Coherent Diffraction Imaging as a tool to investigate the mechanics of Polycrystals

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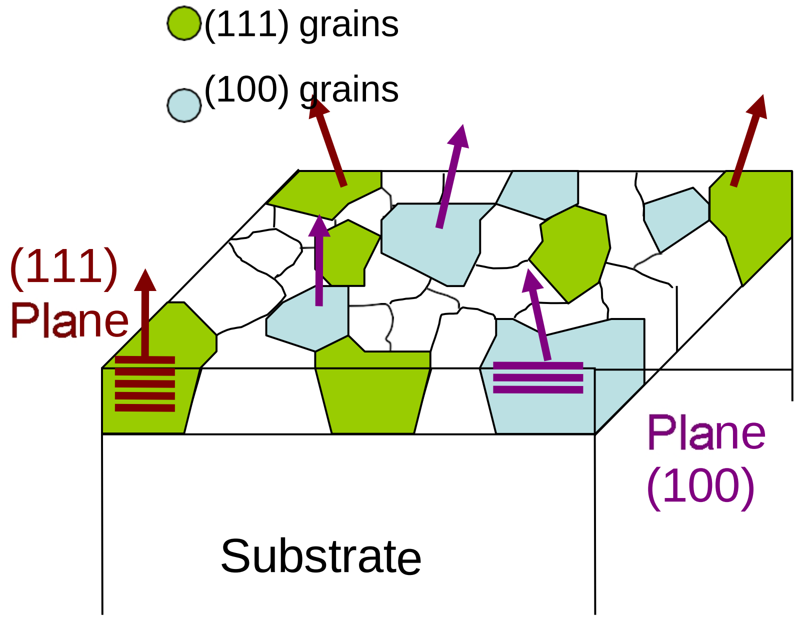
Context and Purpose

- The mechanical properties of small objects play a key role in numerous phenomena
- The local behaviour of Polycrystals is being still debated
- Strong lack of experimental techniques to probe deformation on this nanometer scale
- Coherent diffraction : a promising tool



Single grain behaviour

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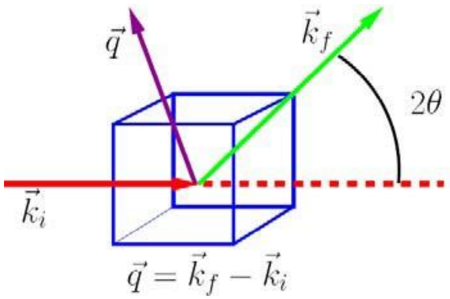
Phase Retrieval: specific case of strain objects

Kinematic theory of diffraction



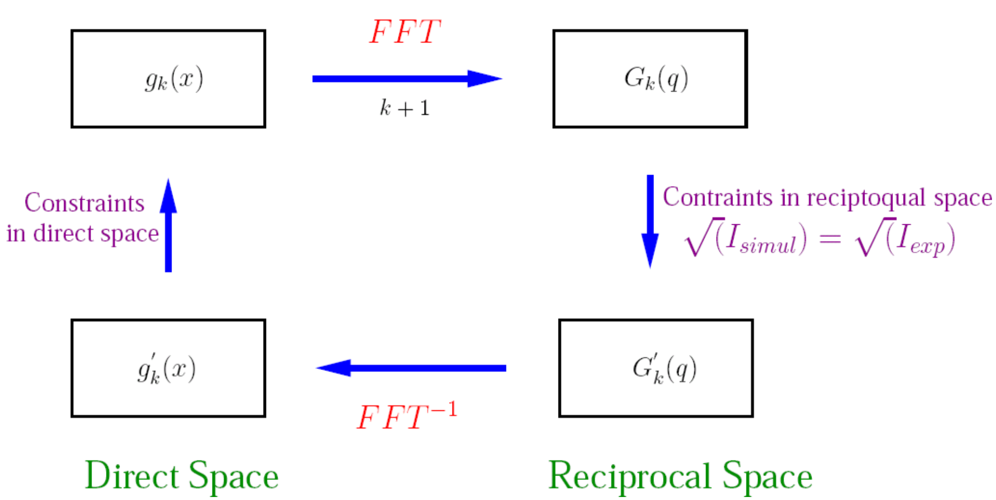
Displacement field

$$\mathbf{r}_n = \mathbf{R} + \mathbf{u}$$

$$I(\mathbf{q}) = \left| \sum_n f_n(\mathbf{q}) \exp \imath \mathbf{q} \mathbf{r}_n \right| \longrightarrow I(\mathbf{q}) \propto \left| TF\left\{ \rho(\mathbf{r}) . \exp\left(\imath \mathbf{G}.\mathbf{u}(\mathbf{r})\right)\right\}\right|^2$$

