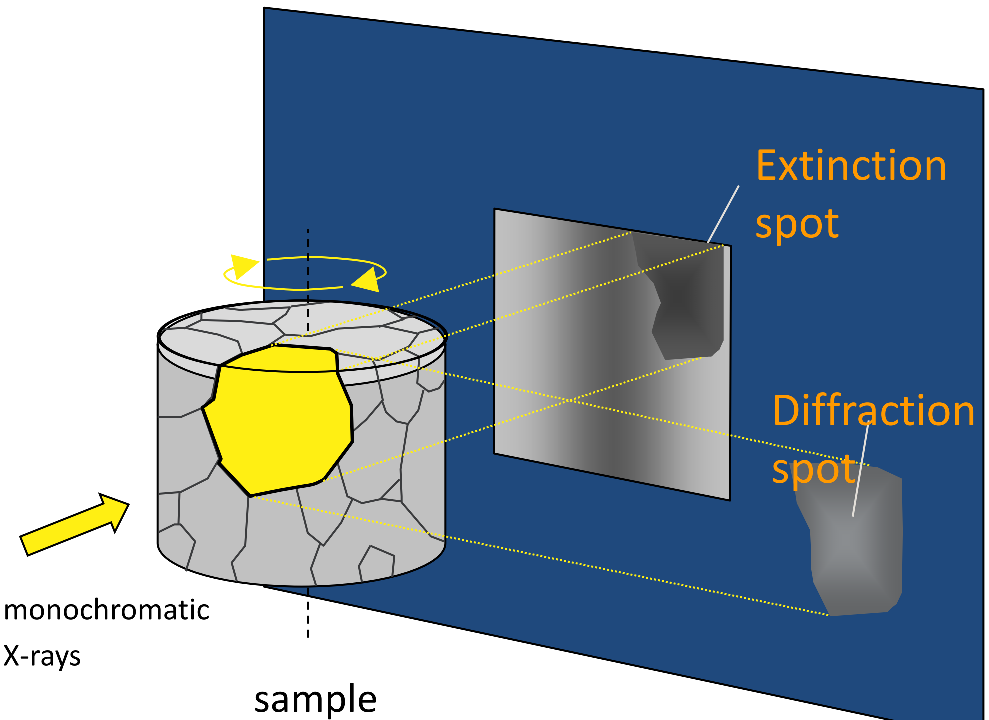


- Sample has well ordered grains
- Experimental ~0.1 deg orientation resolution
- Smallest grains ≈ 10 μm

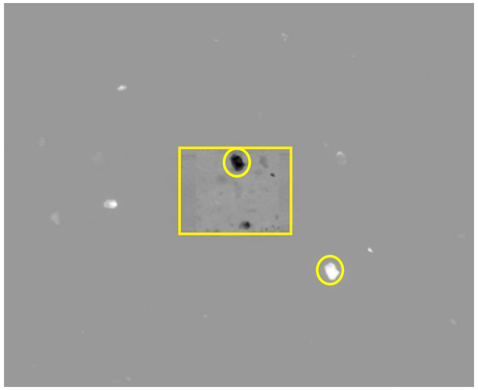
Diffraction contrast tomography (DCT)

