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

MID workshop, 2009 Grenoble

N. Vaxelaire , S. Labat , O. Thomas

IM2NP, Marseille

A. Diaz, T. H. Metzger

V. Jacques,
F. Picca, S. Ravy

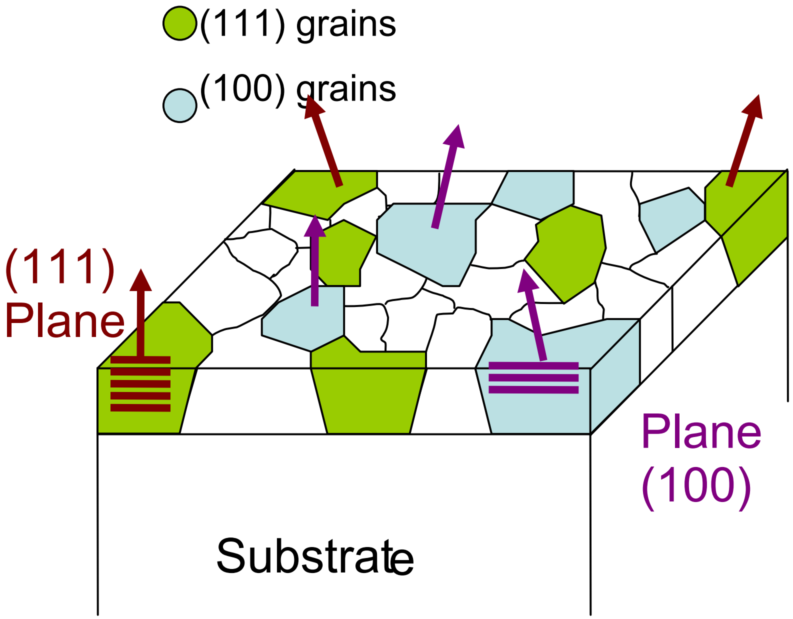
H. Proudhon, S. Forest

J. Keckes,
C. Kirchlechner

Institut Matériaux Microélectronique Nanosciences de Provence
UMR 6242 CNRS, Universités Paul Cézanne, Provence et Sud Toulon-Var

Context and Purpose

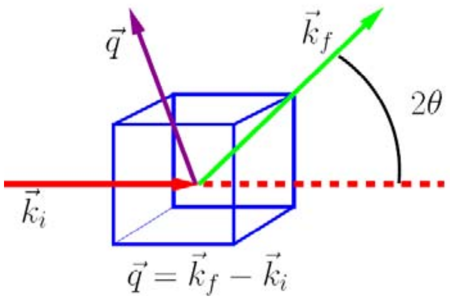
- The mechanical properties of small objects play a key role in numerous phenomena
- The local behaviour of Polycrystals is being still debated
- Strong lack of experimental techniques to probe deformation on this nanometer scale
- Coherent diffraction : a promising tool



→ Single grain behaviour

Phase Retrieval: specific case of strain objects

Kinematic theory of diffraction



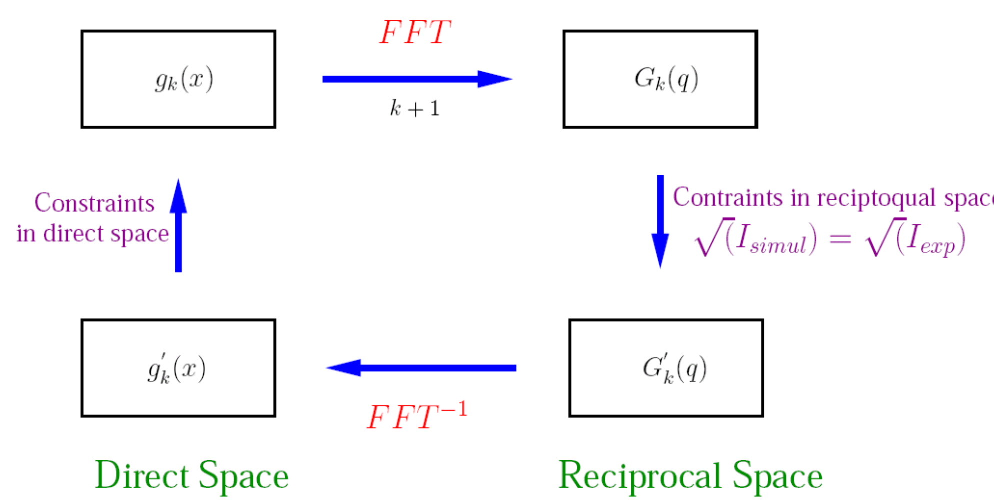
Displacement field

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

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

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)

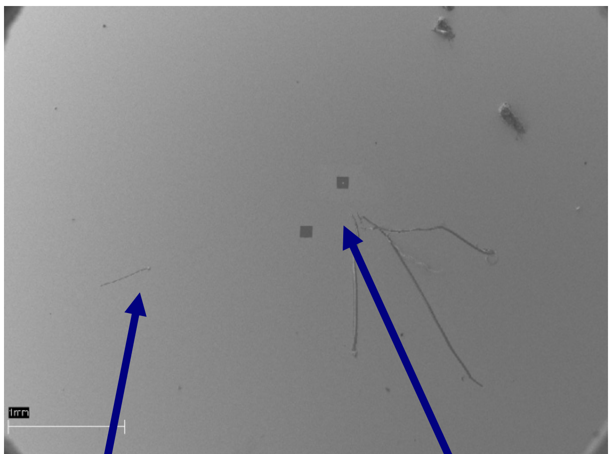
Our model sample

Main features

- Gold sample
- Special design

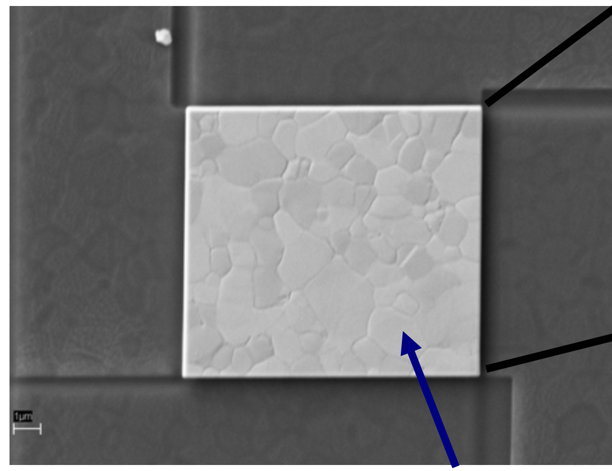
Why

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

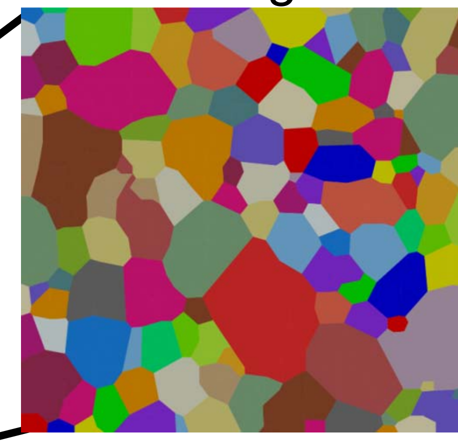


Homogeneous film (375nm thick)

