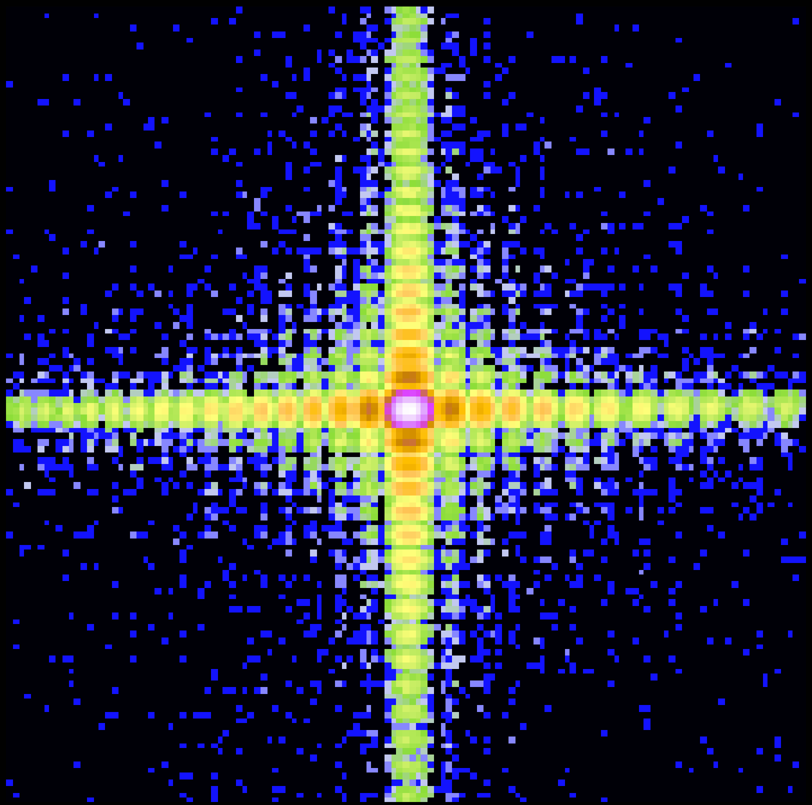
Coherent X-ray diffraction for Condensed matter physics



Sylvain RAVY

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91192 Gif-sur-Yvette
France

Collaborations

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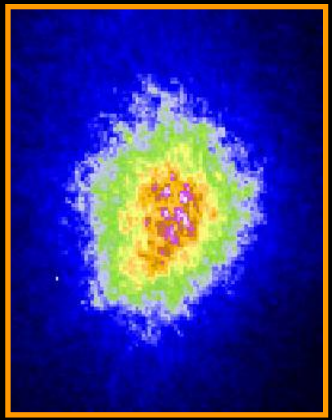
CRISTAL: E. Elkaïm, P. Fertey, F. Legrand
CRISTAL's users…

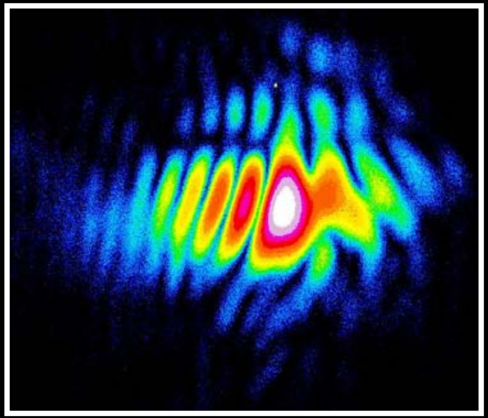
Starting from CXD experiments
performed successfully or not,
on 3rd generation X-ray source (ESRF, SOLEIL)

The goal of this talk is
to give some examples of experiments
taking advantage
of the brilliance of XFELs

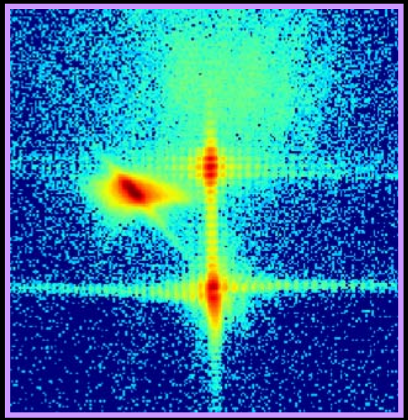
All images obtained in 100-1000s with 10^9-10^{10} ph/s
On XFEL the same in one shot 10^{12} ph