Diffracted Intensity

Iterative Phase Retrieval Algorithm



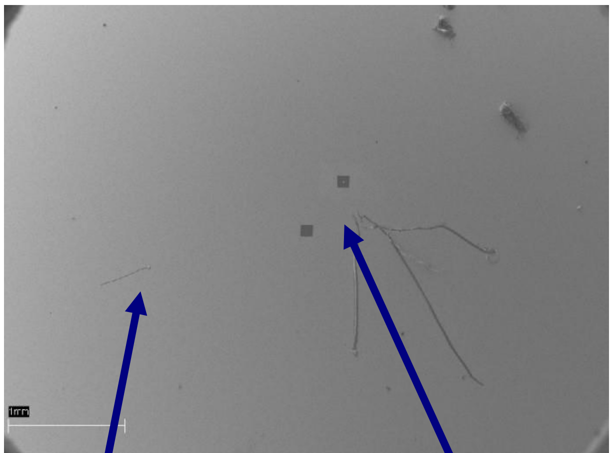
Difficulties

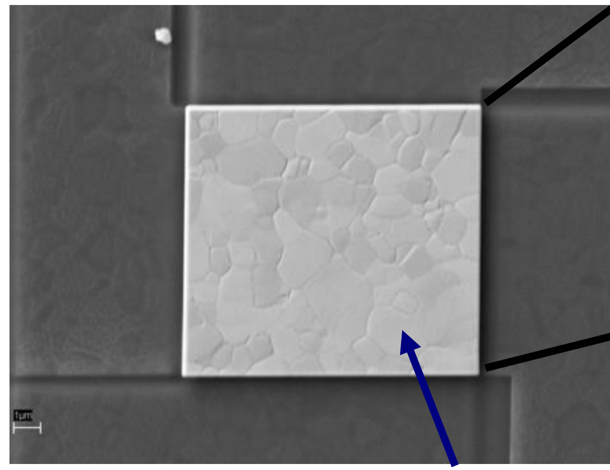
- Tight support is needed for good reconstruction
- Initial support cannot be determined from autocorrelation (Patterson)
- Convergence more difficult (add condition of density)

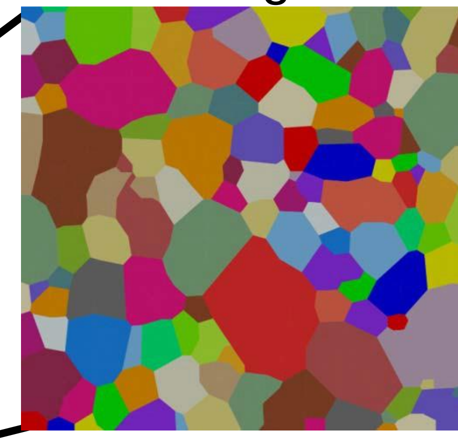
Our model sample

Main features
- ➢ Gold sample
- ➢ Special design

Why
- ➢ Good diffraction power, no corrosion issue during heating
- ➢ Find which single grain is diffracting







133 grains

Homogeneous film (375nm thick)

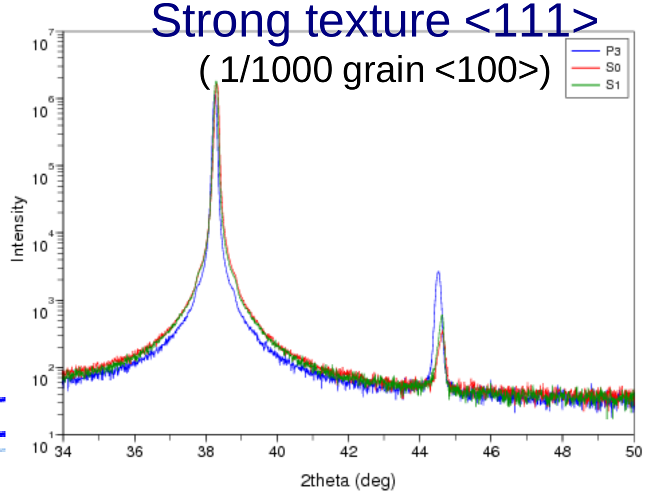
150µm square area drilled by FIB

10*10µm polyscrystal

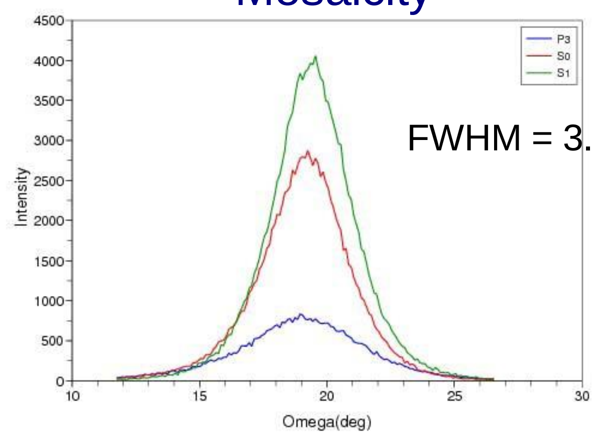
Single grain behaviour

Strong texture <111>

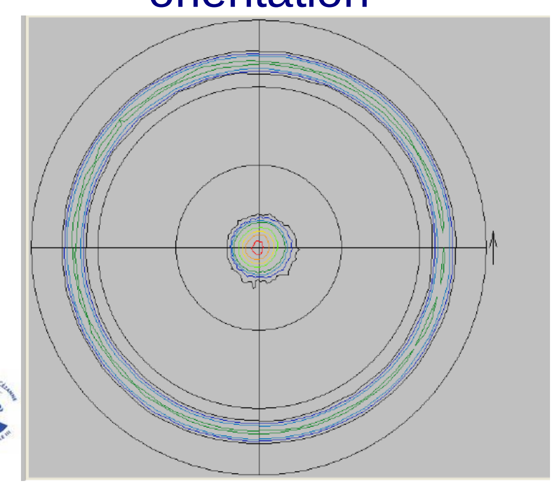
(1/1000 grain <100>)



