High Spatial and Temporal Resolution Detectors for 3D studies using 30-100 keV X-rays.

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Risø National Laboratory, Denmark

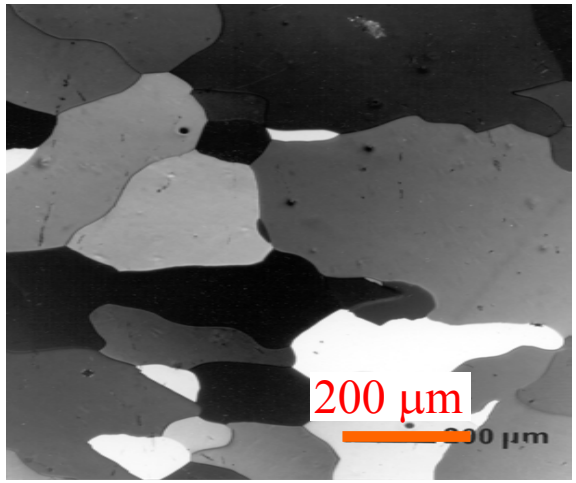
Metal Structures in 4D

ESRF

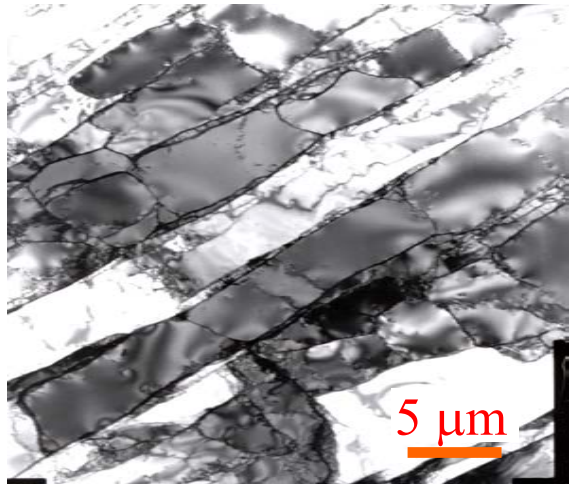
RISØ

Metal Structures

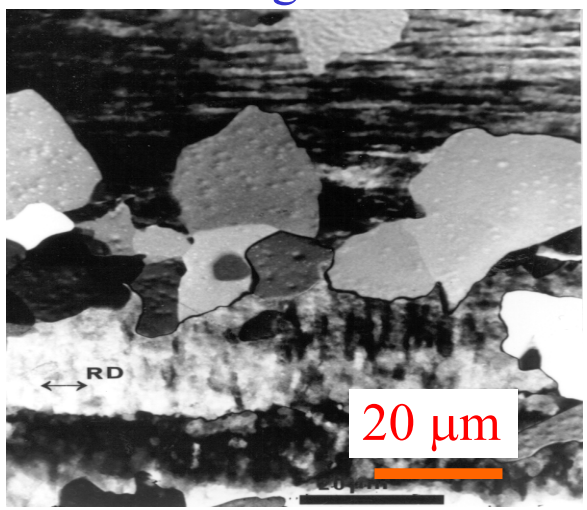
1. Undeformed :



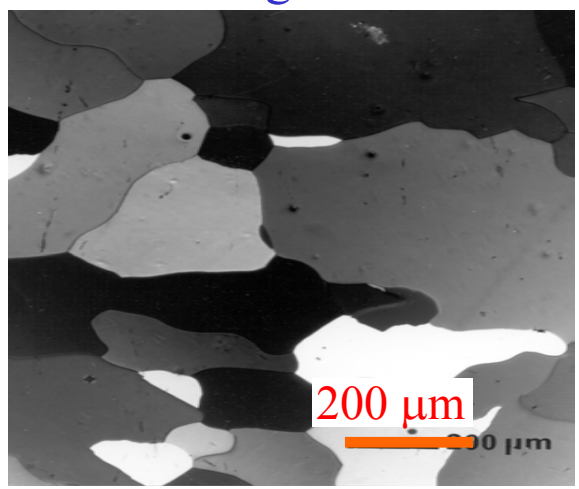
2. Deformation:



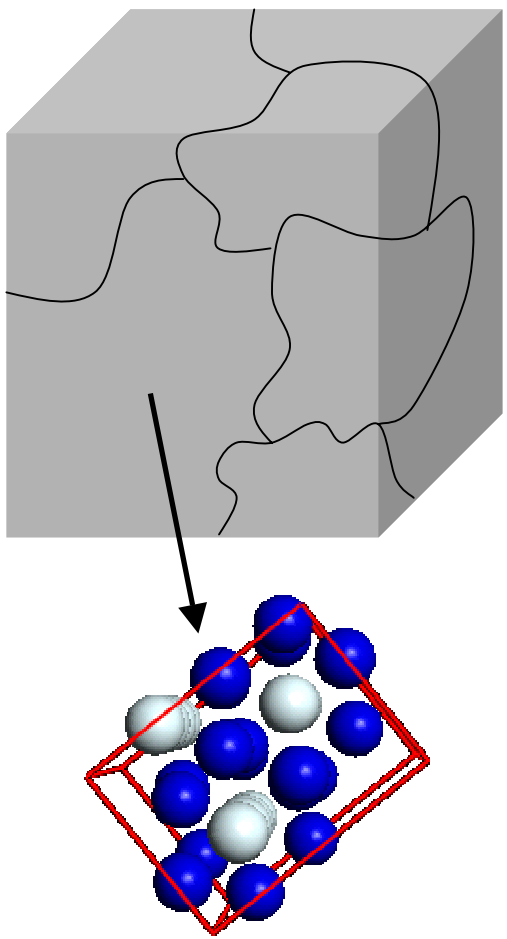
3. Annealing:



4. Annealing:



4D Vision



3D characterisation on a 100 nm – 1cm scale:

- position, morphology
- orientation
- plastic and elastic strain
- phase, crystallography
- chemical information

- Statistics over 100-1000 grains
- In-situ studies

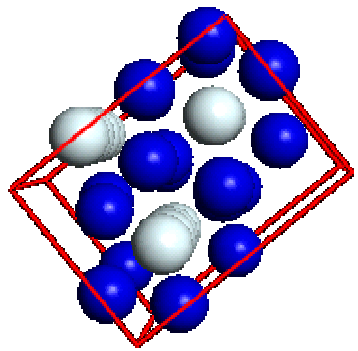
H.F. Poulsen: *Three-Dimensional X-ray Diffraction Microscopy* (Springer, 2004).

Parallel data acquisition

Position: 3D

Orientation: 3D

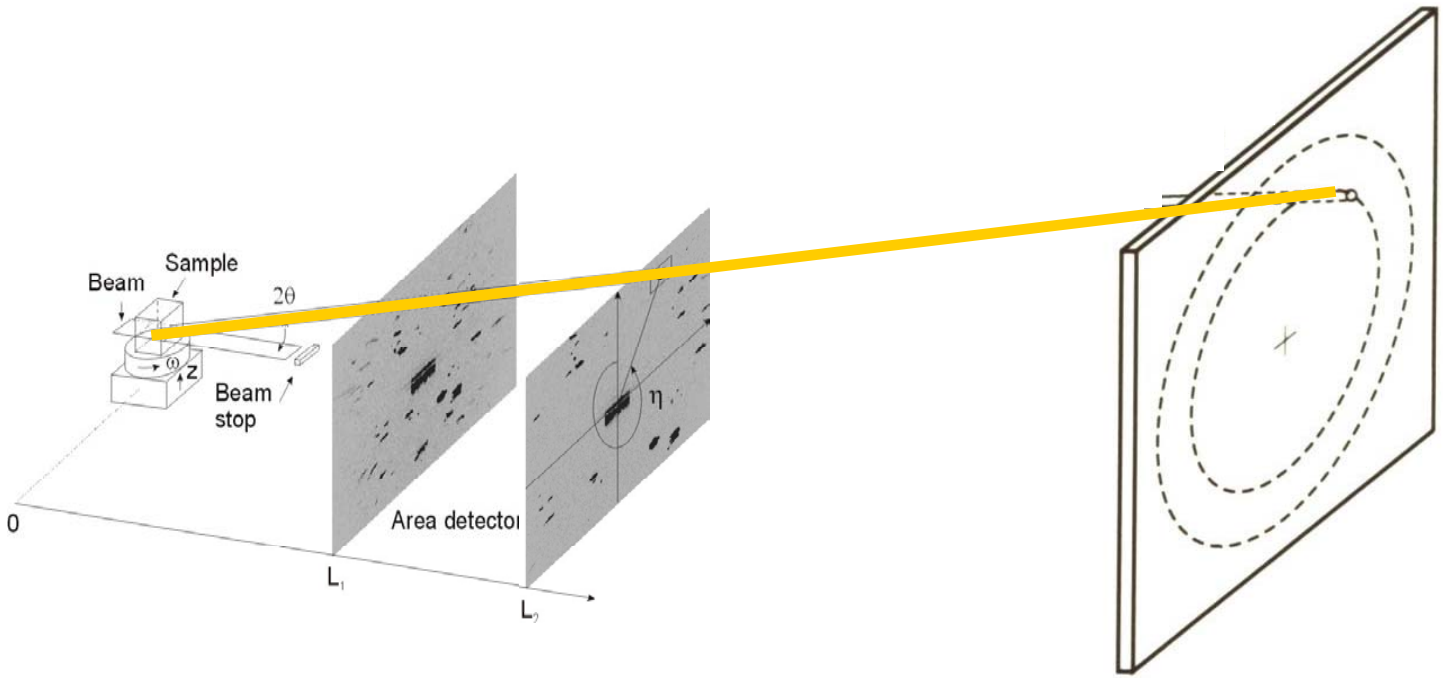
Lattice parameters: 6D



12D Space:

1000 x 1000 x 1000 positions:	10^9
0.5 deg orientation resolution	10^4
10^-4 strain resolution	10^6
	10^{19}