150µm square area drilled by FIB



10*10µm polyscrystal

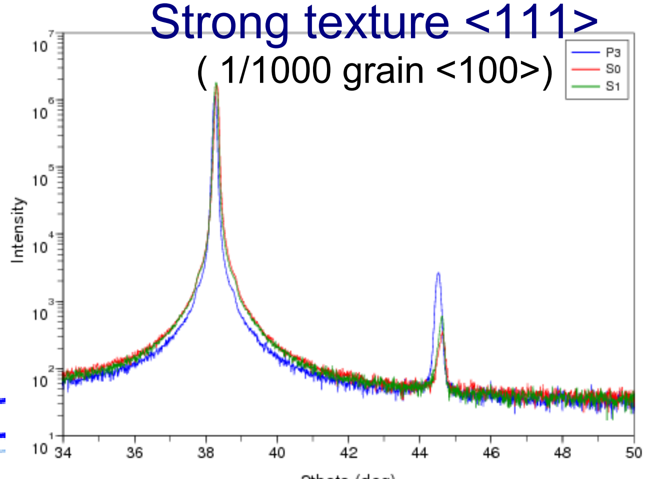


133 grains

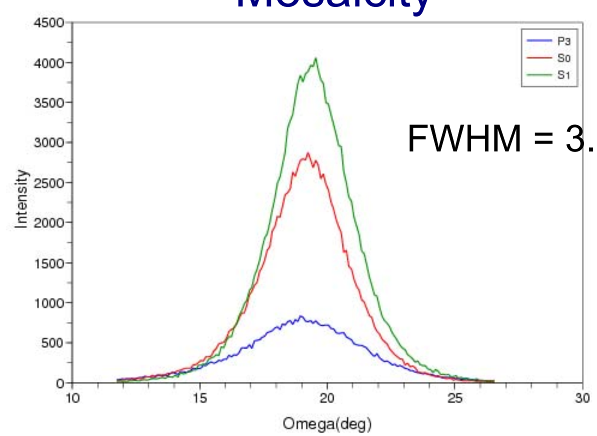
Single grain behaviour

Strong texture <111>

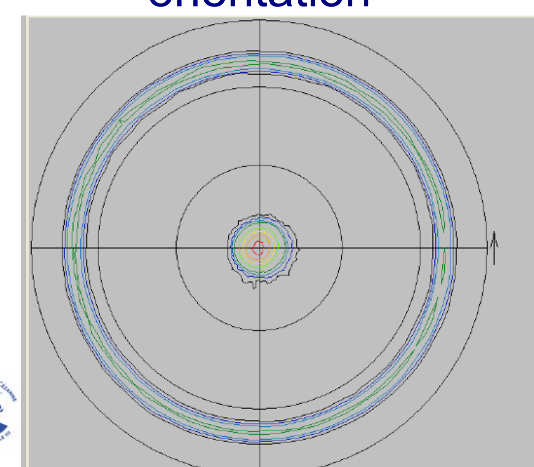
(1/1000 grain <100>)