Mosaicity

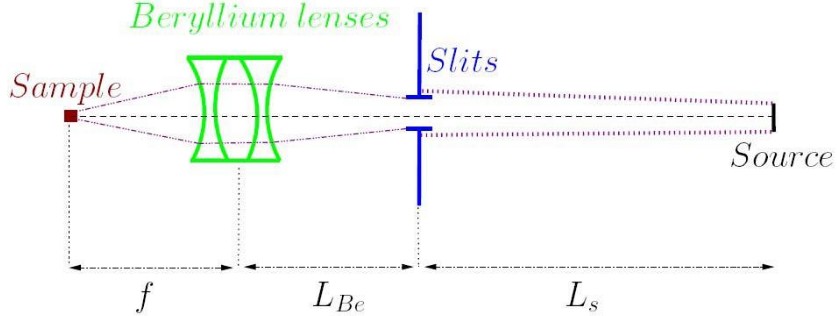


In-plane random orientation



Set up used on ID01 Beamline

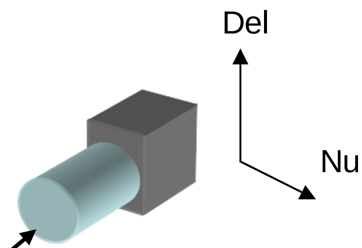
Beam caracteristics



Focal length ~ 40cm
Beam size ~ 1x3µm
Divergence ~ 50µrad

(111) Bragg peak

$2\theta_{111} = 37{,}8°$

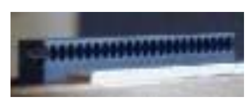


CCD camera

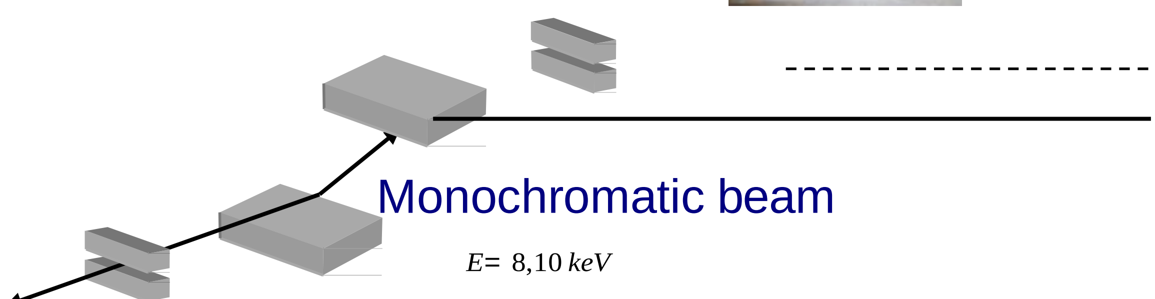
- Indirect illuminated deep depletion detector
- Pixel size : 22µm