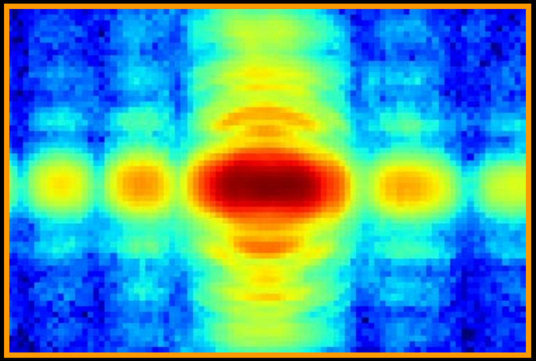
Outline



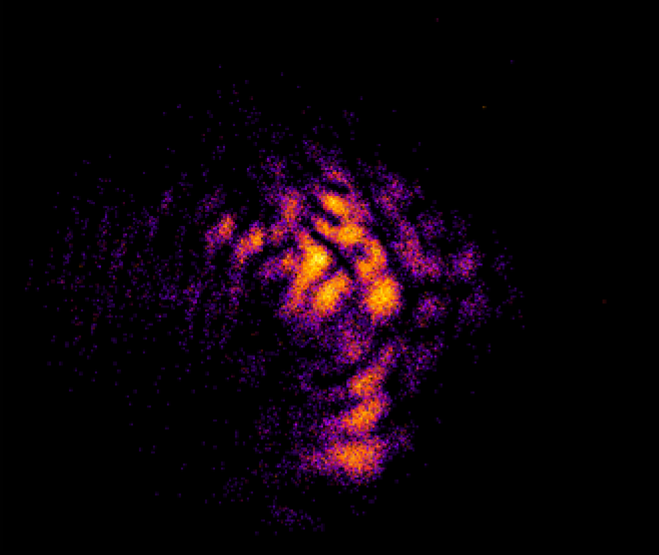


1. Coherent diffraction
2. Fluctuations
3. Imaging
4. Topological defects
5. CXD under pressure
6. Soft surfaces
7. Conclusion



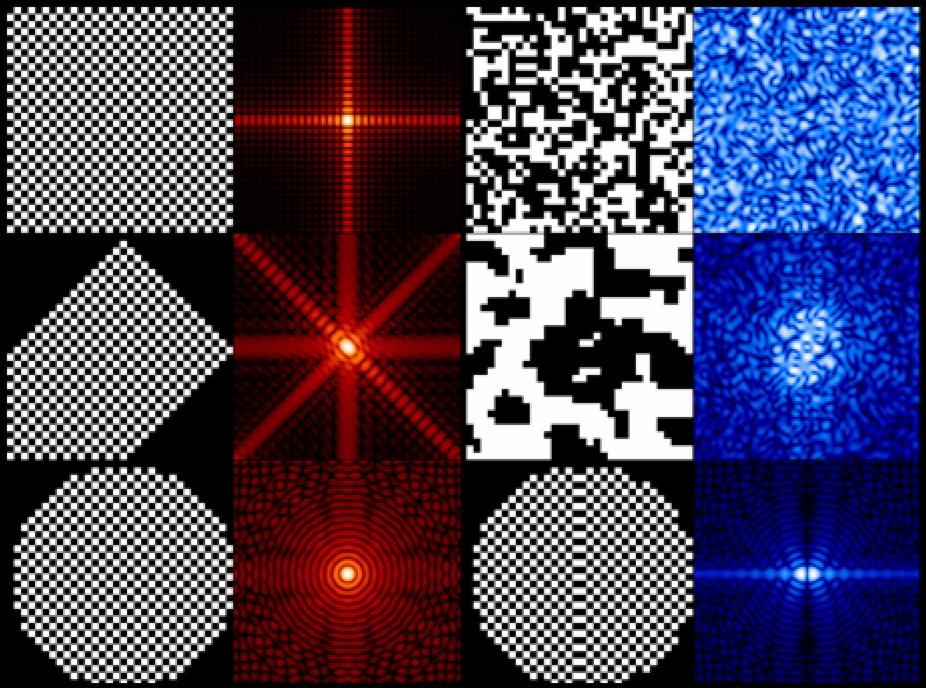


1.Coherent Diffraction



In condensed matter, typical experiments consists in measuring Fringes or Speckles