INSA-Lyon, Manchester, ESRF

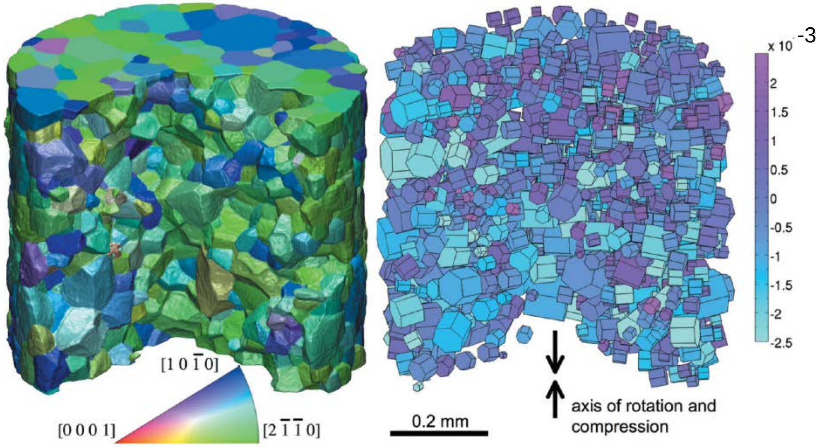
- E: 15-50 keV



Raw data



Ti loaded in compression

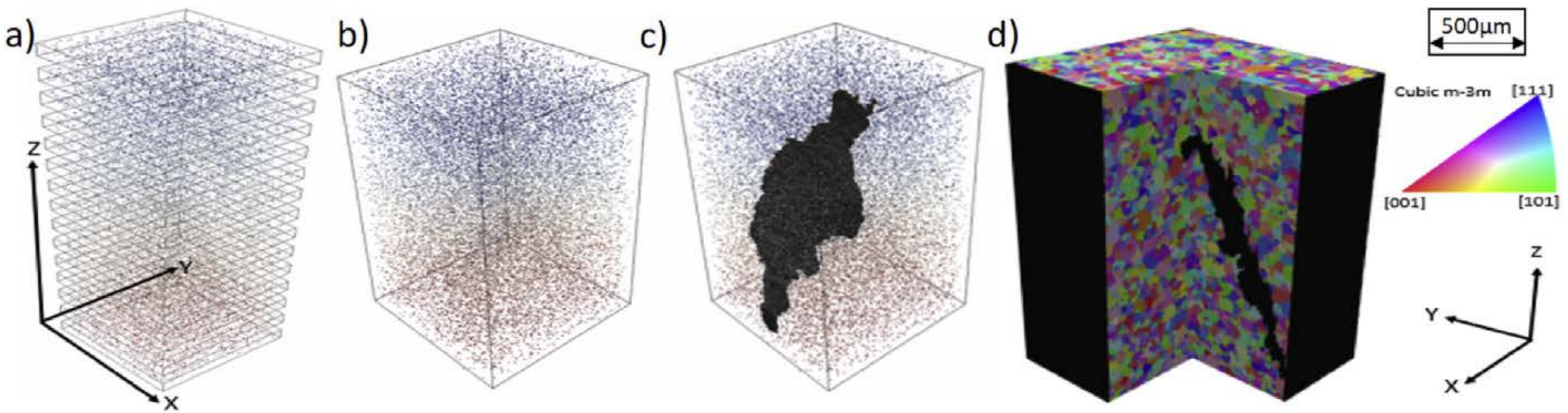


- Average grain orientation (& lattice strain)
- Grain boundary accuracy > 1 μm
- Grain mosaicity < few degrees

G. Johnson, A. King, M. G. Honnicke, J. Marrow, W. Ludwig. J. Appl. Cryst. (2008). **41**, 310-318,
W. Ludwig, P. Reischig, A. King, M. Herbig, E. M. Lauridsen, G. Johnson, T.J. Marrow, J.Y. Buffiere. Rev. Sci. Instr. (2009), **80**, 033905
P. Reischig, A. King, L. Nervo, N. Vigano, Y. Guilhem, W.J. Palenstijn, K.J. Batenburg, M. Preuss, W. Ludwig, Appl. Cryst. (2013), **46**, 297.