Projected Beam footprint on sample:
3x3 µm

Focused beam



Furnace

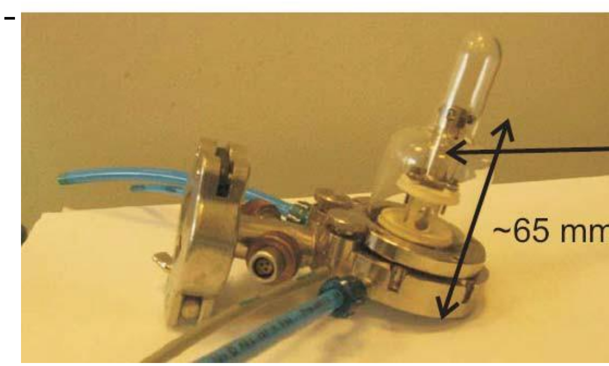


Monochromatic beam

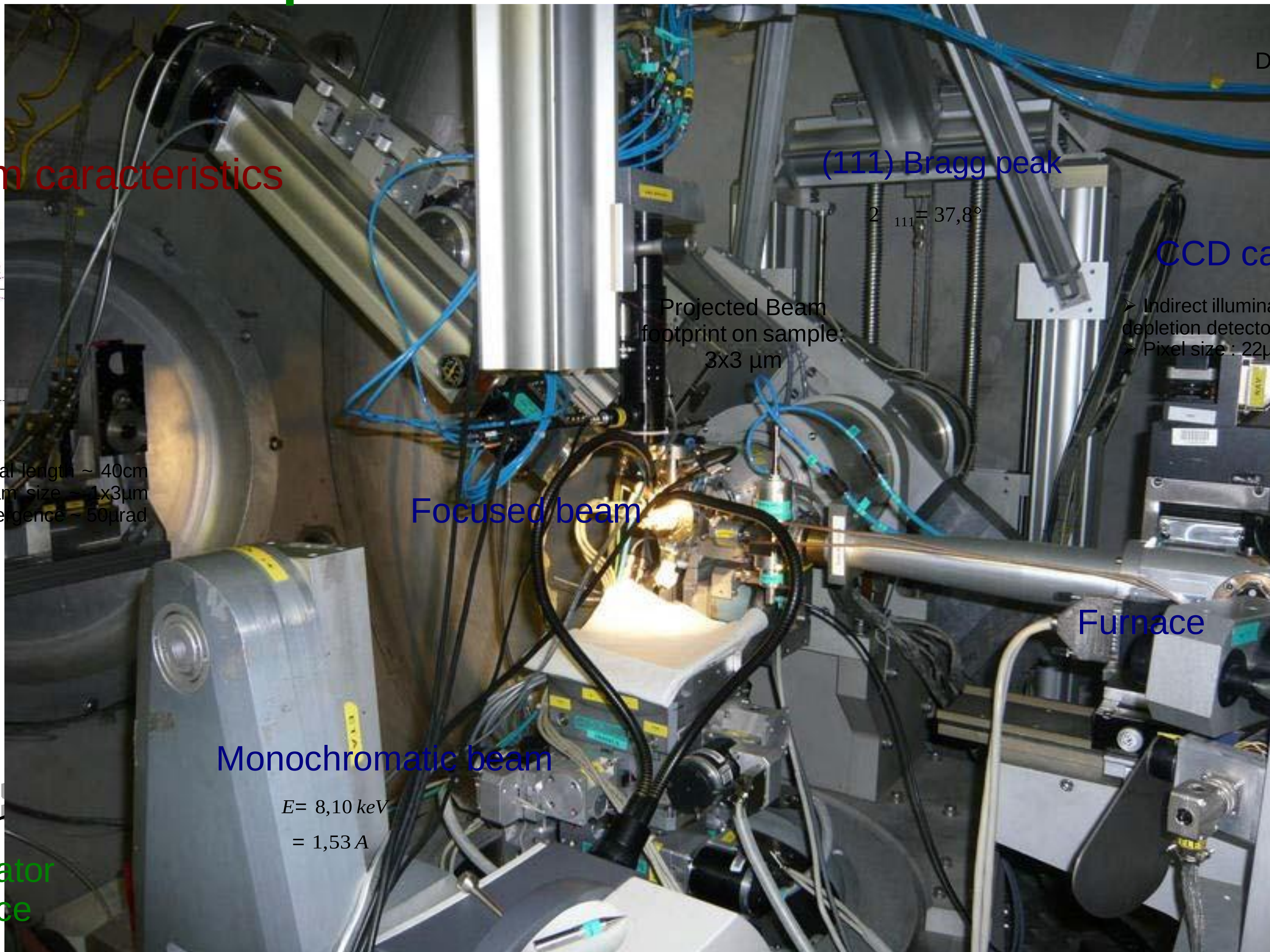
$E = 8{,}10\ keV$

$\lambda = 1{,}53\ Å$

Undulator source



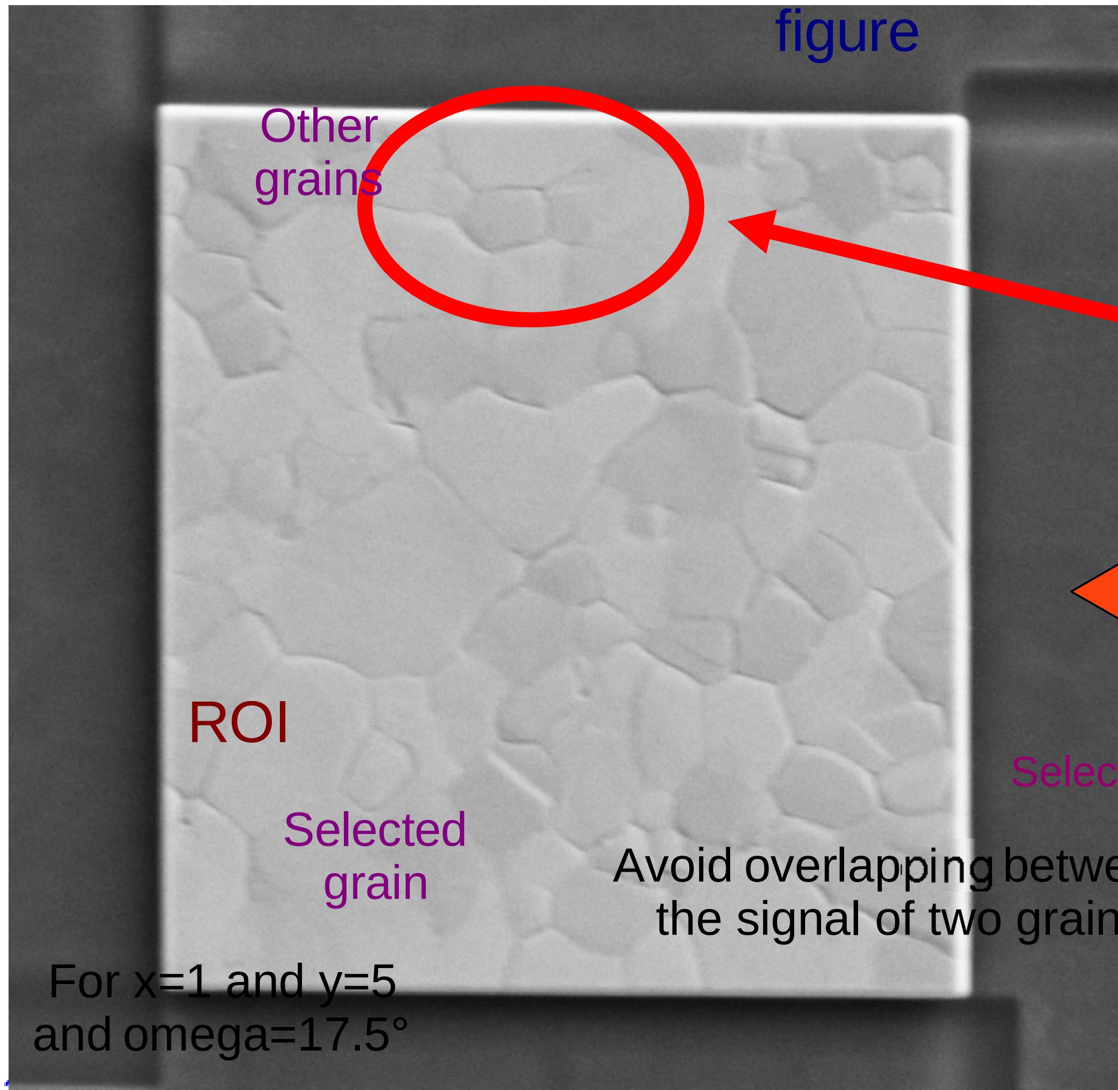
Set up used on ID01 Beamline