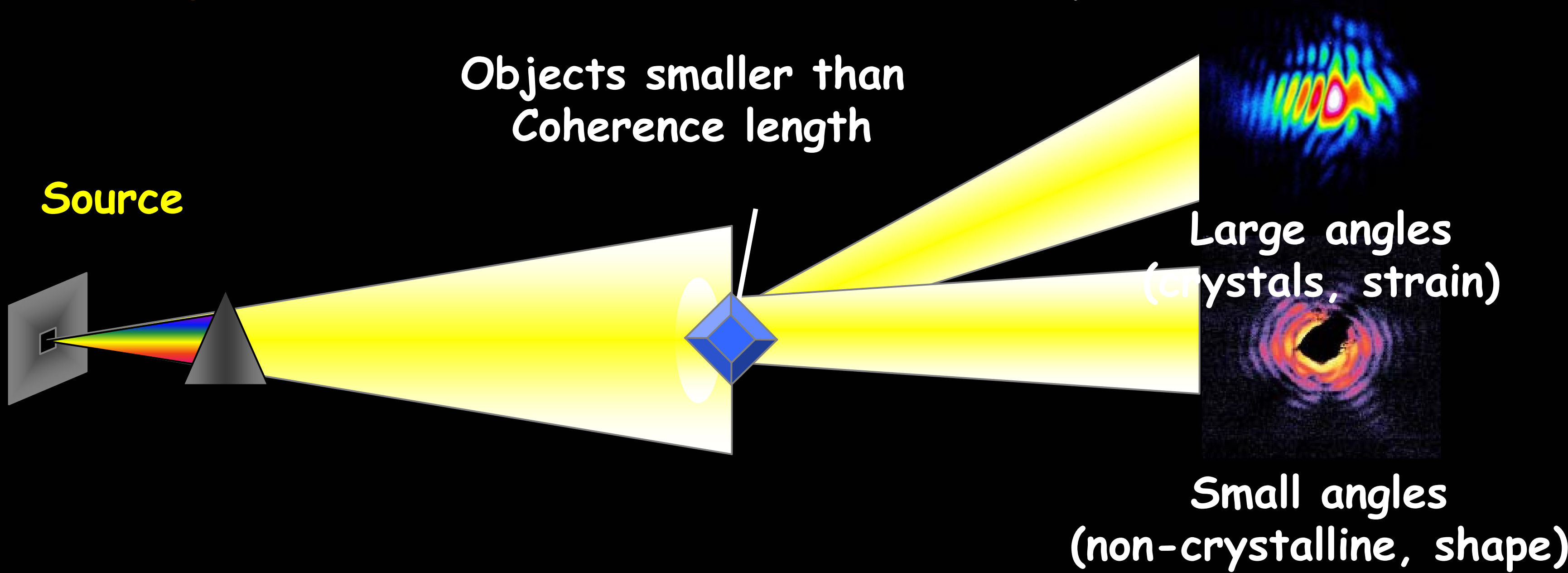
μ-crystal
e.g. strains

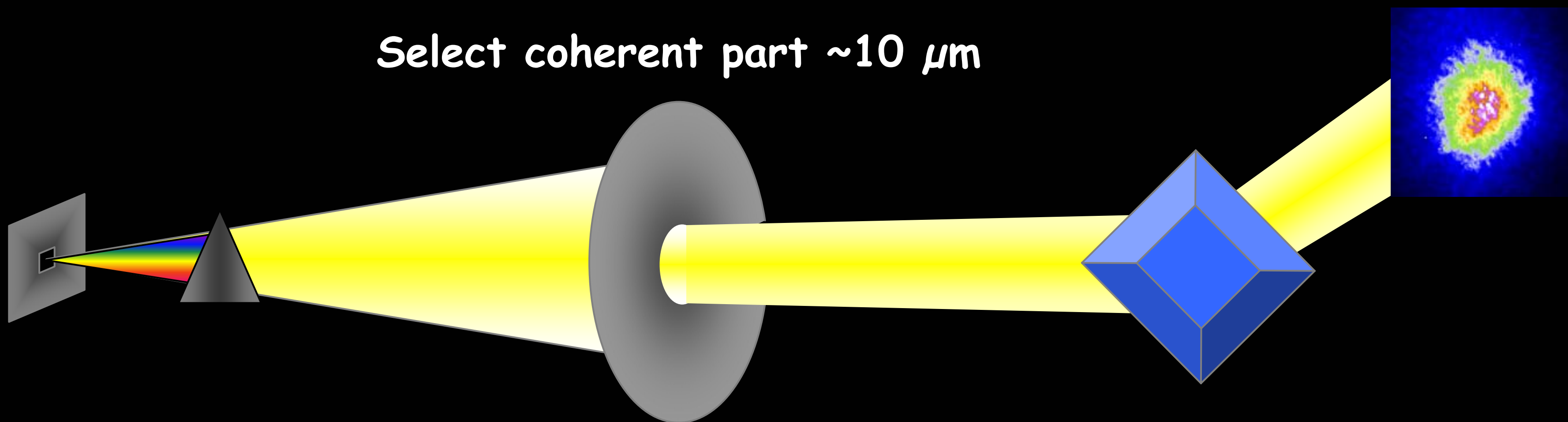


Disorder
e.g. fluctuations

Defects
e.g. dislocation