Mosaicity

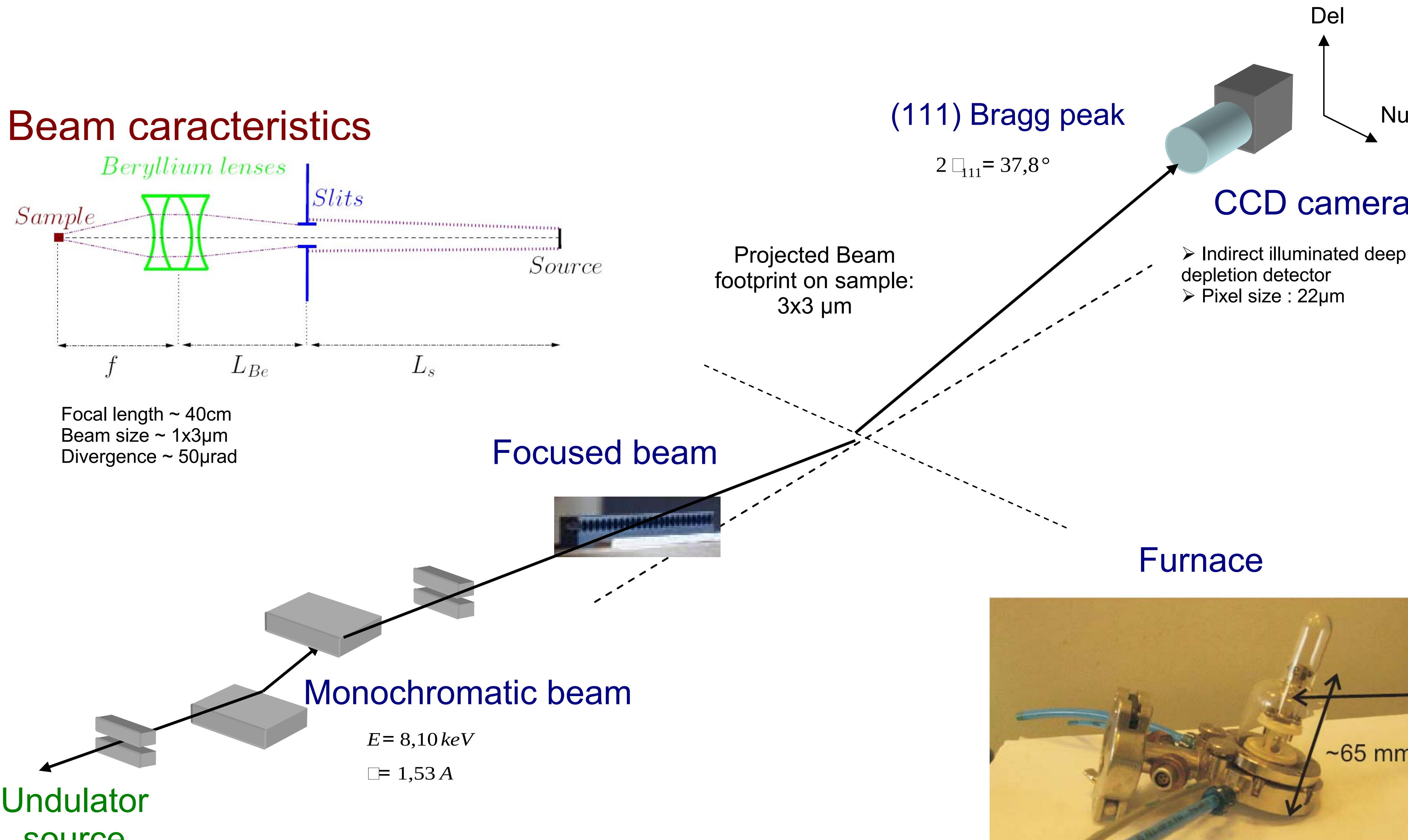


In-plane random orientation



Set up used on ID01 Beamline

Beam caracteristics



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

$E = 8{,}10\,keV$

$\lambda = 1{,}53\,Å$

CCD camera

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

Furnace

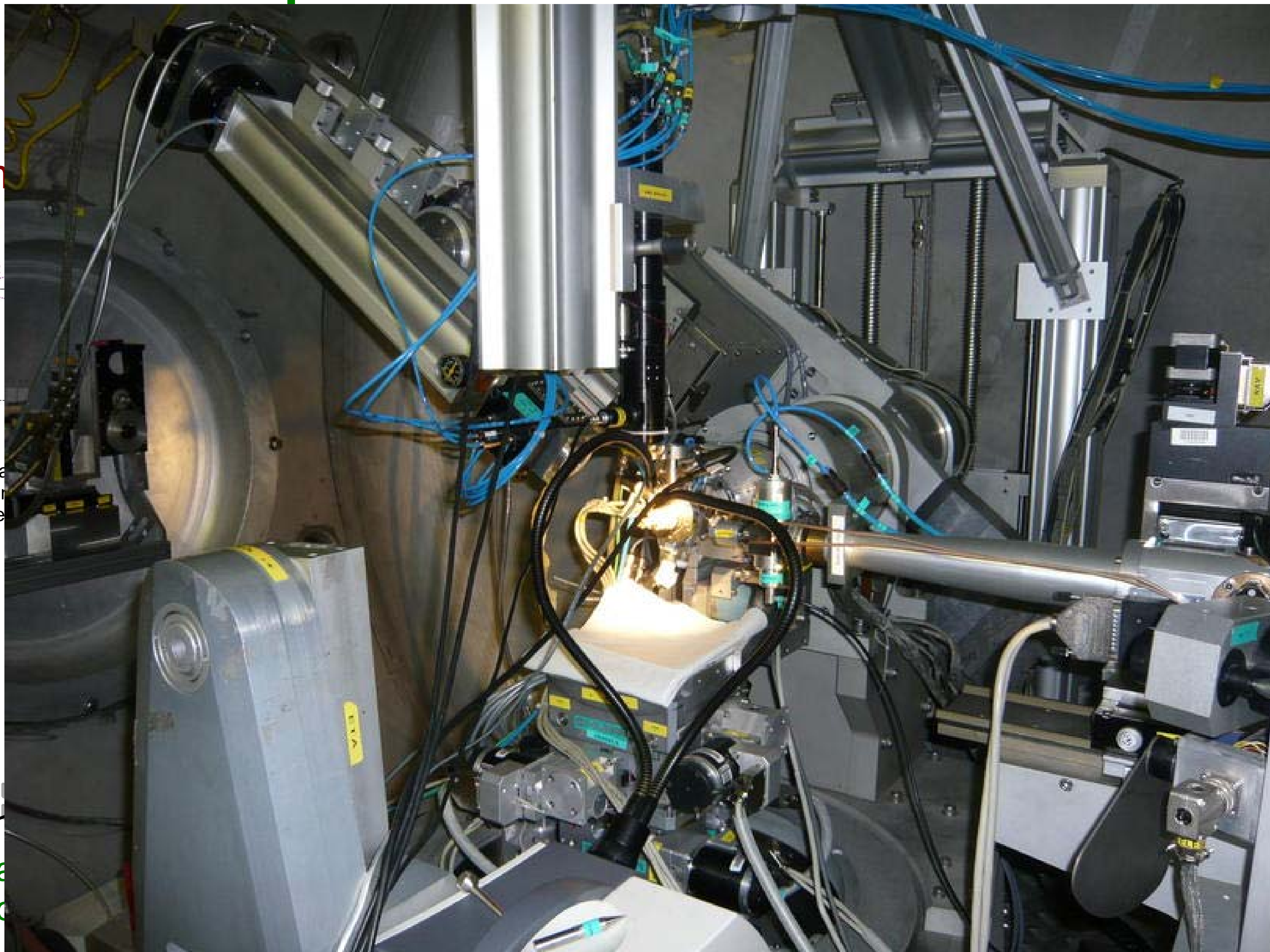
Set up used on ID01 Beamline

Bean

Sample

Foca
Bear
Dive

Undula
sourc



l

Nu

mera

ed deep

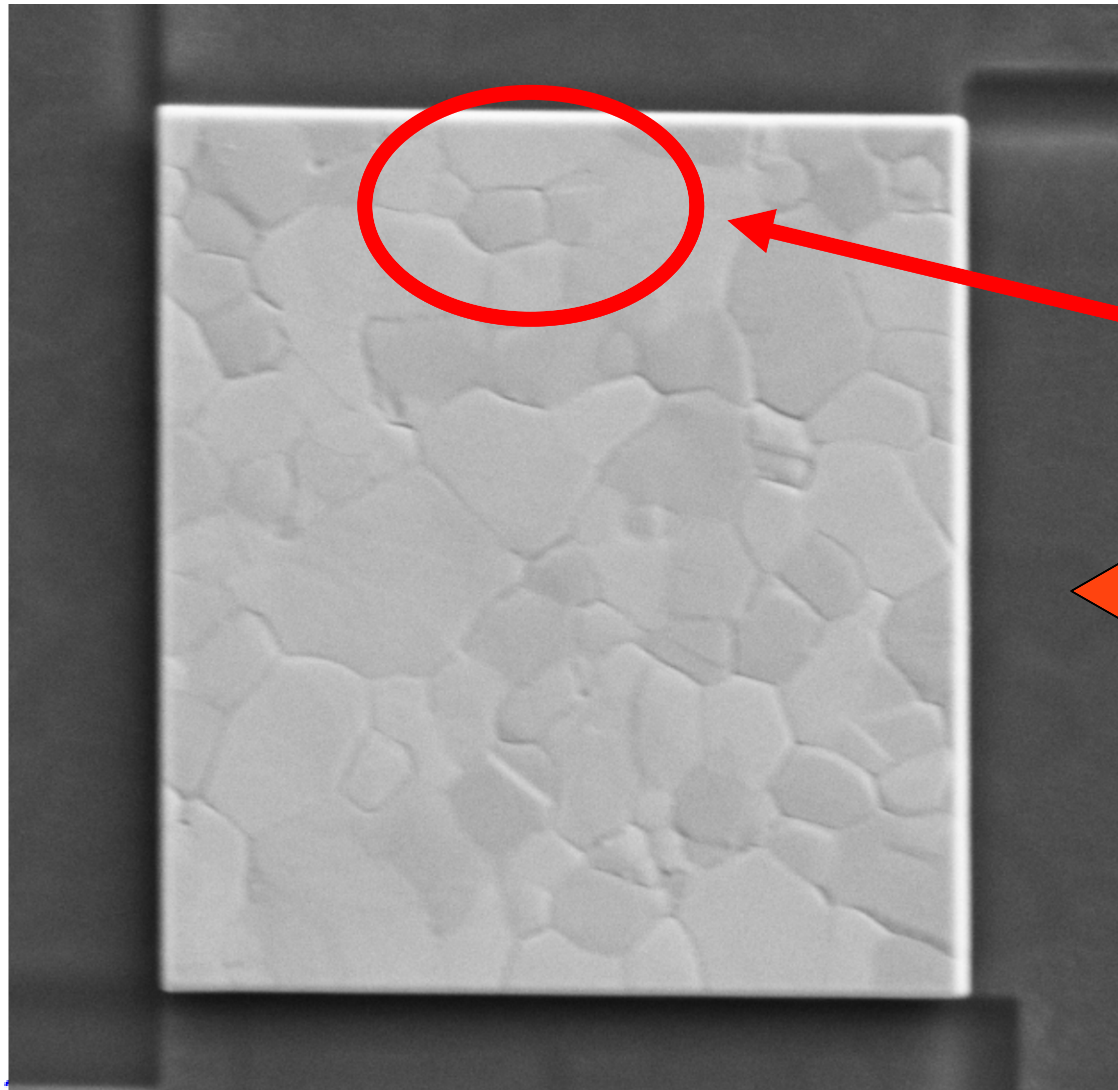
n

65 mm

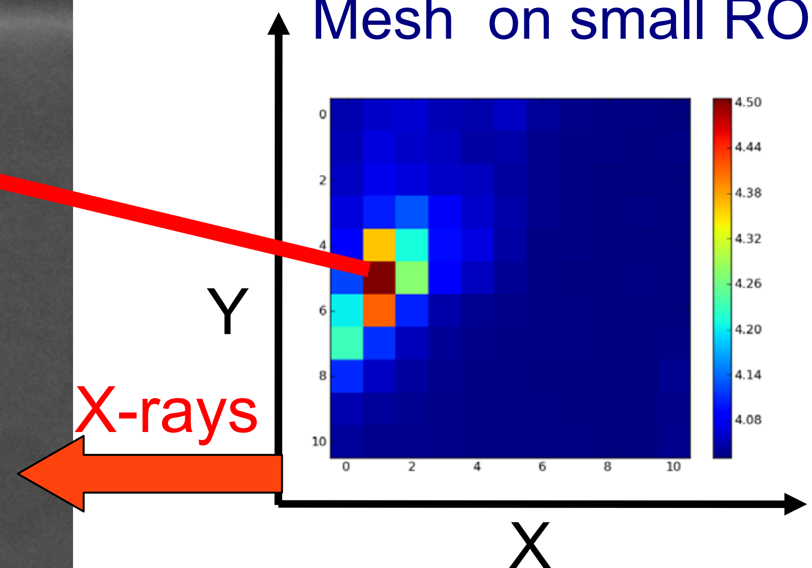
Grain determination

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

Granular Debye ring   Angular Map – Pole



Direct space
Mesh on small ROI



ed grain

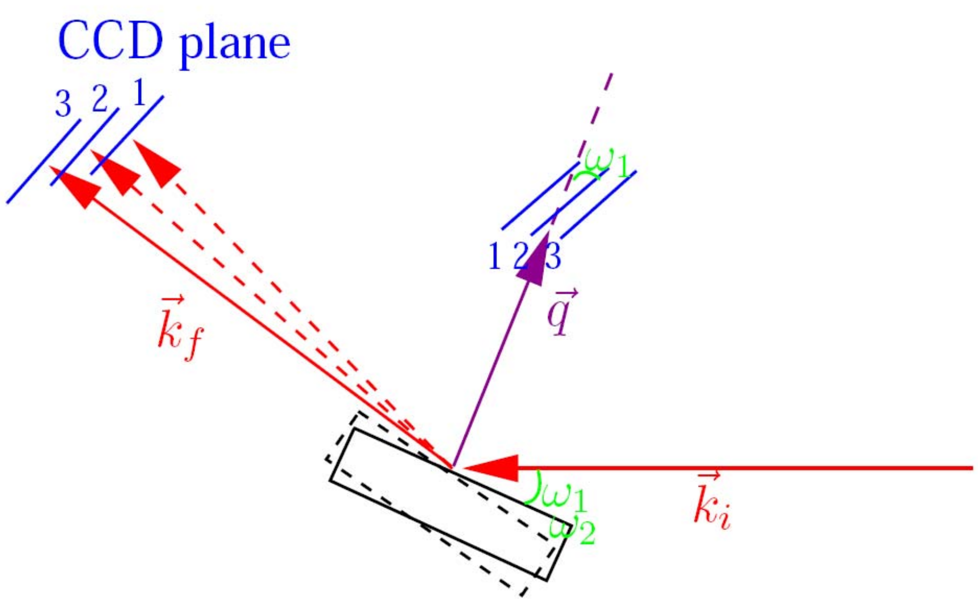
een
s

Projected
Beam size :
3*3 µm

3D Bragg Peak acquisition

Rocking curve peak (111)