Far-field & tomography

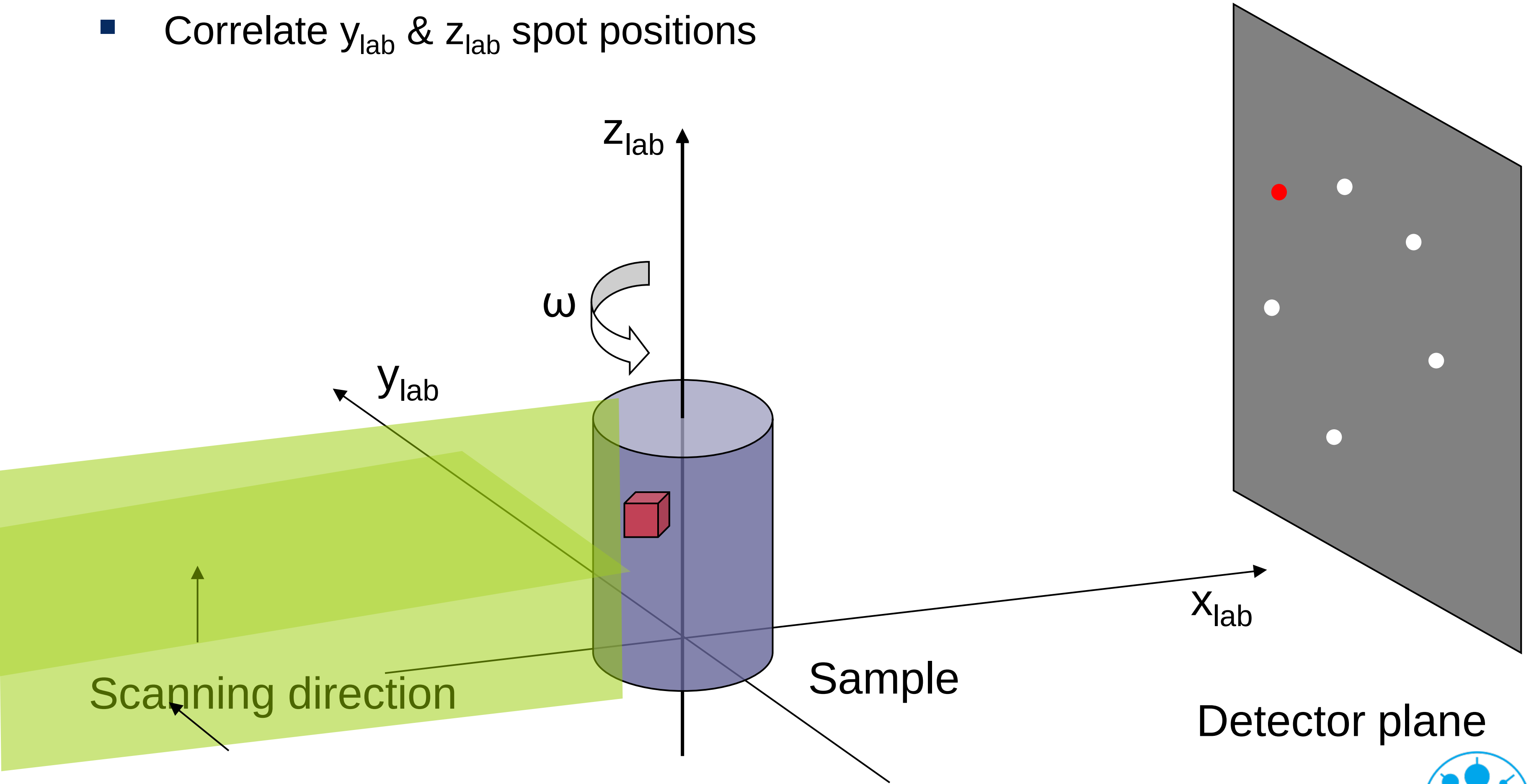
- Fatigue crack initiation from non-metallic inclusion
- APS, 1-ID



Naragani et al., Acta Mater. 137 (2017) 71-84

Box scan: experimental procedure

- A. Lyckegaard, E. Lauridsen (Risø DTU)
- Use horizontal & vertical line beams: $2N^2$ images
- Translating beam & repeat ω scans
- Correlate y_{lab} & z_{lab} spot positions