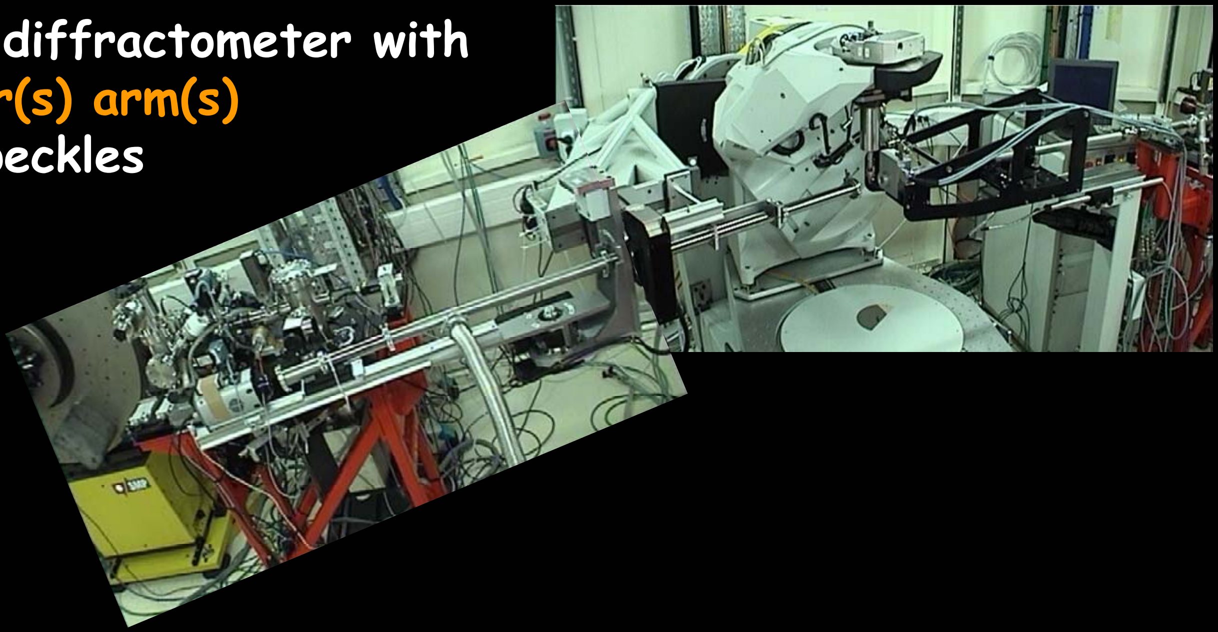
With 3rd generation sources, there is different ways to work



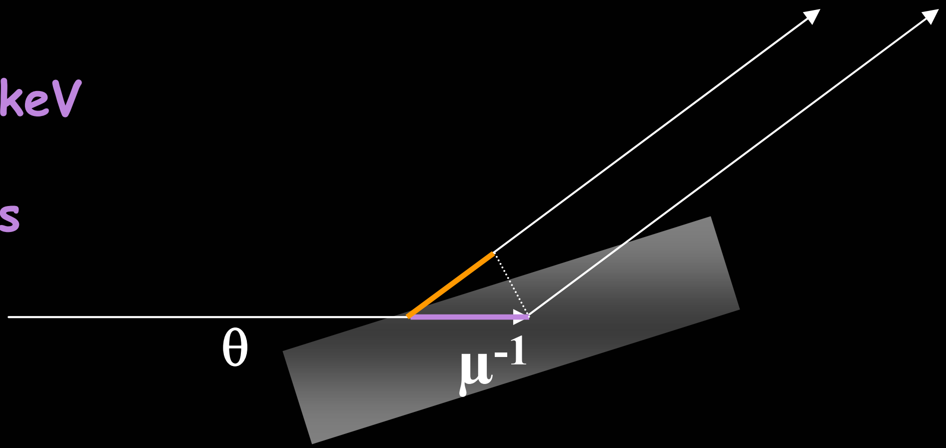


Specific issues for large angle CXD:

• Need of a diffractometer with
long detector(s) arm(s)
to resolve speckles



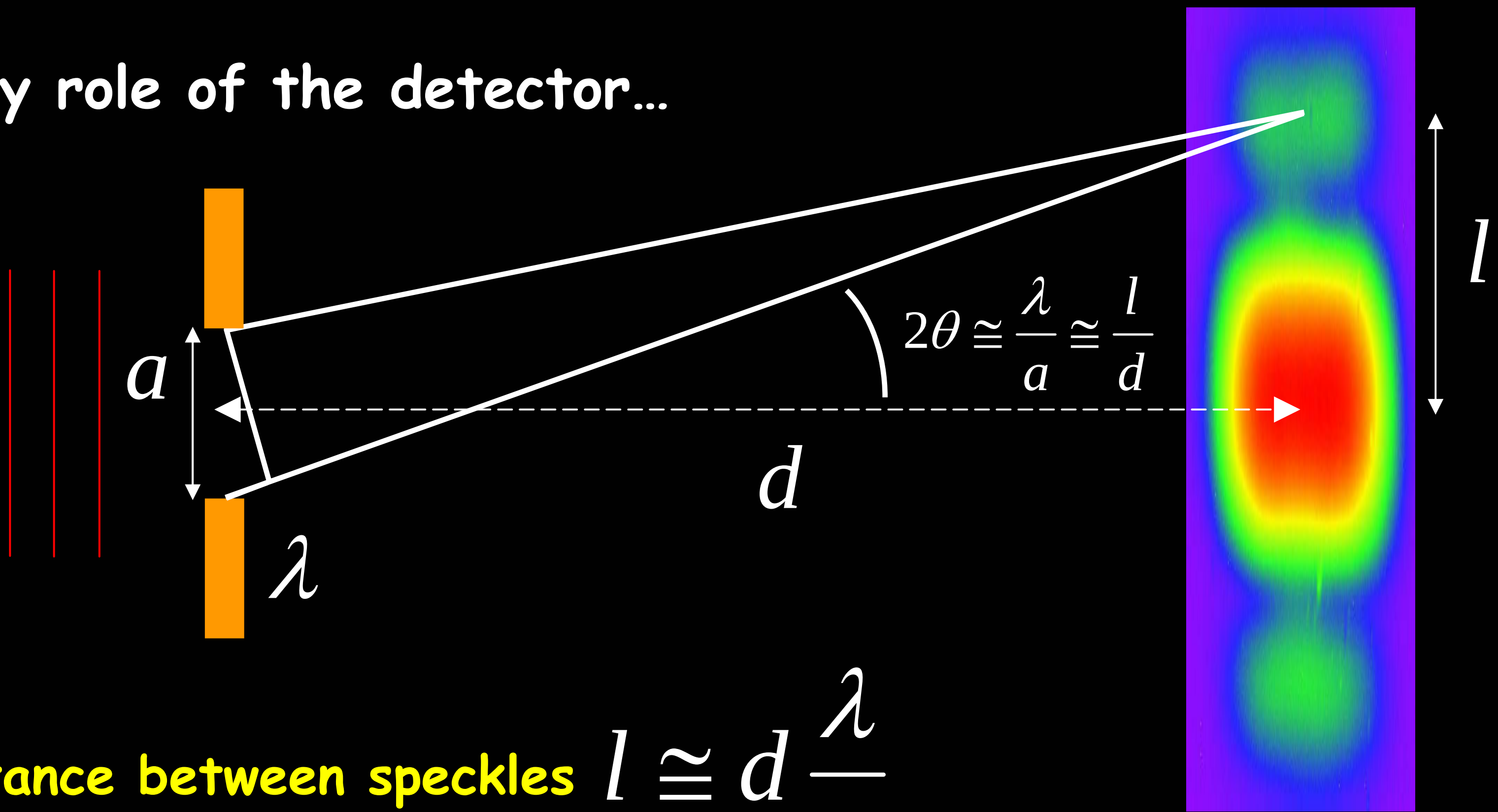
• For coherent diffraction @ 12.4 keV
the longitudinal coherence length
might be a limitation at large angles



Path length difference:
$\delta = \mu^{-1} - \mu^{-1}\cos(2\theta) = 2\mu^{-1}\sin^2\theta$

$\delta < L_L \sim 0.5\ \mu m$ Si(111)

Key role of the detector…

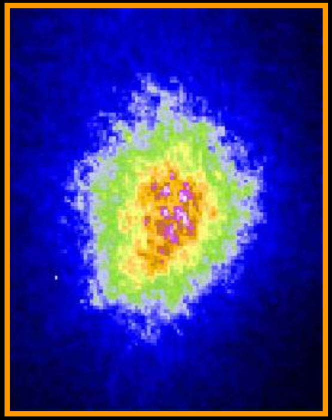


Distance between speckles $l \cong d\,\frac{\lambda}{a}$

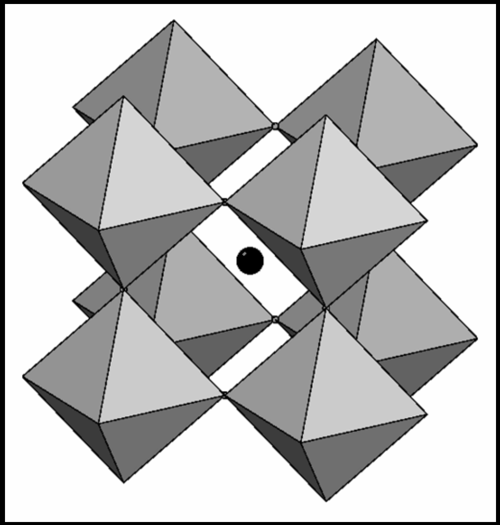
'object' size		
Det. distance	1 m	10 m
10 μm	10 μm	100 μm
1 μm	100 μm	1 mm
100 nm	1 mm	10 mm