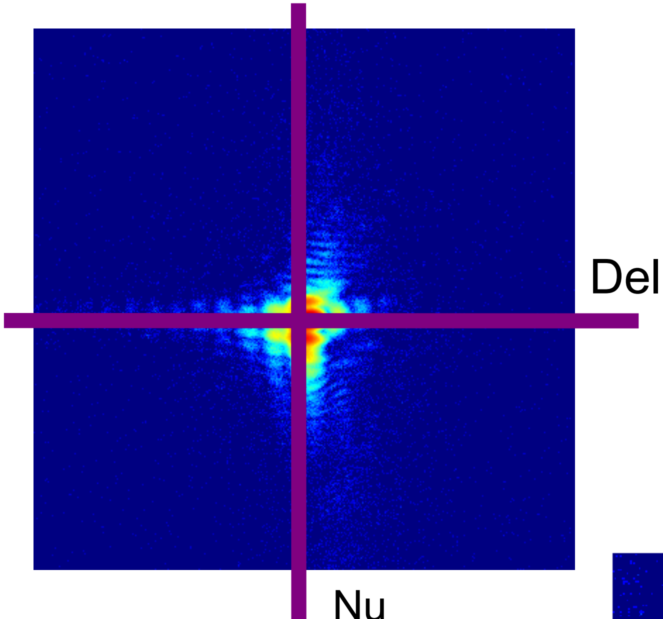
- Diffraction geometry
- Oblique cut in reciprocal space
- Weak resolution in Omega direction

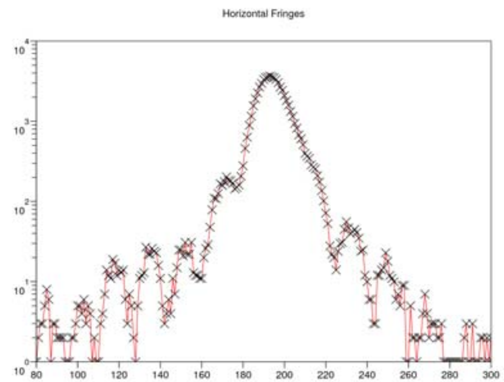


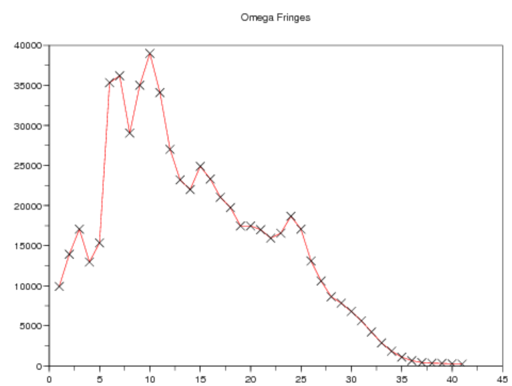
Acquisition characteristcs

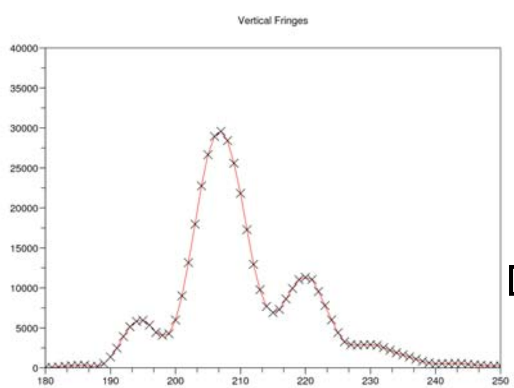
- Detector Indirect Deep depletion CCD 22µm pixels
- Single photon counting mode
- 3D acquisition time = 8 hours
- Max photons = 11 957 on one pixel
- Sum photon (all Bragg Peaks) = 32 .10^6 photons

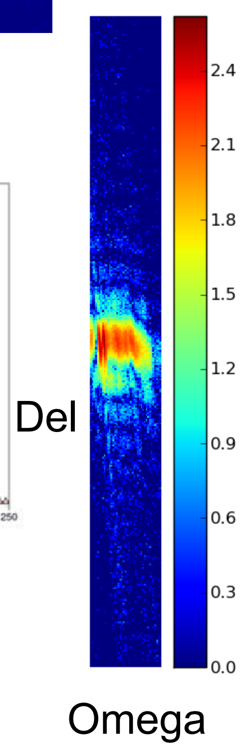
Oversampling and Characteristic dimensions











Reciprocal Space Resolution

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

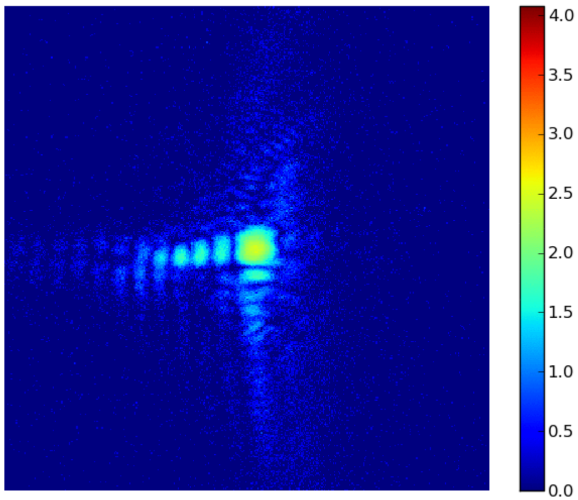
3D Oversampling

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

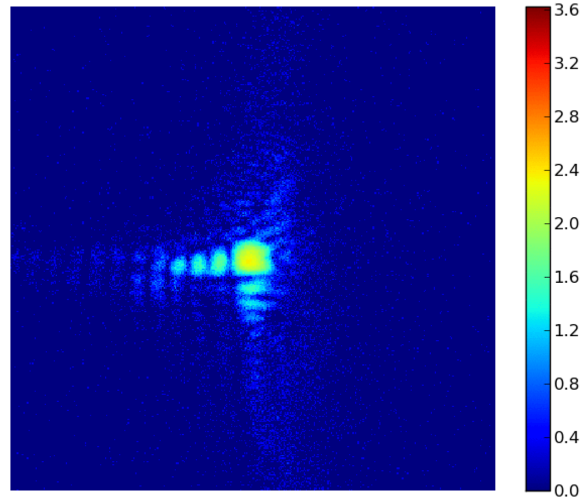
Dimension and Resolution in direct space

- 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)