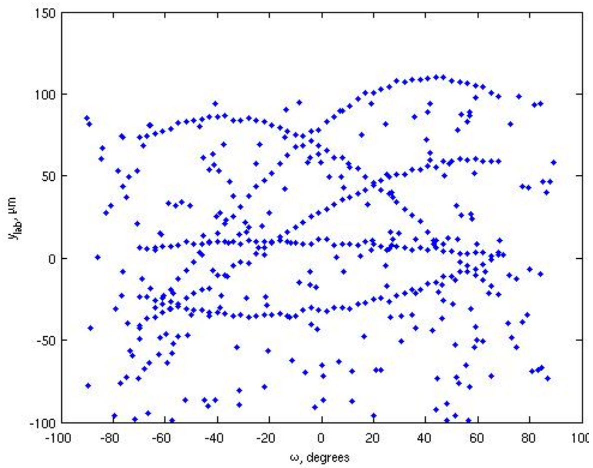
Scanning direction

Box scan principle

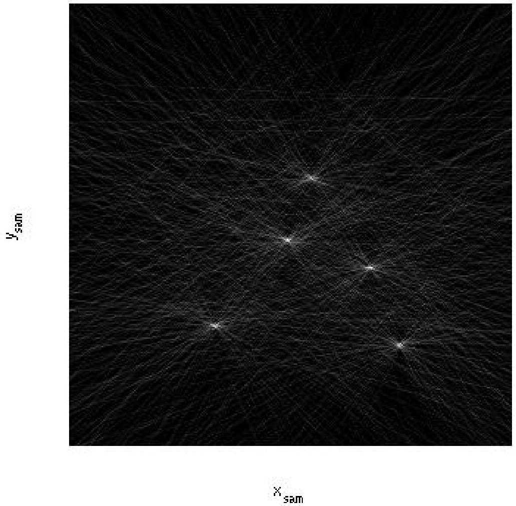
- Far-field: large working distance
- Decouples strain, position, and orientation
- Grain COM position, volume, aspect ratios, strain