…divided by
an oversampling ratio > 3

2. Fluctuations
The case of $SrTiO_3$



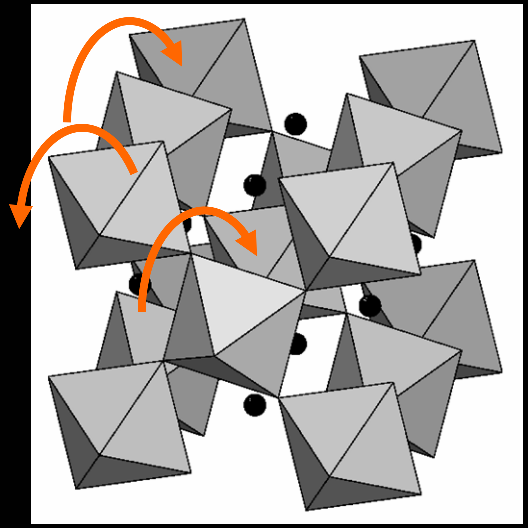
The $SrTiO_3$ antiferrodistortive transition



Cubique phase: Pm3m (Perovskite)

T_c=110 K

Tetragonal phase: I4/mcm



Alternate tilts of TiO_6 tetrahedra

Doubling of the unit cell in the 3 directions.

New reflections (1/2,1/2,1/2)

$SrTiO_3$: a classical example of phase transition

Soft mode at the R point of the BZ
on a tranverse acoustic branch

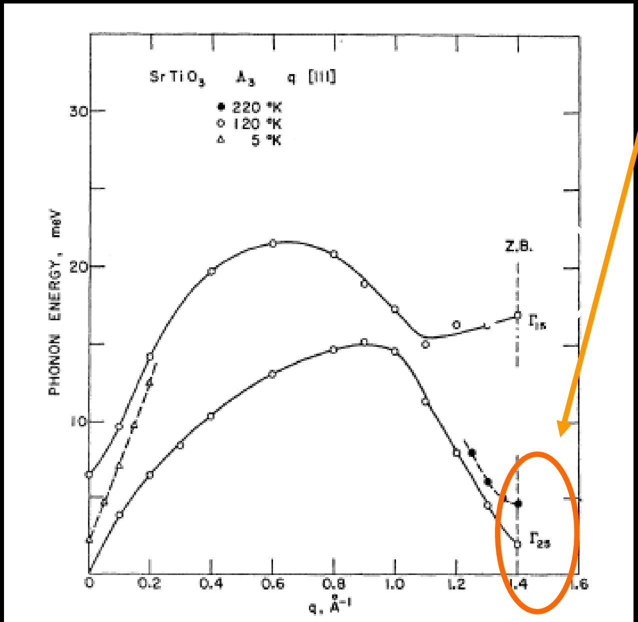
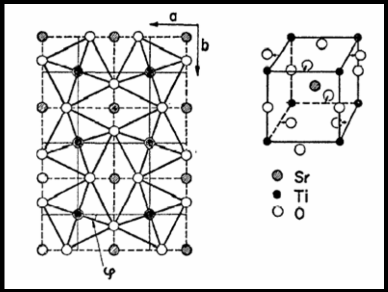
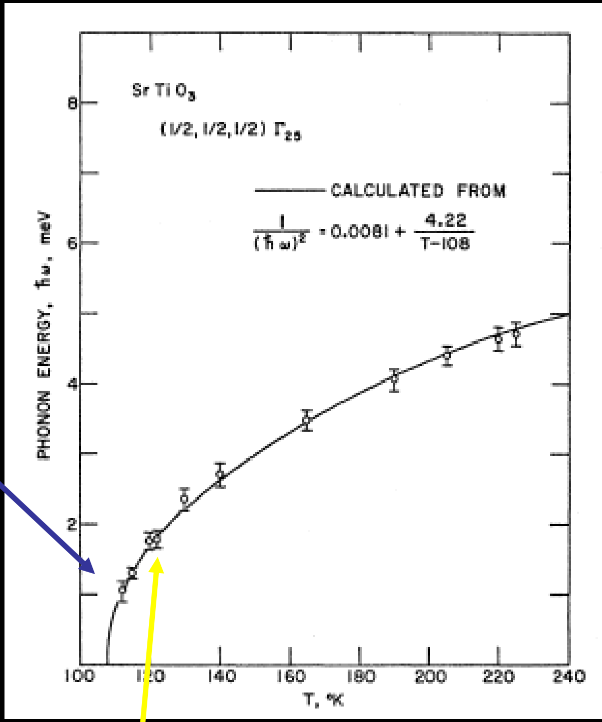


FIG. 3. The lowest transverse branches with q∥[111]. The zone boundary corresponds to $q=1.396\ \text{Å}^{-1}$ at 120°K. Conversion factor from meV to the optical wave number (cm^{-1}) is 8.07.