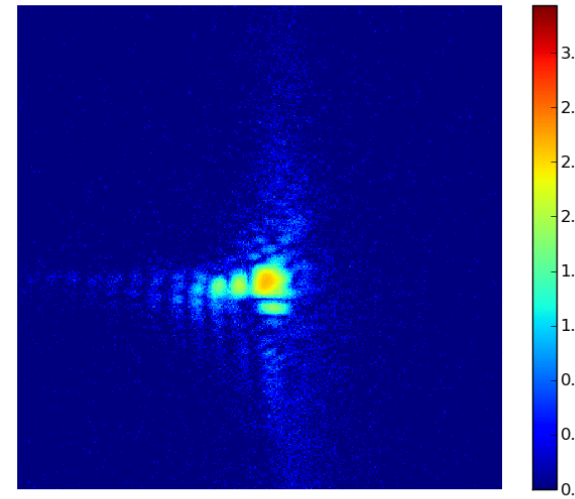
T=50deg



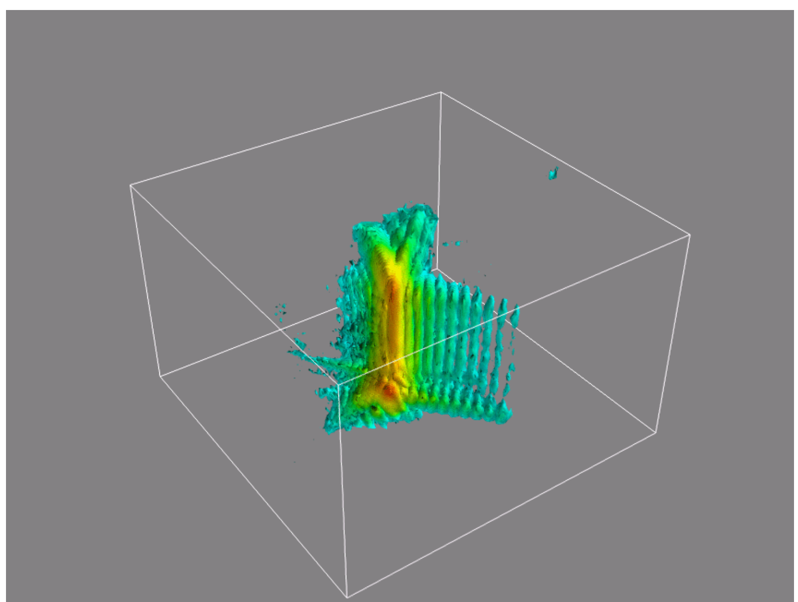
T=100deg



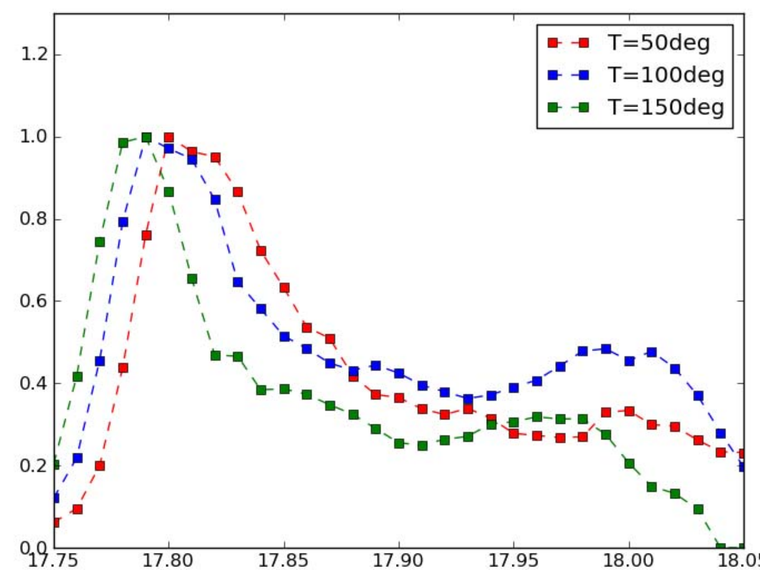
T=150deg



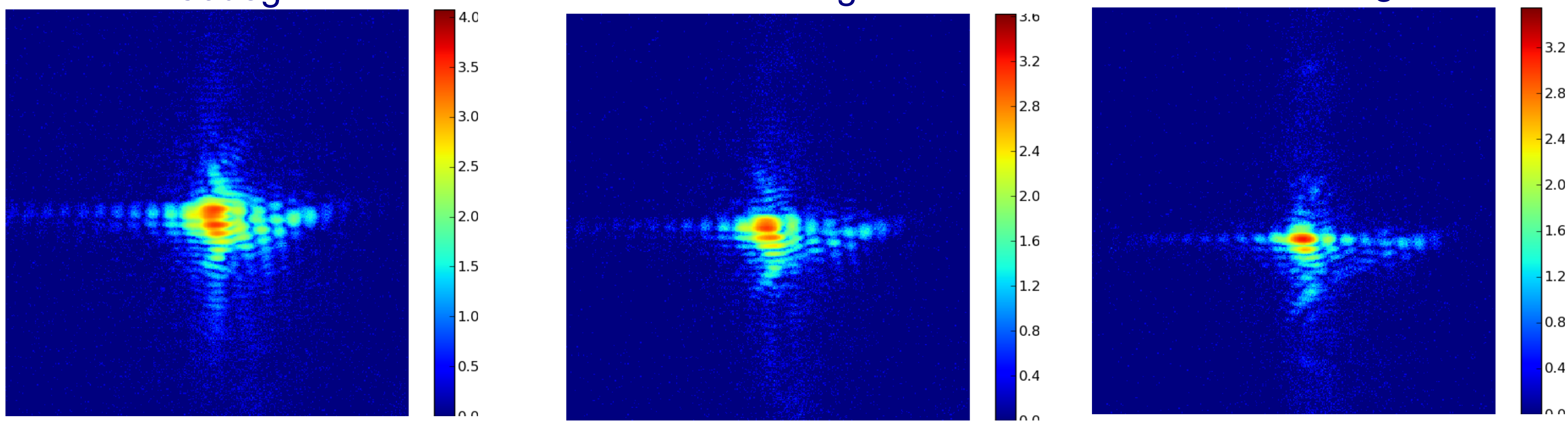
T=100deg



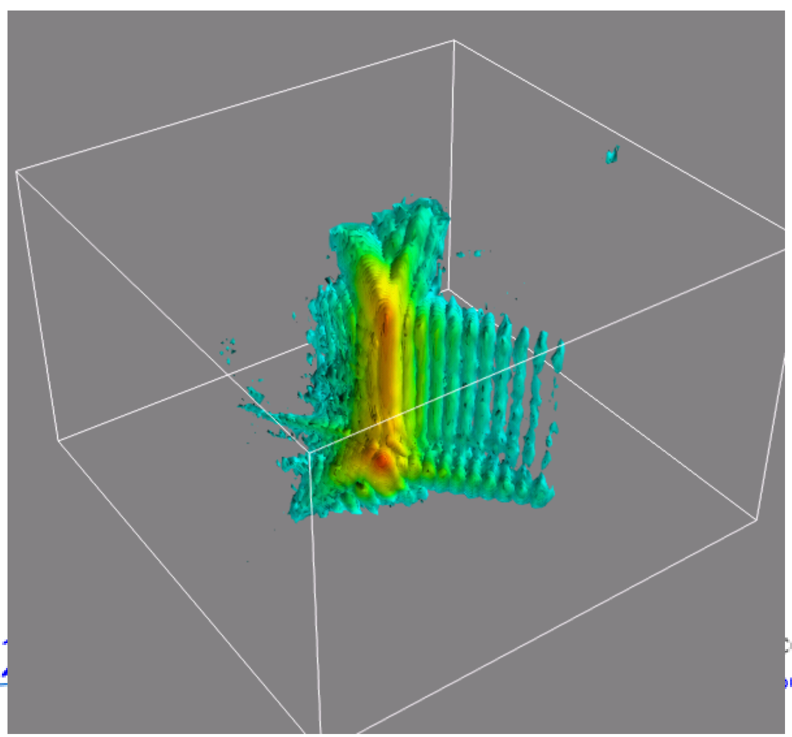
Rocking curve comparison



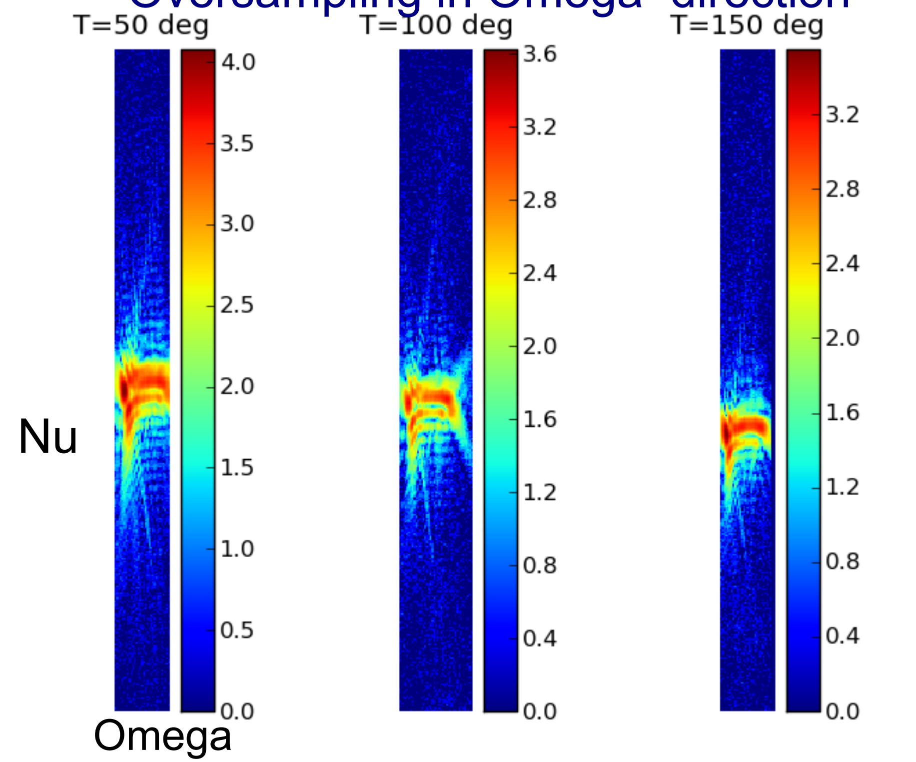
Experimental results



3D representation at T=100Deg



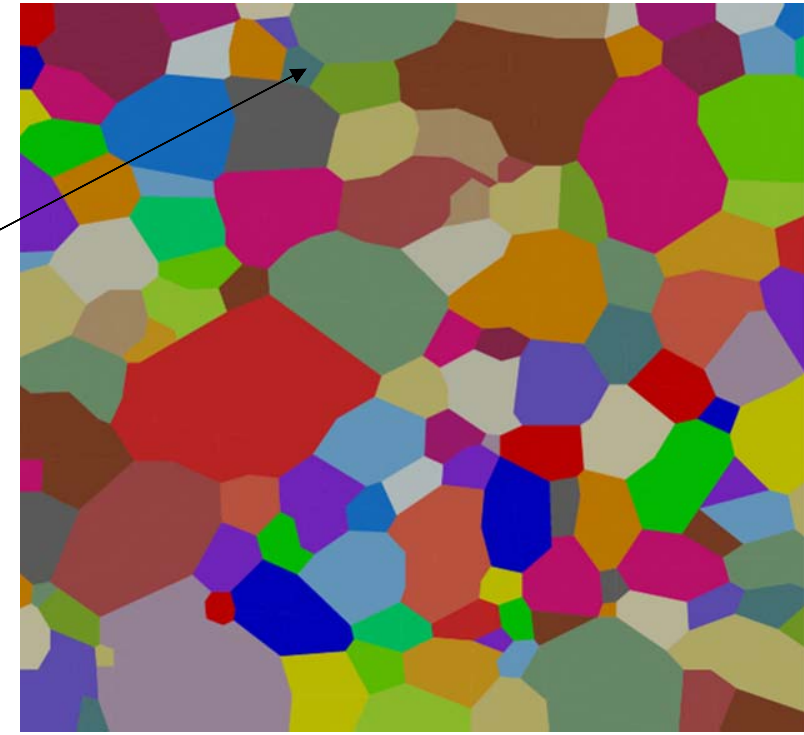
Oversampling in Omega direction



First reconstructions

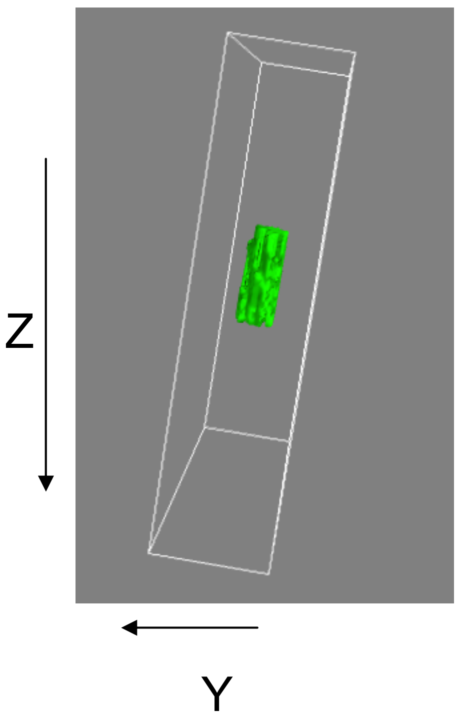