Position space

Profile trajectories

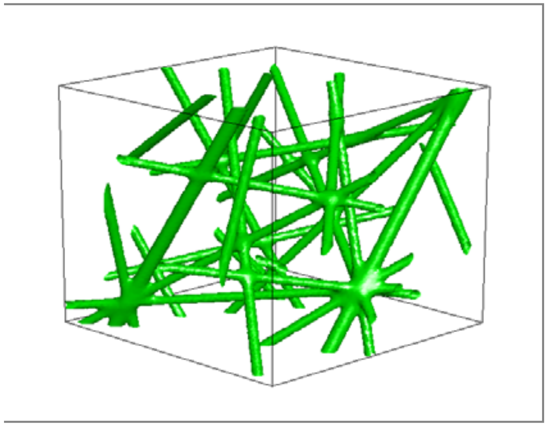


Inv. Hough transform



Orientation space

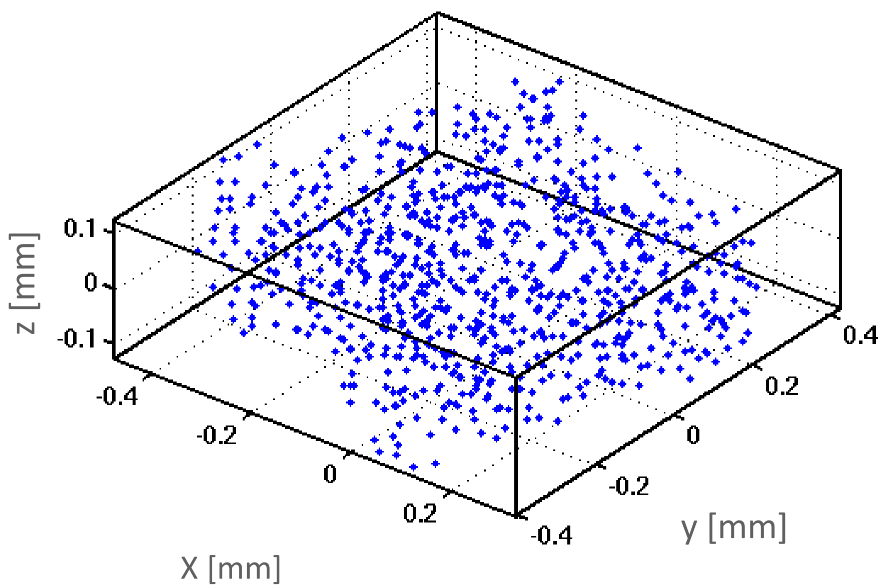
Rodrigues fibers

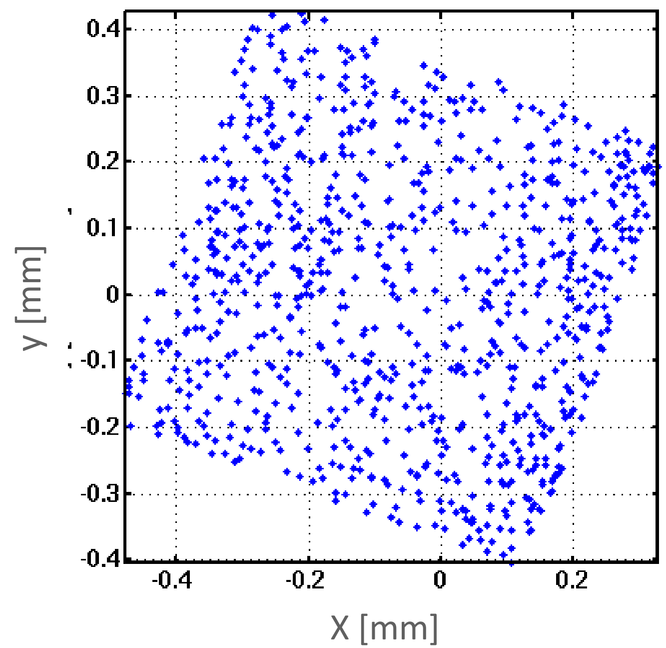