Shirane, Yamada, Phys. Rev. 177, 858 (1969)

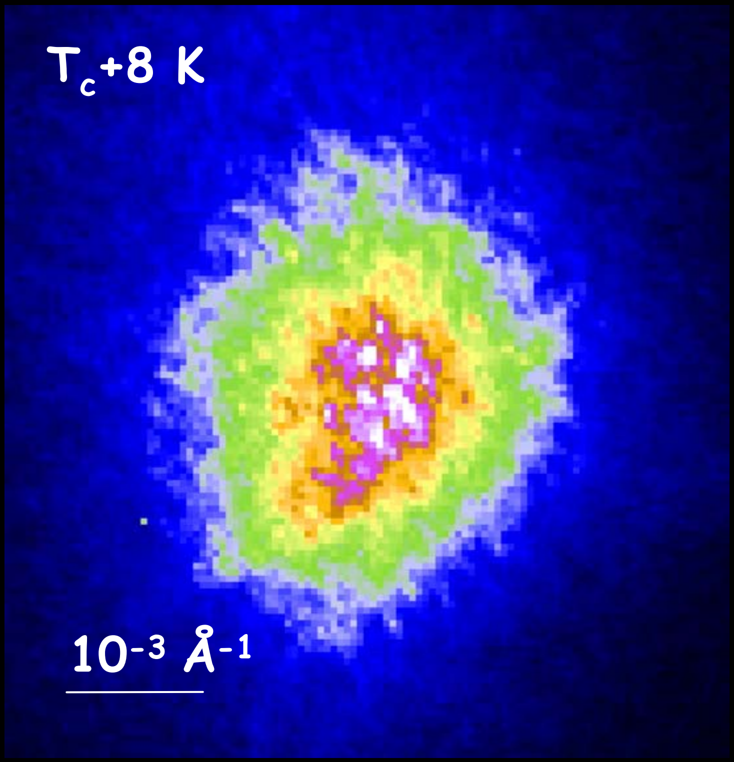
Mode critically softens



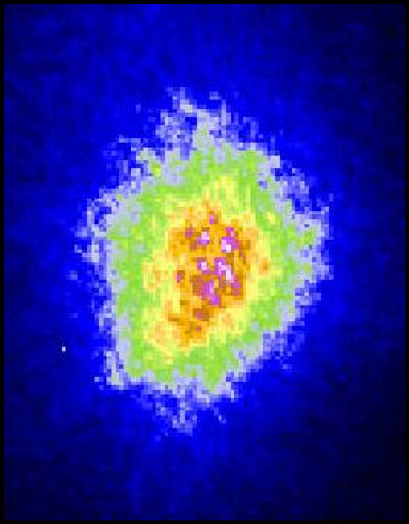


CXD in the fluctuating regime

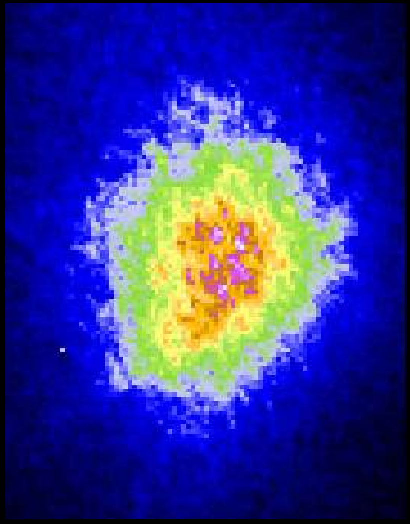
Coherent X-ray diffraction @ ESRF (ID10a+ID1)



400 s average

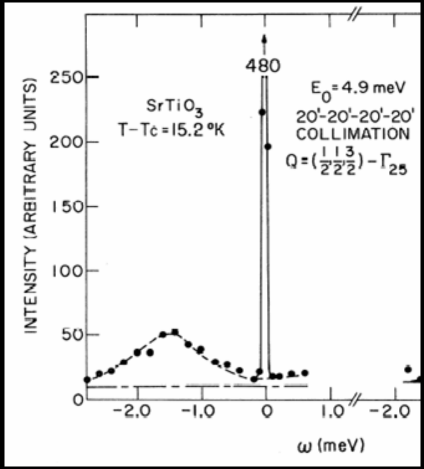


0-200 s



200-400s

S. Ravy, D. Le Bolloc'h, R. Currat
C. Mocuta, A. Fluerasu and B. Dkhil
Phys. Rev. Lett. 98, 105501 (2007)



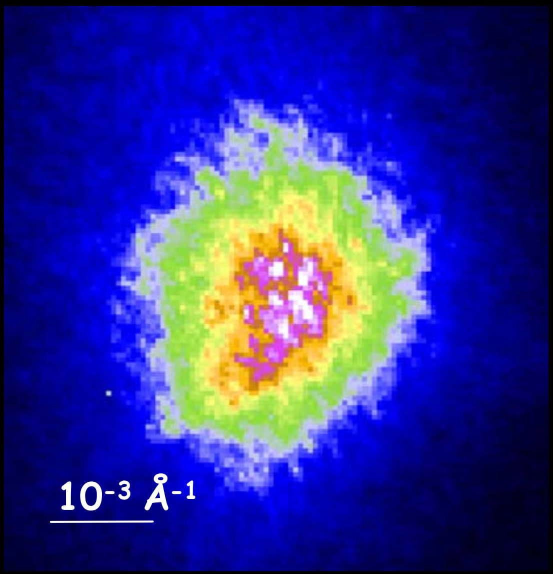
Riste *et al.* SSC. 9, 1455 (1971)