Retrieval Procedure

- Initial Support=cube estimated from fringe spacing
- 5 shrinkwraps
- Each Cycle 5*{750ER+250HIO }
- = 50 000 FFT (50 hours)

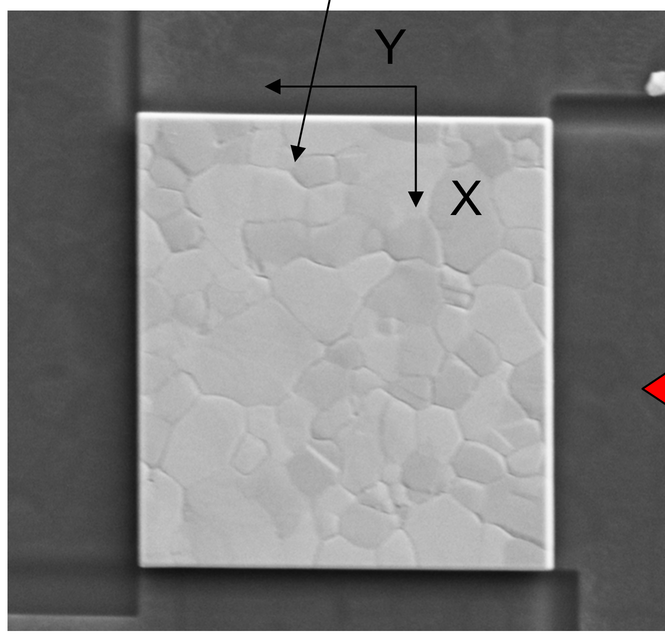


Shape

Reconstructed shape



Grain



X-rays

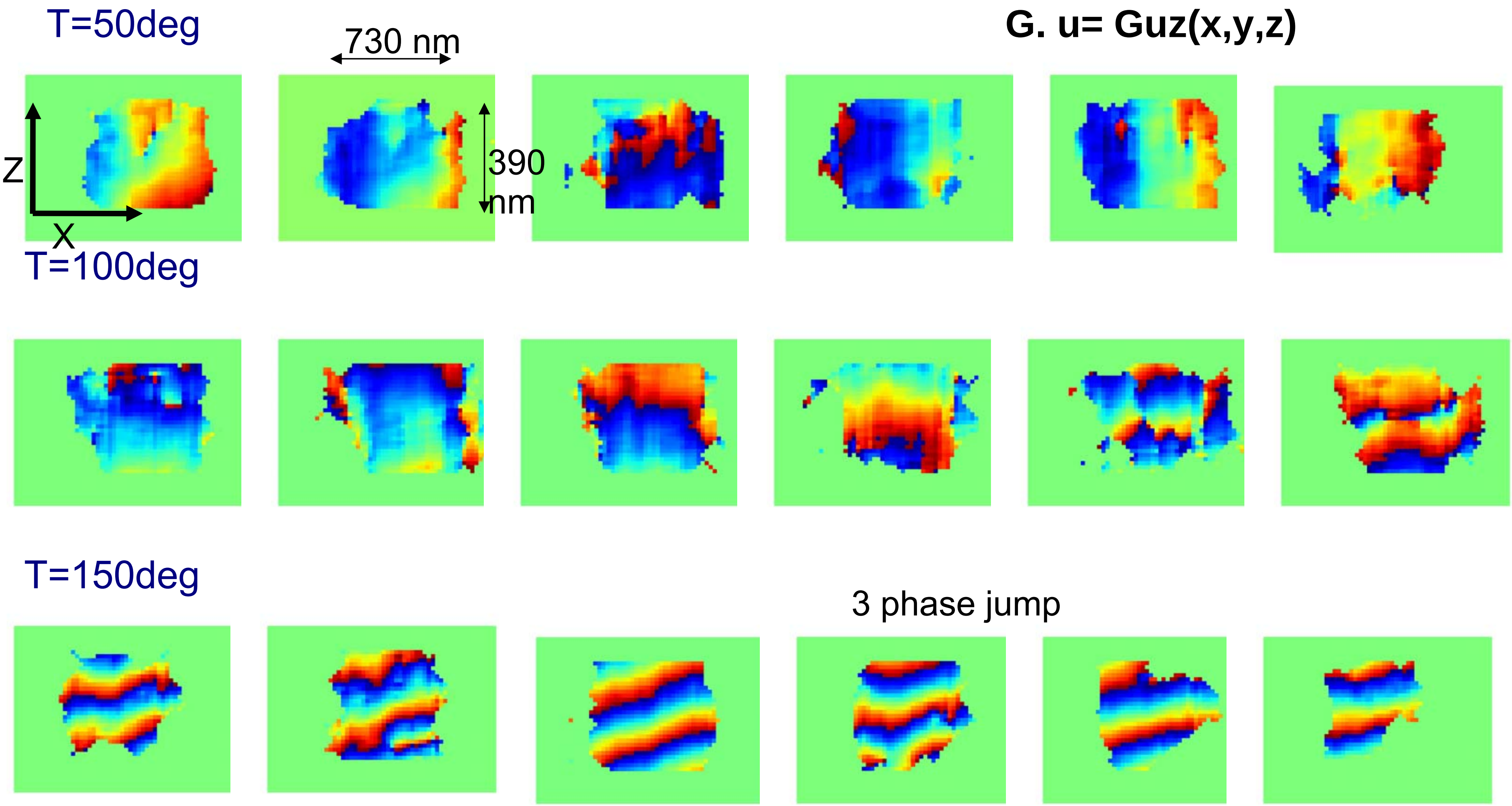
Grain raw dimensions

X= 750nm

Y= 240 nm

Z= 390 nm

3D Phase in direct space : G.u (Y perpendicular plane)



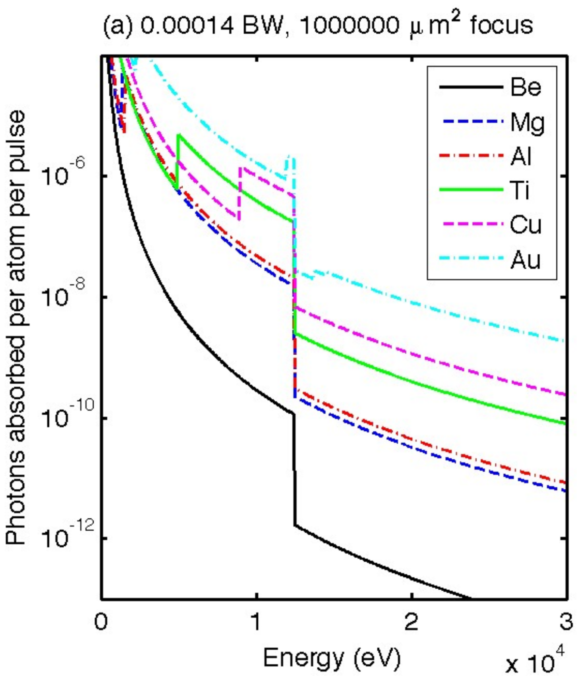
Progressive apparition of a gradient in Z direction