Box scan: β21s grain map

- COM grain center within < 2μm
- Grain statistics and neighborhood

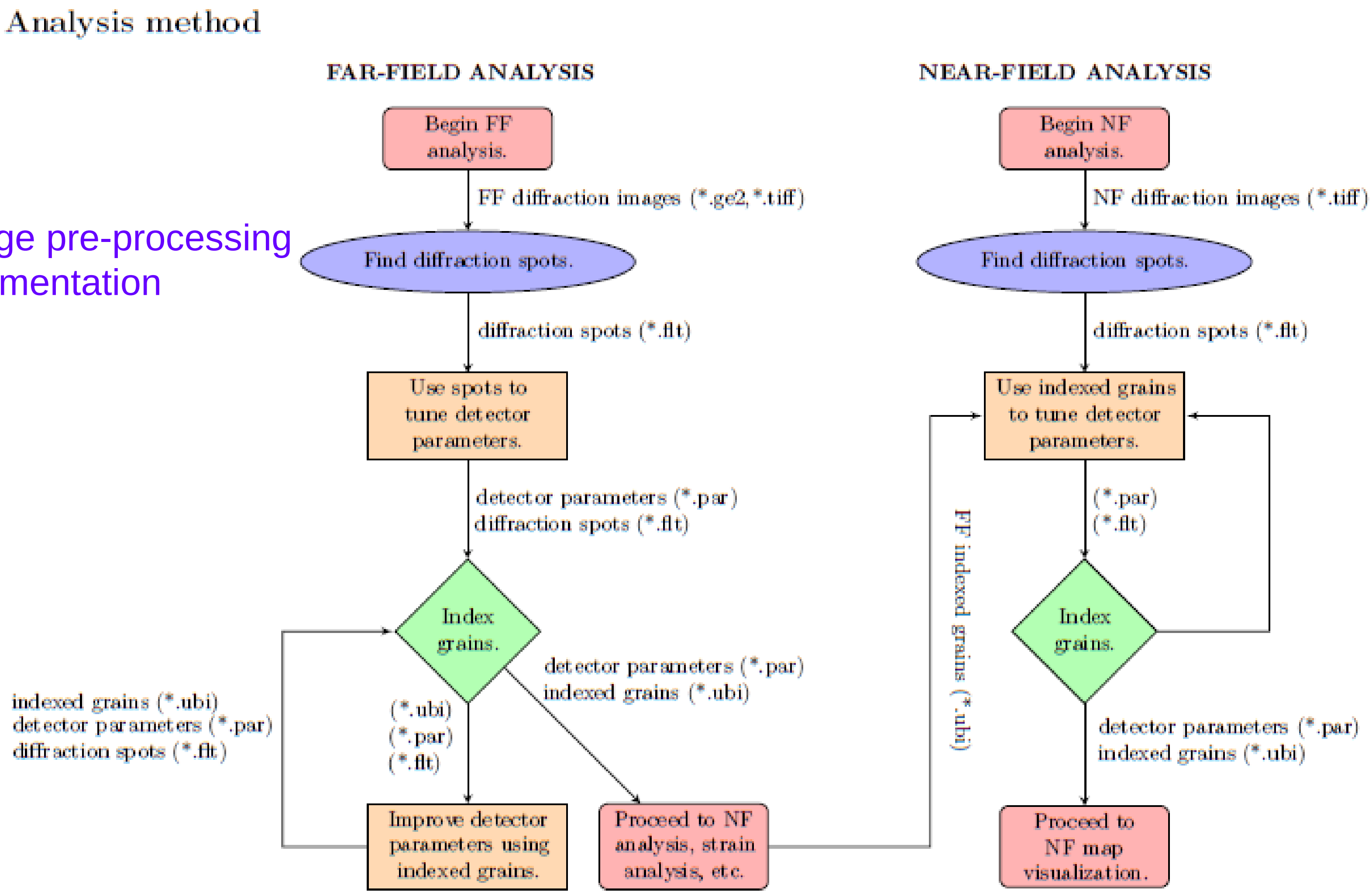
Reconstructed β-Ti volume, 650x650x250 μm^3. 845 grains.