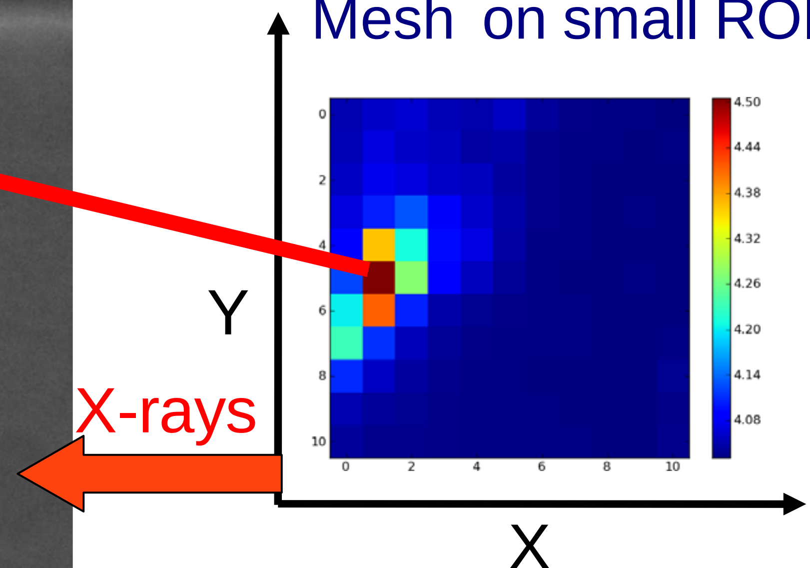
Grain determination

Scanning 3DXRD approach {x,y}* {Nu,Del,Omega}

Granular Debye ring Angular Map – Pole figure



Direct space
Mesh on small ROI



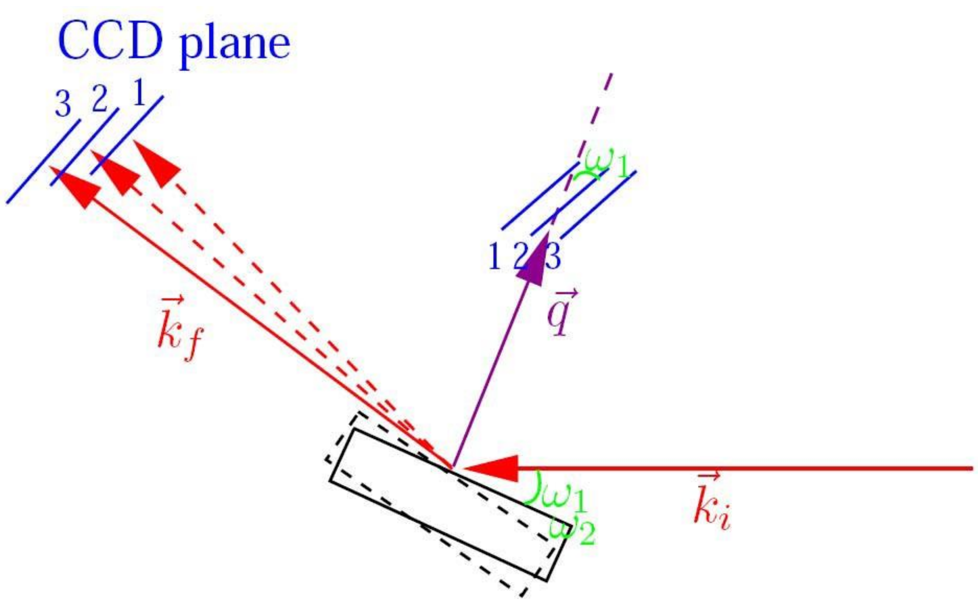
Selected grain

Projected Beam size : 3*3 µm

3D Bragg Peak acquisition

Rocking curve peak (111)