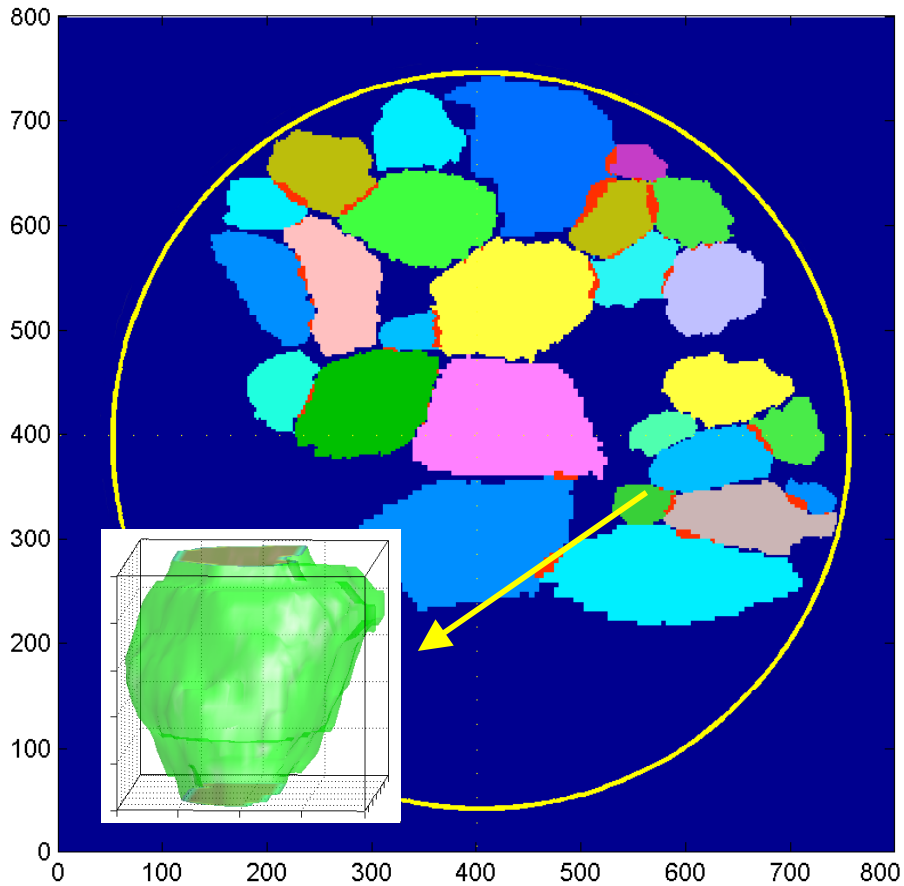
3DXRD Geometry @ ID11



Spatial +
Orientation

Strain +
Orientation +
Crystallography

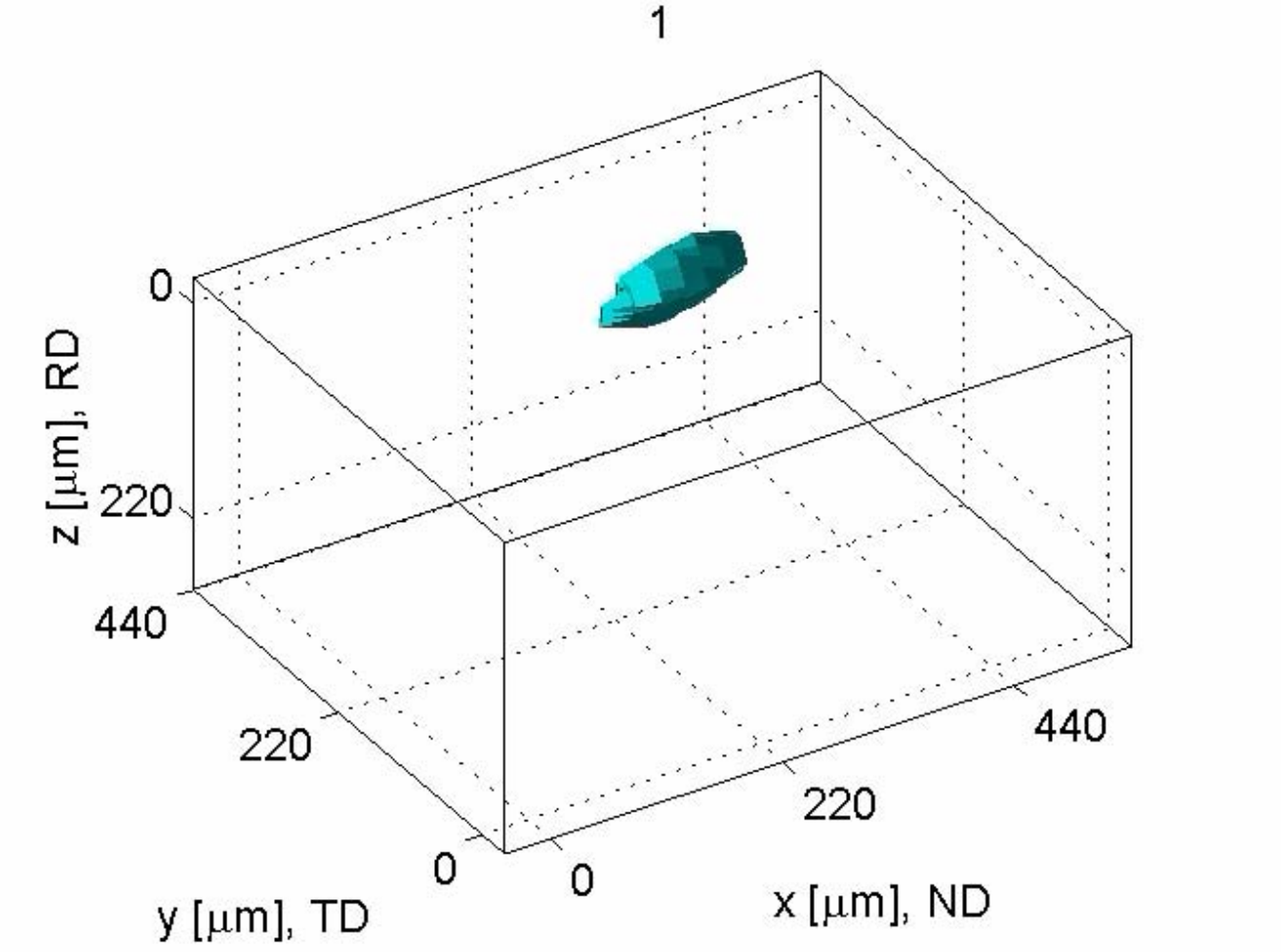
Reconstruction of grain map



5 min acquisition time
Resolution 5 μm

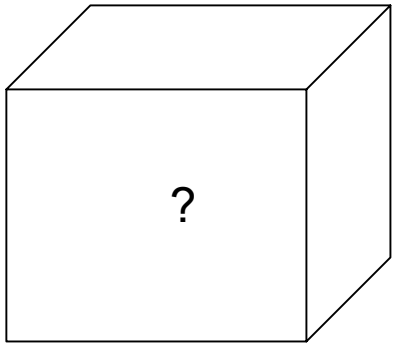
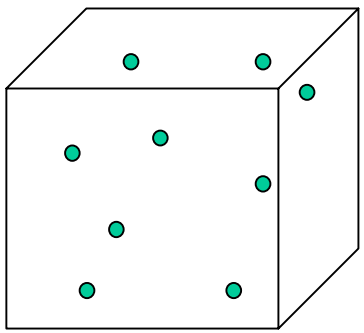