Near-field mapping with box beam using FABLE and GrainSweeper.3D

- Image pre-processing
- Segmentation



Single grain diffraction software & algorithms

near-field undeformed | near-field deformed | far-field | Pre-processing | simulation

- > FABLE, <http://sourceforge.net/apps/trac/fable>
  - ImageD11; GrainSpotter; PolyXSim; QNFS; Fabian; FabIO; fabric
  - FitAllB (J. Oddershede *et al*., J. Appl. Cryst., 2010)
- > HexRD, <http://sourceforge.net/projects/hexrd/>
  - J.V. Bernier et al., J. Strain Analysis, 2011
- > DIG
- > IceN
- > Grai

3DMS satellite meeting on technical aspects of single grain diffraction techniques and data evaluation

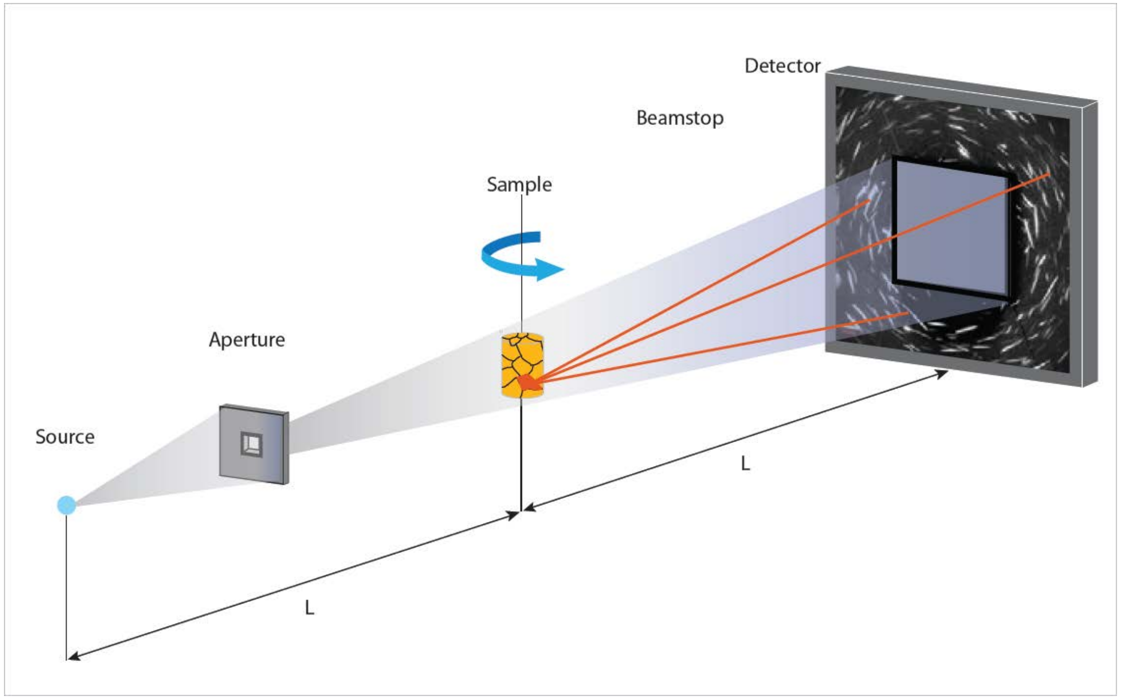
8-9 June 2018

DESY Hamburg

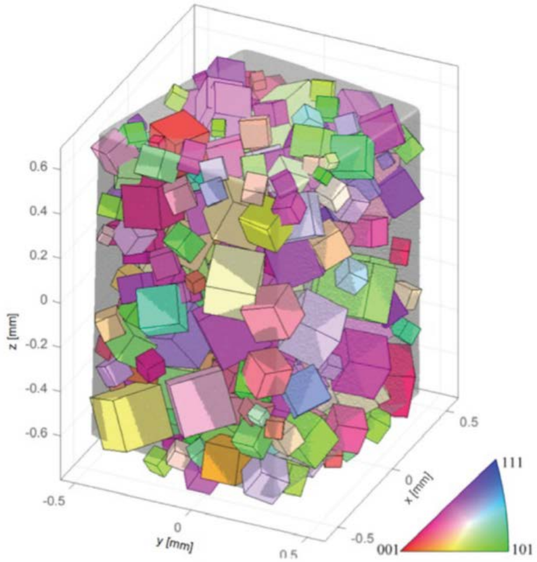
- > DCT, W. Ludwig, P. Reischig, et al., Rev. Sci. Instrum. 80, 2009
- > ART, H.F. Poulsen & X. Fu, J. Appl. Cryst. (2003)
- > H. Sharma et al., J. Appl. Cryst. 45 (2012) 693.
- > H. Sharma et al., J. Appl. Cryst. 45 (2012) 705.
- > M. Moscicki et al., Mater. Sci. Eng. A (2009)
- > BoxScan, A. Lyckegaard et al., Proc. 31st Risø Symp. (2010)

Laboratory Diffraction Contrast Tomography

- ZEISS Xradia 520 Versa
- Xnovo Technology GrainMapper3D™ software

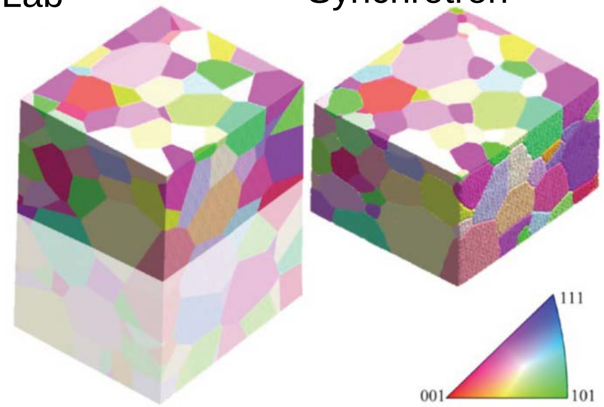


Timet 21S



Lab

Synchrotron



Conclusions

- Applied mathematics
  - Segmentation (3D, automated object identification)
  - Search over sparse 6D spaces
  - Inversion of discrete straight line projections
- Variety of experimental configurations (near, far, very far field, box-scan)
- Diffraction spots spread with orientation distribution
- Full use of diffracted intensities !!!
  - Precise detector synchronisation & pre-processing
  - Multi-grain crystallography
  - Sub-grain orientation & strain mapping?
- Online data analysis
  - “software alignment”
  - Provide to users: experimental parameters & reconstructions
  - Optimization of experimental parameters
  - Intelligent decisions during in-situ processing
  - “zoom-in” data acquisition
- Involve international software development community