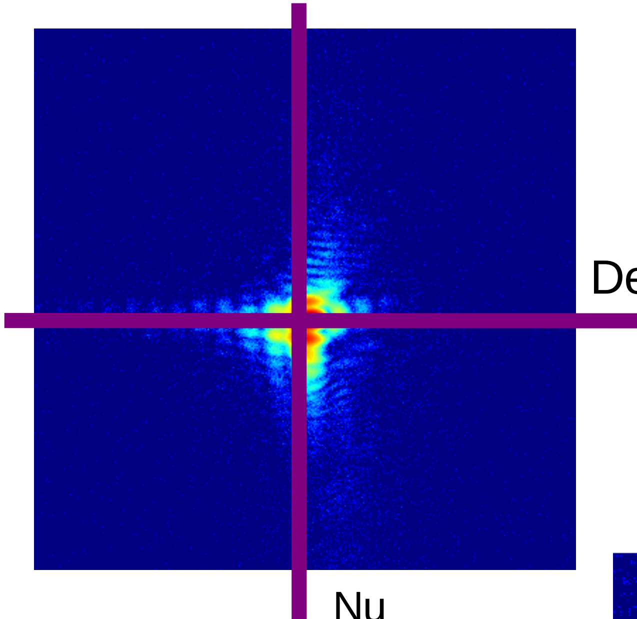
- Diffraction geometry
- Oblique cut in reciprocal space
- Weak resolution in Omega direction

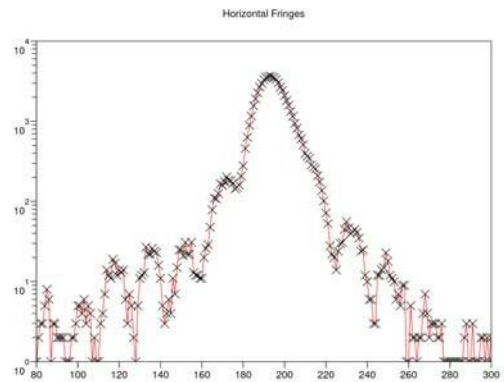


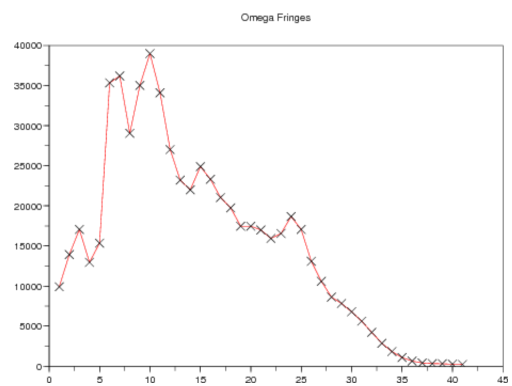
Acquisition characteristcs

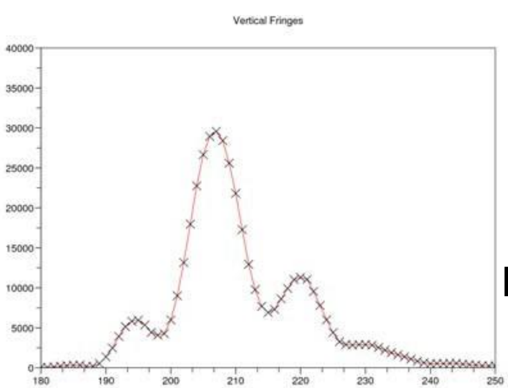
- Detector Indirect Deep depletion CCD 22µm pixels
- Single photon counting mode
- 3D acquisition time = 8 hours
- Max photons = 11 957 on one pixel
- Sum photon (all Bragg Peaks) = 32 .10^6 photons

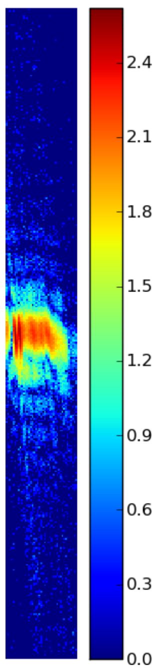
Oversampling and Characteristic dimensions