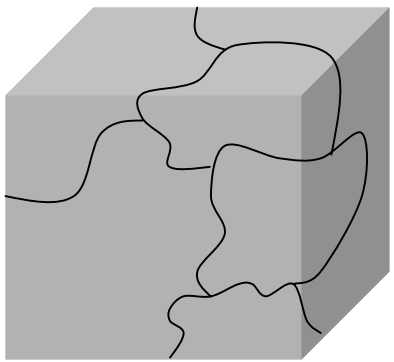
Growth of an internal grain

Recrystallization of 42% deformed pure Al during annealing at ~200 C.



S. Schmidt et al. *Science*, 2004, 229-232.

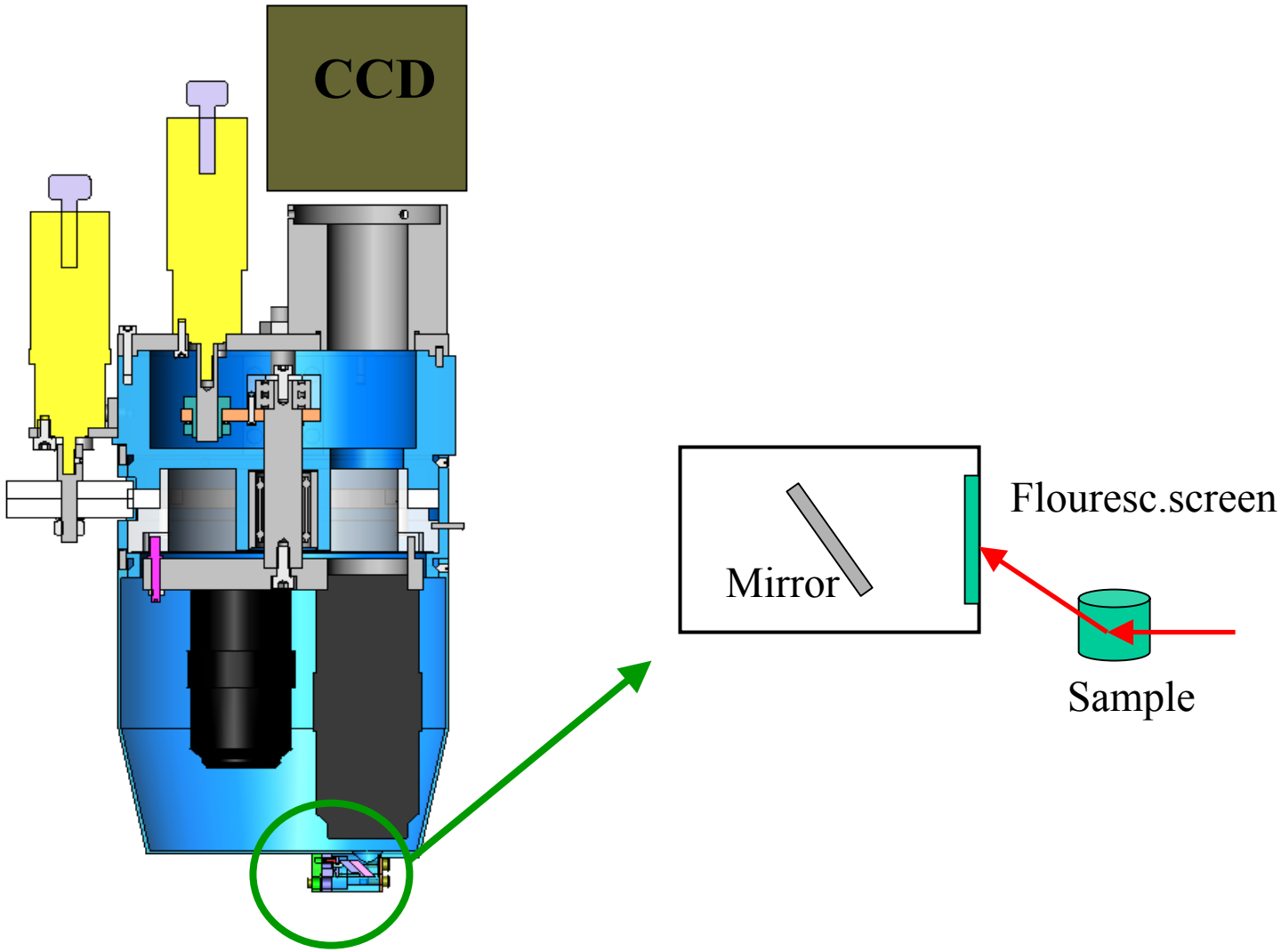
3DXRD resolution

	Spatial	Time for 100
	50 nm 2005: 20 nm	1 sec msec
	1 – 10 μm	30 sec
	5 μm limit: 2 μm	1-10 hours