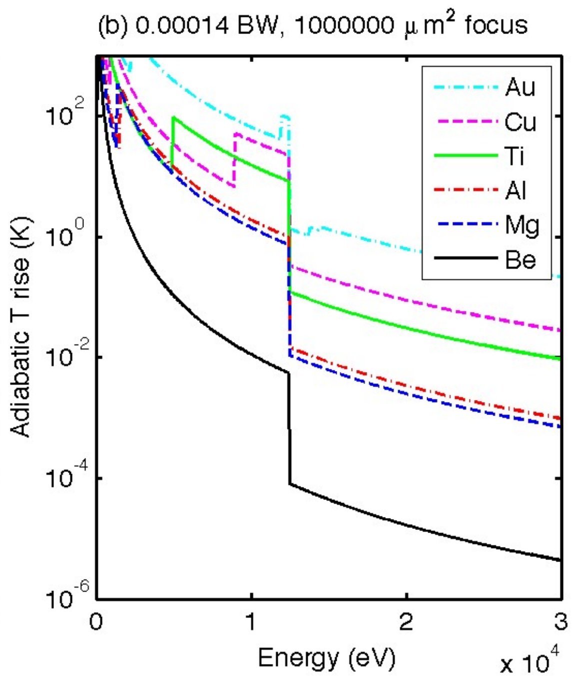
complementary calculations under progress

Xfel Perspectives

SASE1 Beamline

Parameter	Unit	Value
Photon energy	keV	12.4
Pulse duration	fs	<100
Bandwidth	0.1 %	0.8
Coherence time	fs	0.2
Divergence	µrad	1
Source size	µm	70
Source distance	m	1000
Photons per pulse	#	10^{12}
Peak brilliance	B	5×10^{33}





Coherent longitutinal length = 60 nm

could be improved (Monochromator)

Comparison

Photons/s = 10e12 ID01 focused beam

Au sample

- At 12.4 KeV 10% absorption for 400nm thick sample
- 1 pulse : 20 000 deg C

→ Aluminum polycrystal

Evaluation of thermic relaxation between two pulses

New possibilities ?

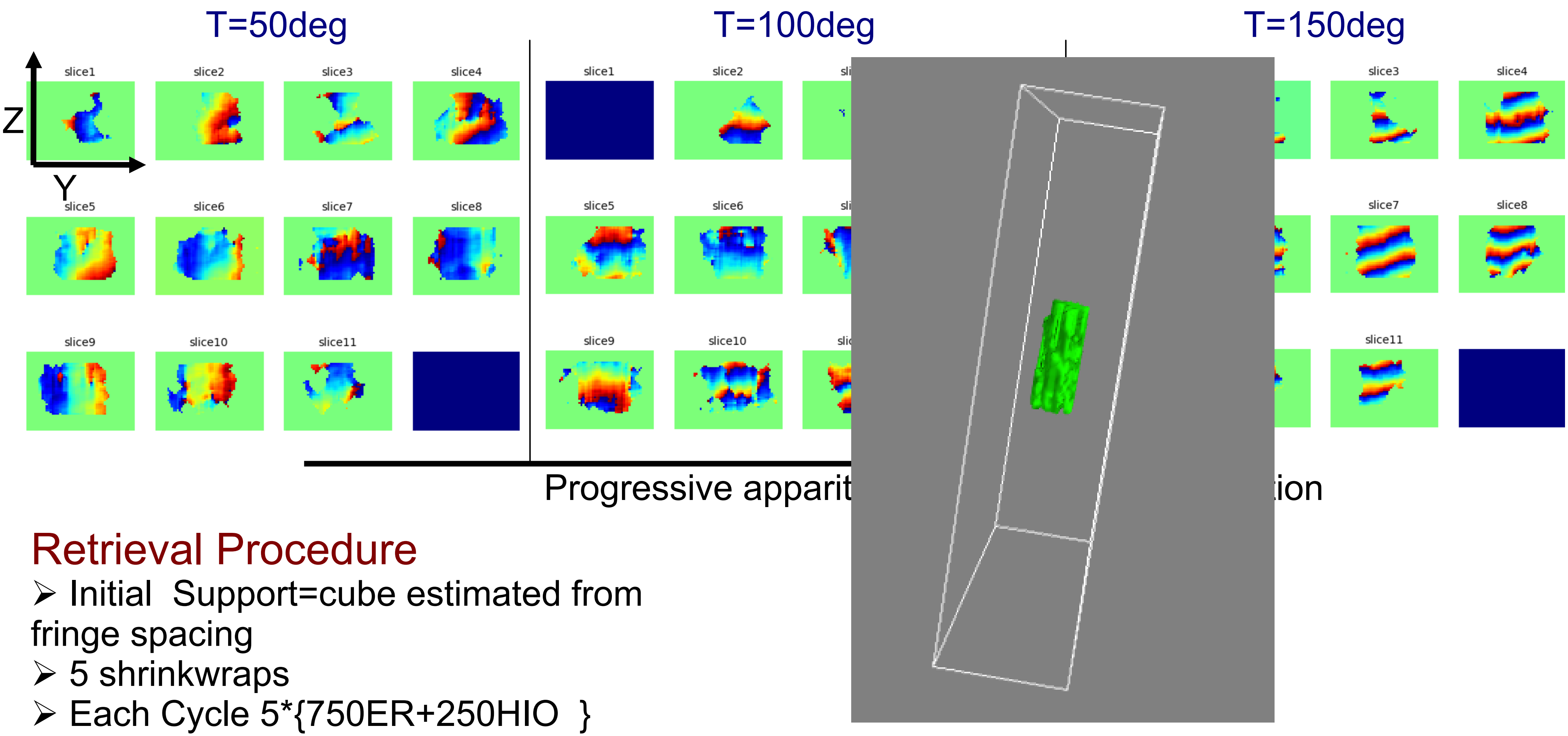
- Dynamic of defect (dislocations)
- Grain growth
- ....

Thank You For Your Attention

First reconstructions

3D Phase in direct space : G.u

(111) Bragg peak
G. u= Guz(x,y,z)



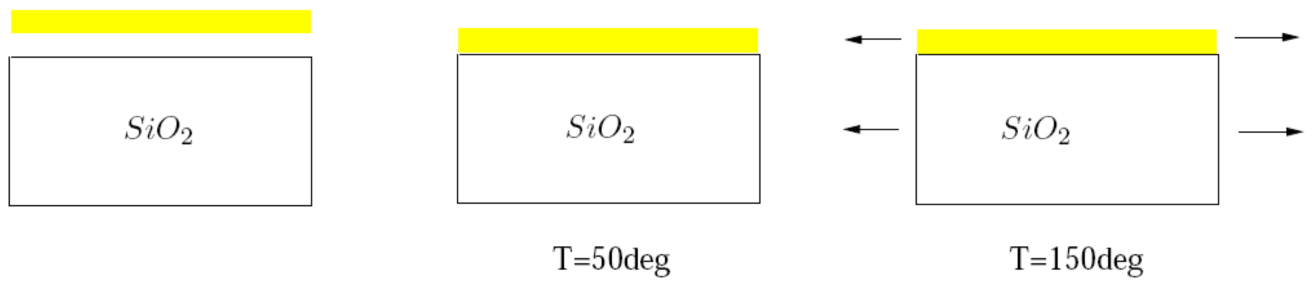
Progressive apparit… …ion

Retrieval Procedure

- Initial Support=cube estimated from fringe spacing
- 5 shrinkwraps
- Each Cycle 5*{750ER+250HIO }
- = 50 000 FFT (50 hours)