Reciprocal Space Resolution

- Δq (ccd) = 1,7 e-4 nm-1
- Δq (omega) = 1,118e-3 nm-1

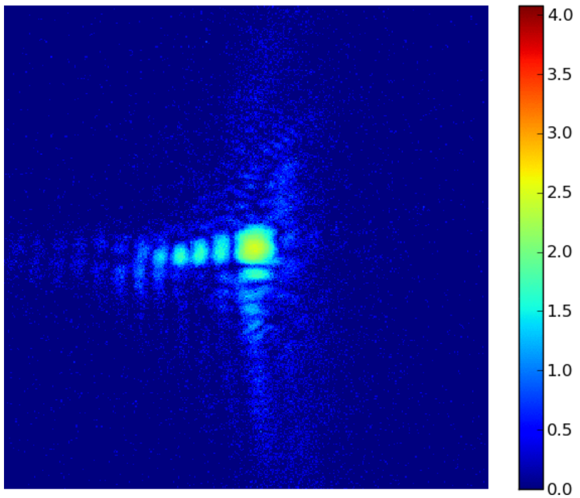
3D Oversampling

- Sx = 15
- Sy = 8
- Sz= 4

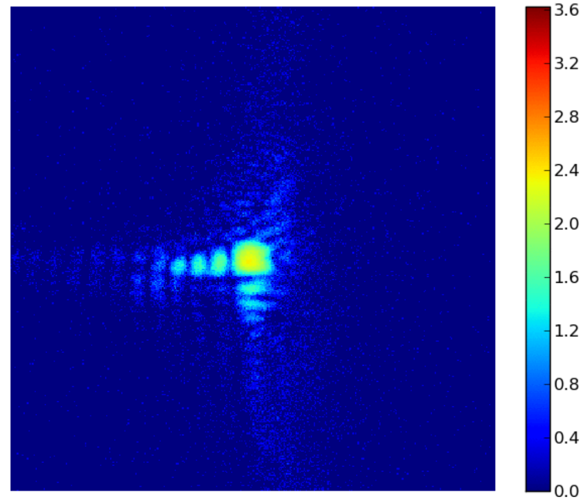
Dimension and Resolution in direct space

- X = 738 nm [Δ(X)=15.4nm]
- Y = 230 nm [Δ(Y)=20nm] (Beam direction)
- Z = 394 nm [Δ(Z)=15.4 nm] (Film thickness)