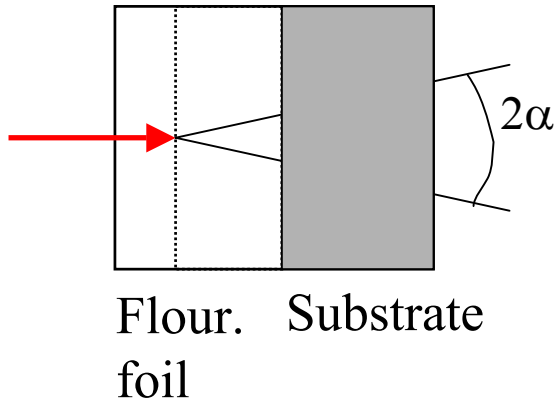
Set-ups at ID11, ESRF & SRI-CAT, APS

Lens coupled CCDs

ESRF: A. Koch, P. Spanne, C. Raven, T. Martin, M. Di Michiel, P. Cloetens ..
HASYLAB: F. Beckmann

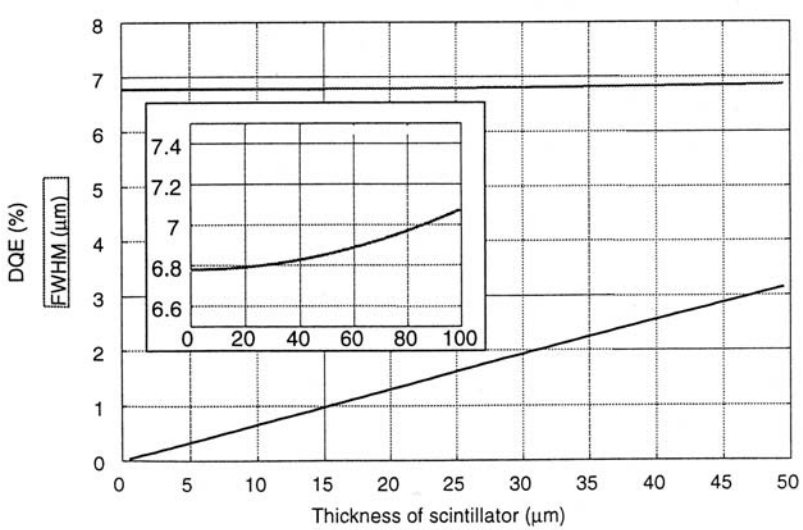


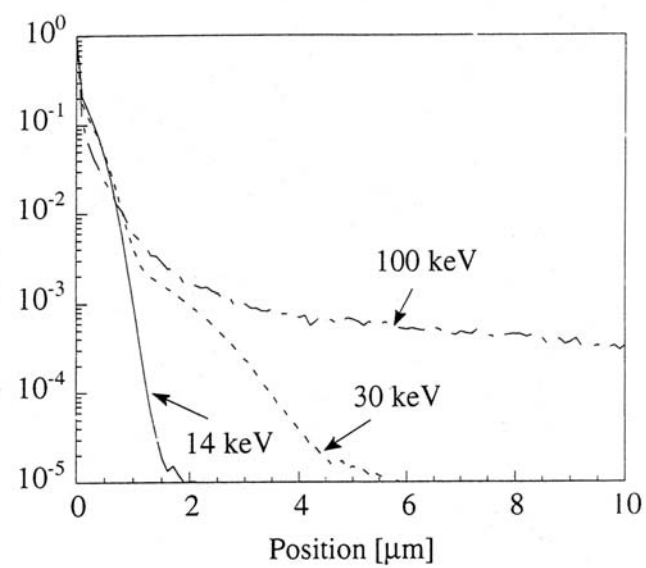
Limitations of lens coupled CCDs



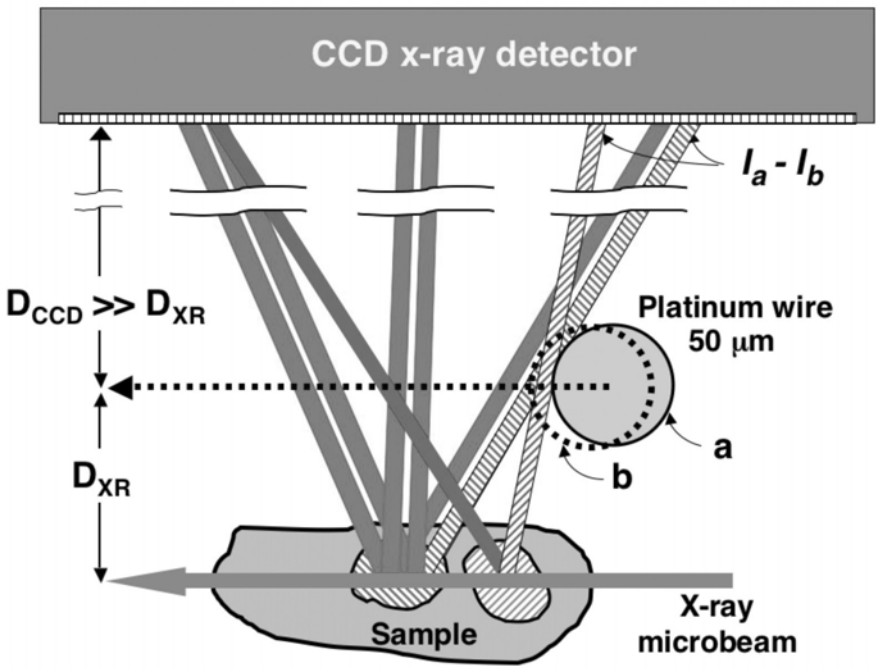
- Optical limit of 700 nm

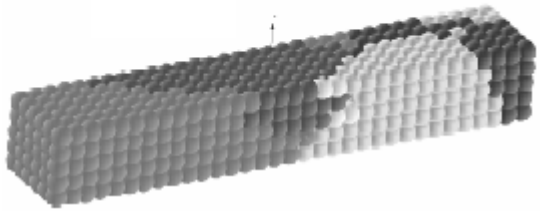
- Thin screens with support:
  - Low efficiency (~1%)
  - Point Spread Function with large tails