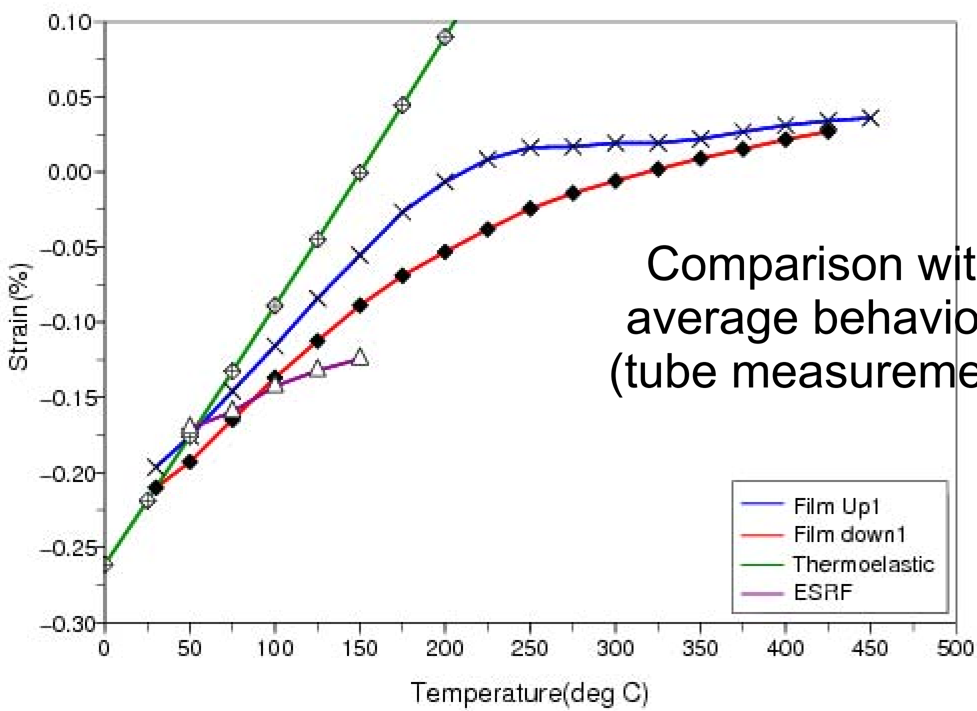
Could be local minima : complementary calculations under progress

Some average result



Thermo-elastic behaviour

$$\epsilon_{//} = -\left[\alpha_{Au}\Delta(T) - \alpha_{SiO_2}\Delta(T)\right]$$



Comparison with average behaviour (tube measurement)

Sample imperfections

- No perfect adhesion with substrat
- Joint grooving
- Incertitude on furnace calibration

Numerical aspect

Our cluster

- 62 processors
- GigE communication

Parallel computing

- 50 000 iterations of retrieval algorithm for a 384*384*31 pixel tensor == 20 days of calculation on a classical computer
- Parallel is needed for large tensor of data
- Helpful for testing numerous parameters

Open Source Solutions

- FFTW 2.1.5 library C/C++/Fortran functions (MPI)
- New 3.3alpha version
- Python distarray Package under developement (B. Granger)

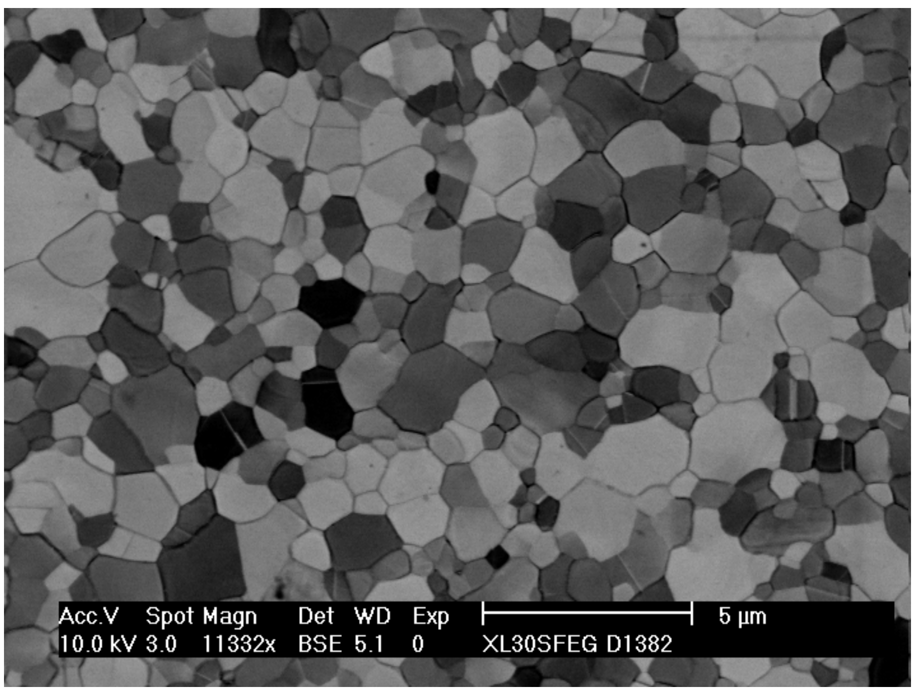
A parallel FFT need significant communication between processors

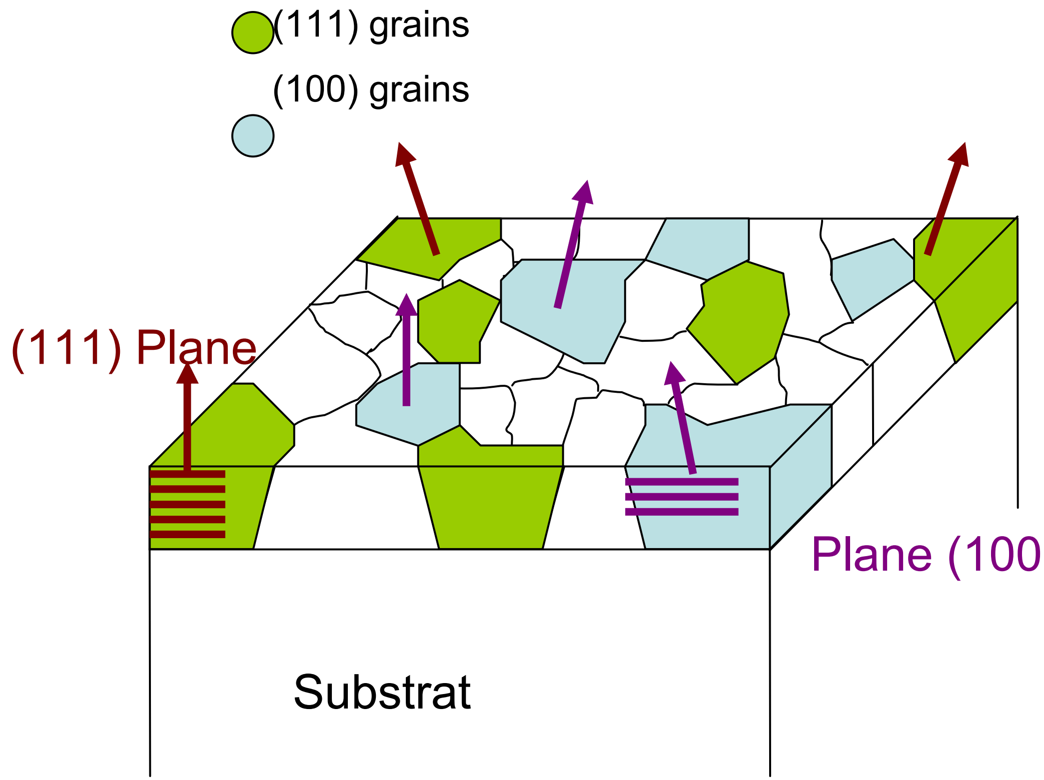
Benefits from multicore

Polycristalline Thin Film

Main Features

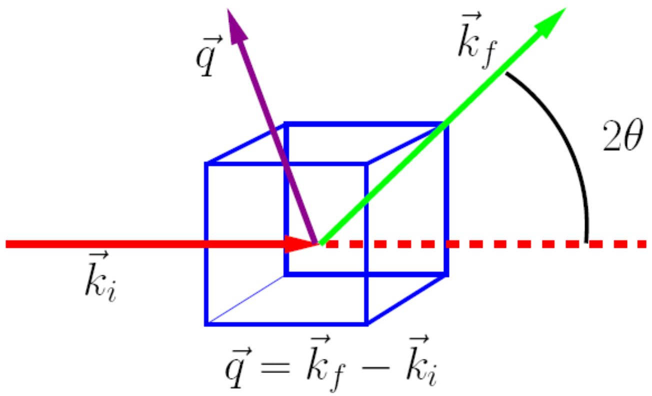
- Grain size distribtion
- Texture
- Orientation inhomogeneities
  - In plane
  - Out-of plane





Kinematic theory of diffraction

Intensity distibution in reciprocal space



Complex Magnitude

$$A(\mathbf{q}) \propto \sum_n f_n(\mathbf{q}) \exp\left(\imath \mathbf{q} \mathbf{r}_n\right)$$

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

$$A(\mathbf{q}) = \sum \left(f_n \exp \imath(\mathbf{q}.u(\mathbf{r})) \right) \exp \imath(\mathbf{q}.\mathbf{R})$$

Takagi approximation

S. Takagi. *J. Phys. Soc. Jpn*, 1969.

$$A(\mathbf{q}) \propto TF\left\{\rho(\mathbf{r}).\exp\left(\imath \mathbf{G}.\mathbf{u}(\mathbf{r})\right)\right\}$$

Diffracted Intensity

$$I(\mathbf{q}) = \left| \sum_n f_n(\mathbf{q}) \exp \imath \mathbf{q} \mathbf{r}_n \right|^2$$

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