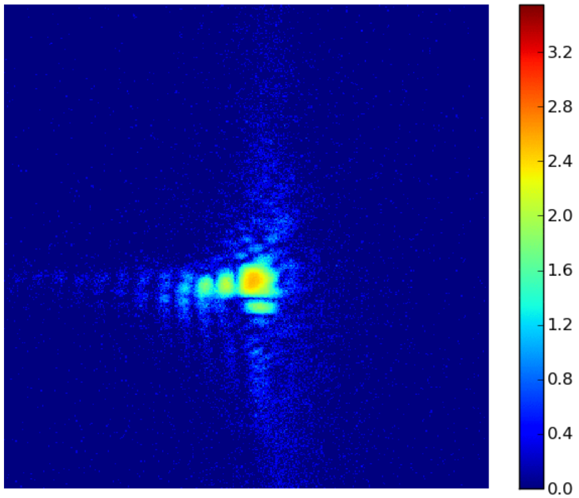
T=50deg



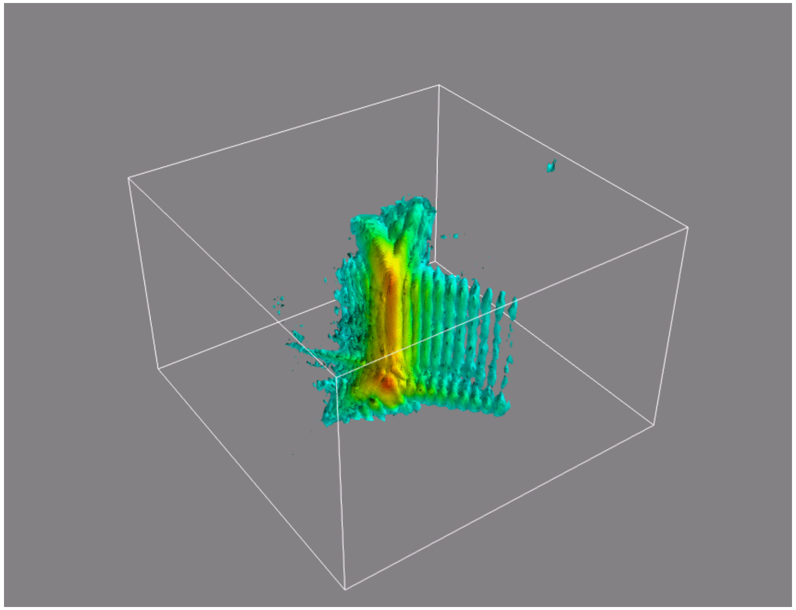
T=100deg



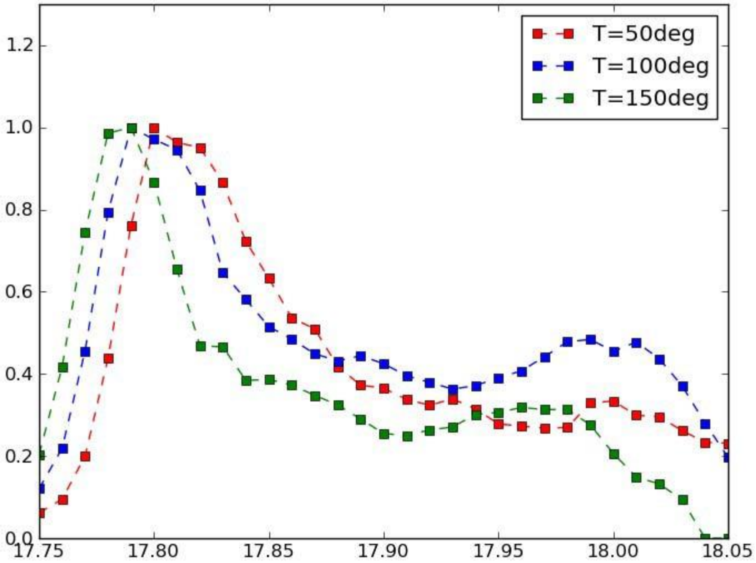
T=150deg



T=100deg

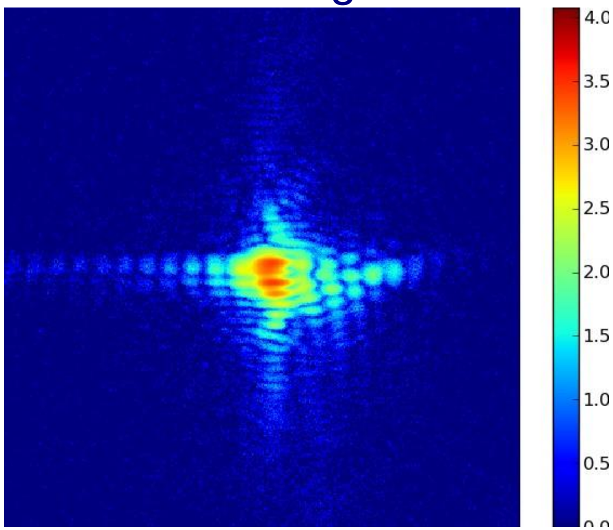


Rocking curve comparison

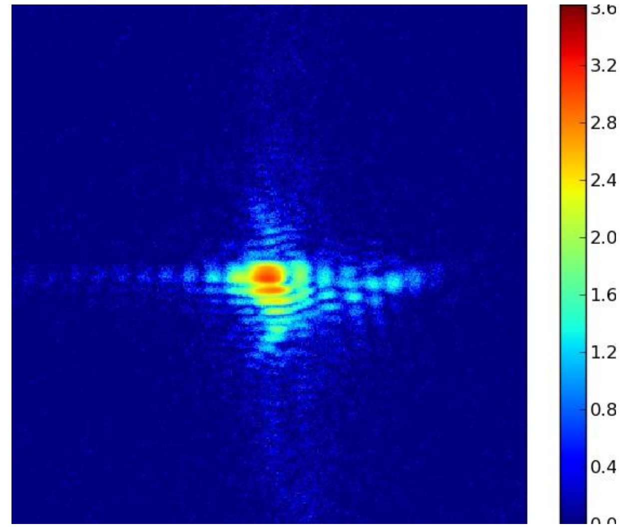


Experimental results

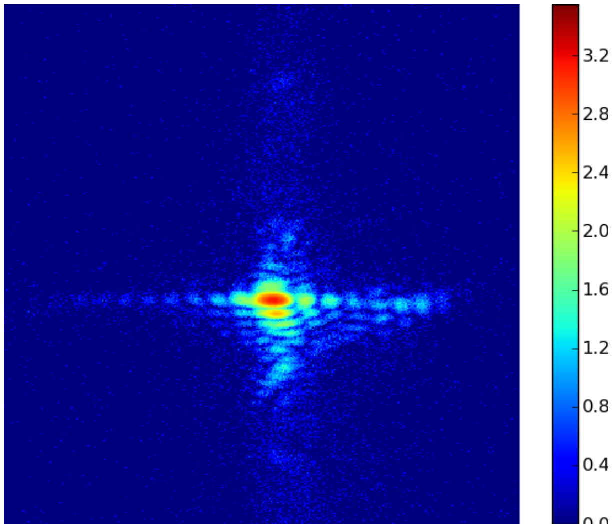
T=50deg



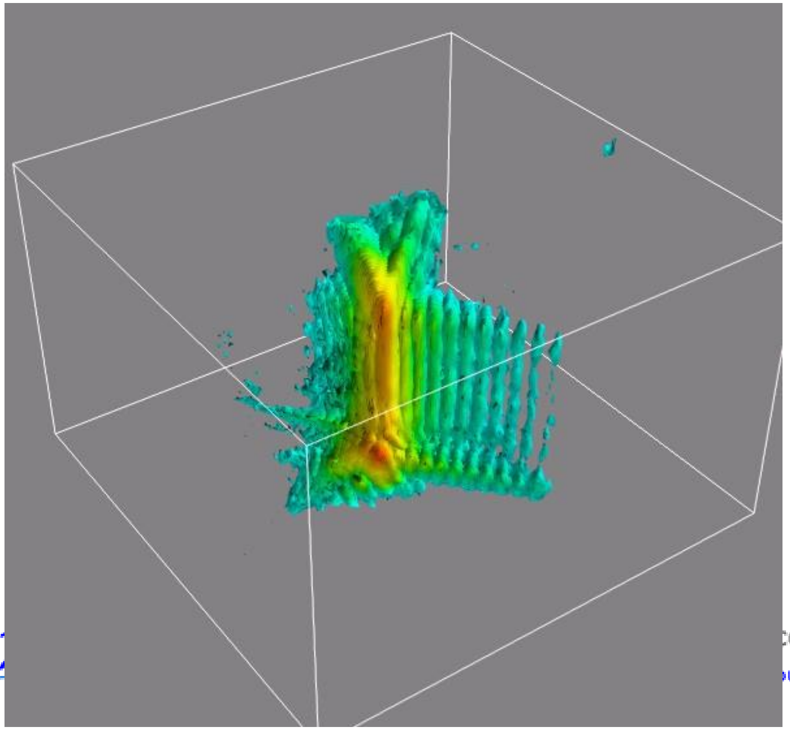
T=100deg



T=150deg



3D representation at T=100Deg



Oversampling in Omega direction