Differential Aperture X-ray Microscopy





Spatial resolution: 0.5 μm

B.C. Larson *et al. Nature* **415**, 890 (2002)

Materials science @ synchrotrons
5 years from now

4D movies with time resolution of < 1 minute

1 mm^3 specimens with a resolution of 1 μm^3

100 μm^3 specimens with a resolution of 100 nm^3

Combination:

tomography

diffraction (3DXRD)

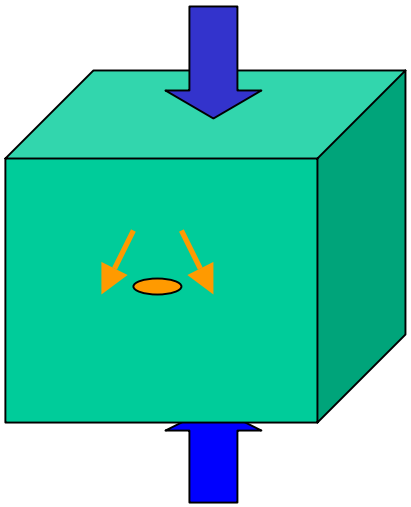
crystallography

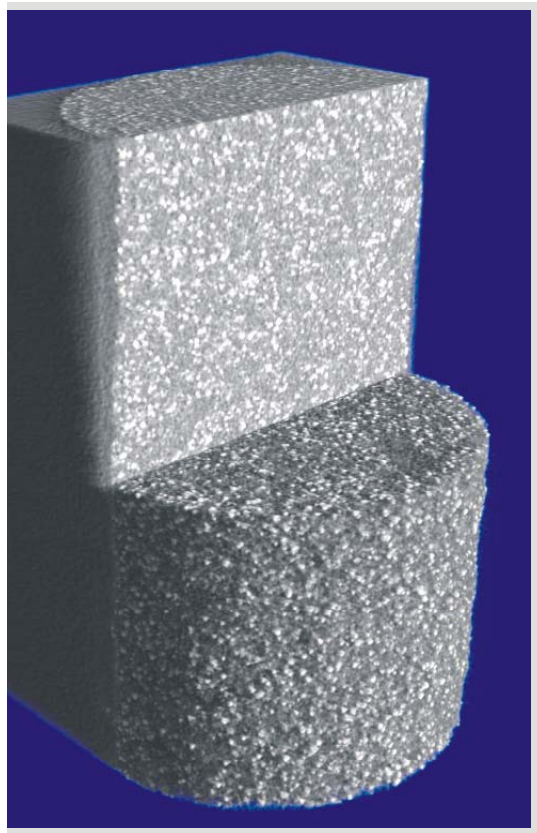
1. 3D detector banks

2. New generation of high resolution detectors

Plastic flow

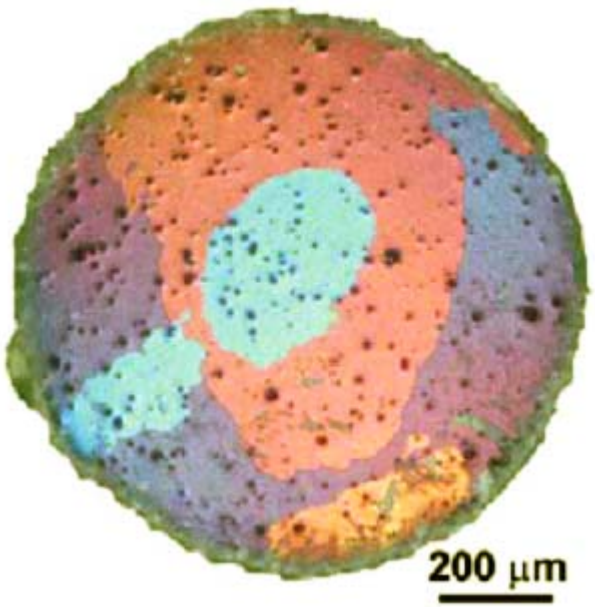
Plastic strain field





Work with F. Beckmann, HASYLAB

Plastic flow in real material



Tomography: Local plastic flow

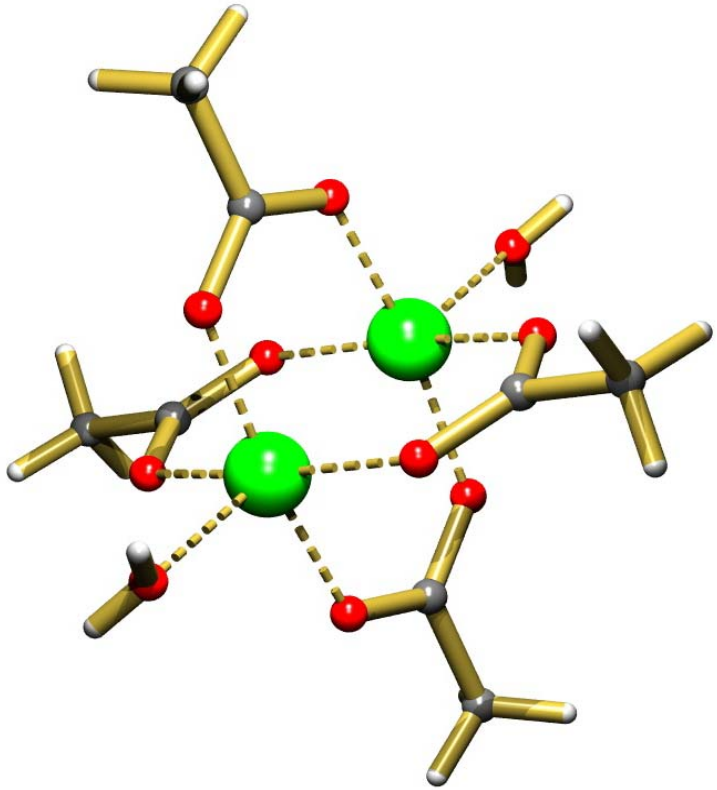
3DXRD: Grains, Rotations of grains

Maps of *the* local slip activity in a 3D, bulk sample

Third route in crystallography: Structural solution of polycrystals

Validation: $Cu(C_2O_2H_3)_2 \cdot H_2O$.

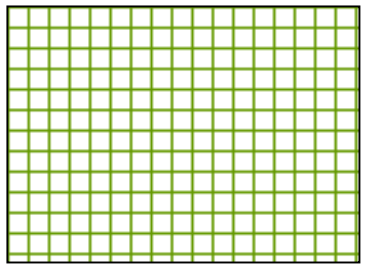
70 grains of size < 1 micron
Cell ~1400 Å^3 (C2/c)



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

Brainstorm, 1st question:

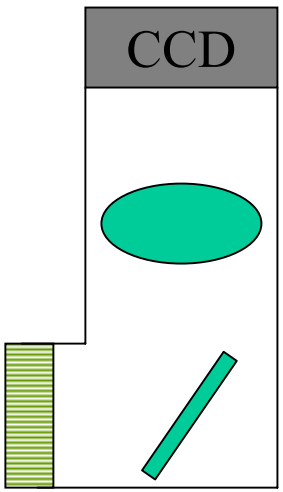
Pixelation to 100 nm



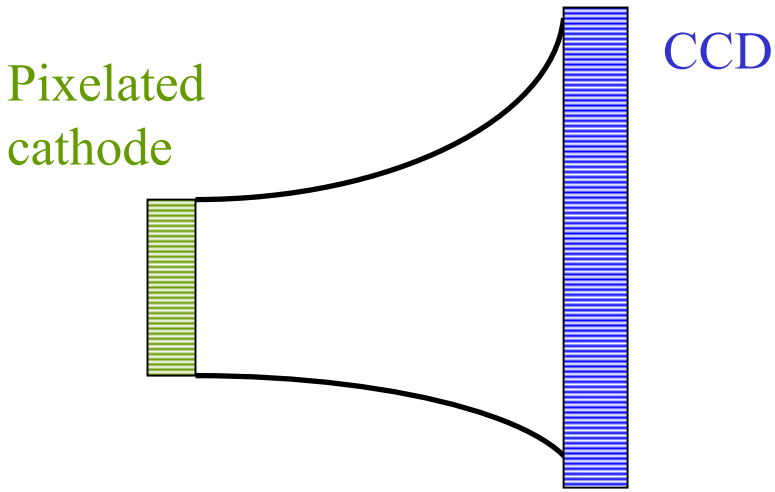
3D screen prodcued by FIB?

Parallax no problem

UV coupling

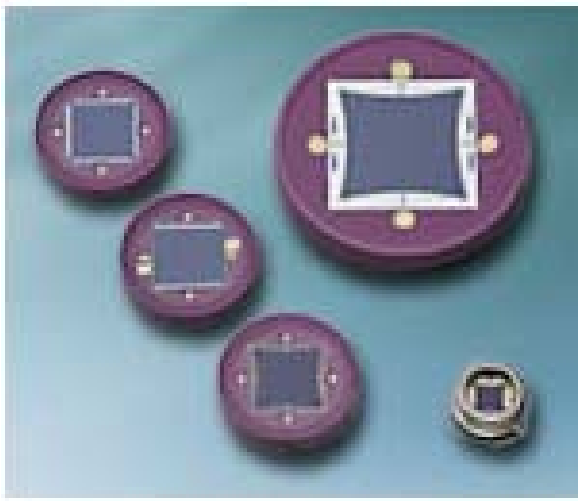


Electron optics

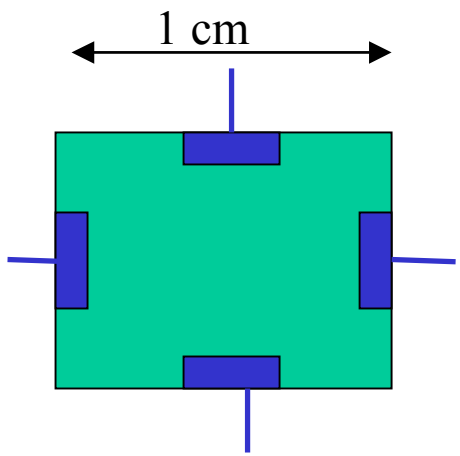


Brainstorm, 2nd idea:

Array of Position sensitive diodes

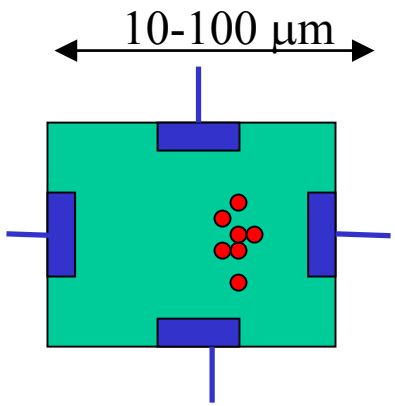


e.g. Hamamatsu



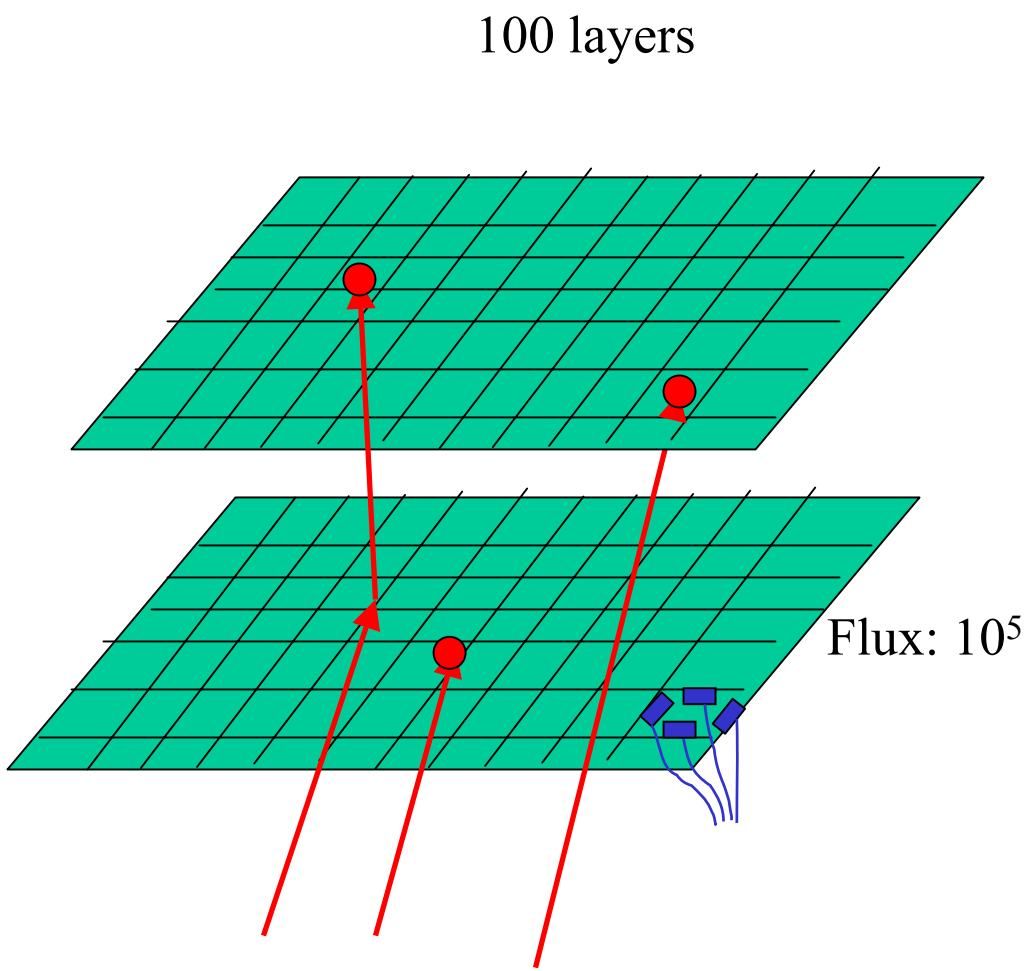
Precision: 1 in 1000

Downscale concept:



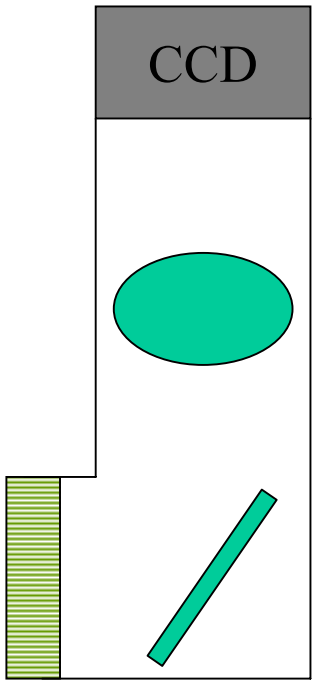
X-ray energy: 30-100 keV

Array of Position sensitive diodes



Brainstorm, 3rd question:

Energy dispersive 3DXRD



Still 2 μm resolution

but much faster

Case: new generation of high resolution detectors

A factor of 100 to be gained in flux

A factor of 20 in spatial resolution

Applications:
3DXRD on metals, ceramics, microelectronics, rocks, ice, bones, sugar, …
Imaging in general, in particular at 2nd generation sources

More speculative:
Total Crystallography on drugs, …
Free electron lasers

Collaborators on instrumentation/software

Risø: X. Fu, E. Knudsen, E.M. Lauridsen, L. Margulies, R. Martins, S.F. Nielsen, S. Schmidt, H.O. Sørensen

ESRF: A. Goetz, Å. Kvick, G. Vaughan, T. Buslaps, V. Honkimäki, T. Martin

APS: U. Lienert, J. Almer

IMSA-Lyon: W. Ludwig

State University of N.Y: G. Herman