Quasi-static (10 mn)

Related to the "central peak" problem

'Slow' dynamics

With XFEL it should be possible to measure
diffuse scattering above
displacive phase transitions

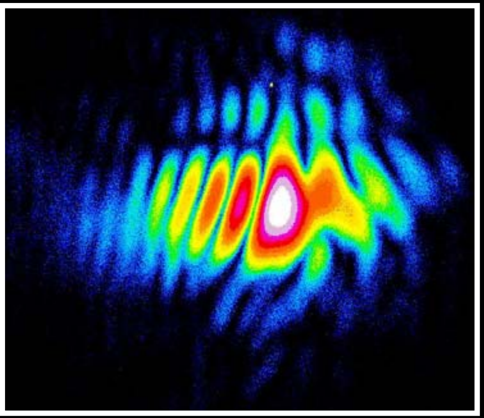


10^{-2} Å^{-1} seems to be the limit for 3rd generation sources.

With 100 fs,
'Slow' dynamics could be probed "central peaks" (CDW, molecular systems)

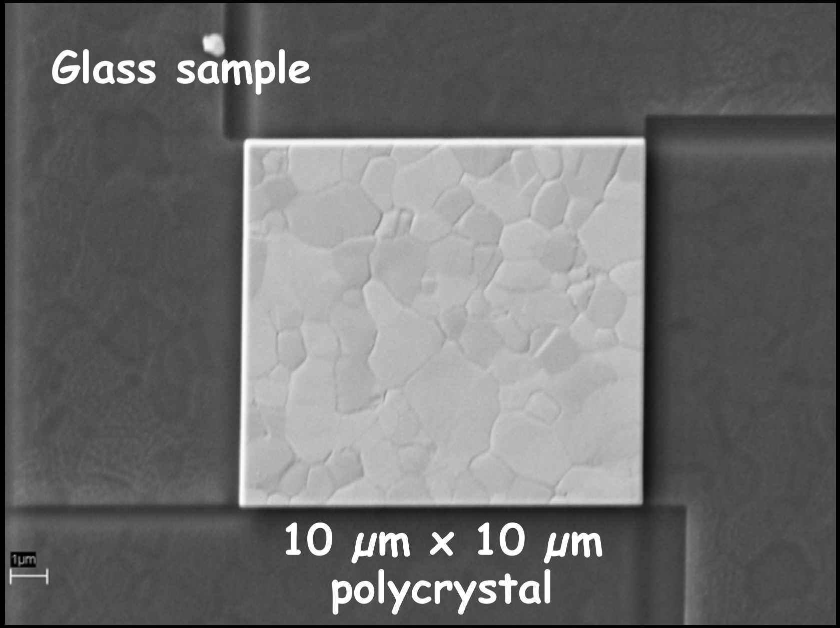
Fluctuations may be measured on micro- or nano-crystals

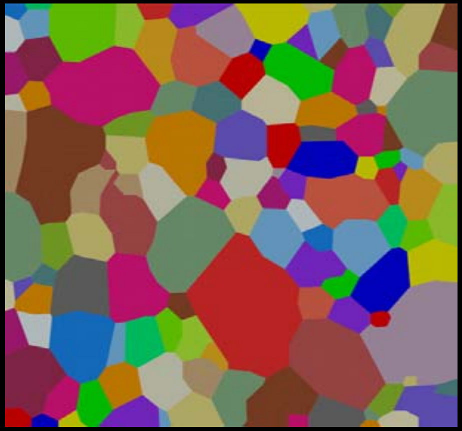
3. Imaging strains



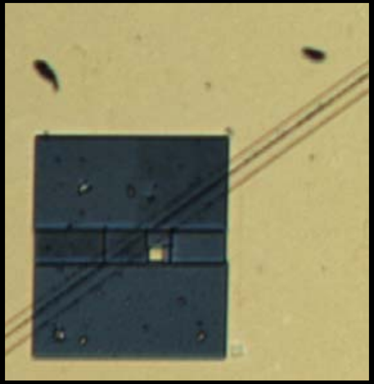
Diffraction by an individual submicronic grain

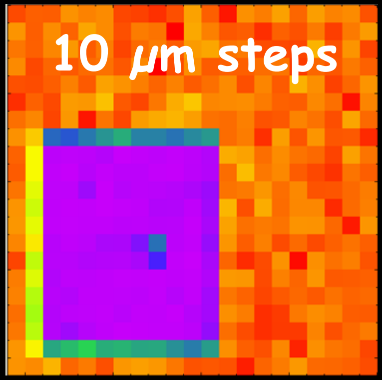
(N. Vaxelaire, S. Labat and O. Thomas, IM2NP, Marseille) :
Goal: Image the strain of a grain in a 375 nm polycrystalline gold film.





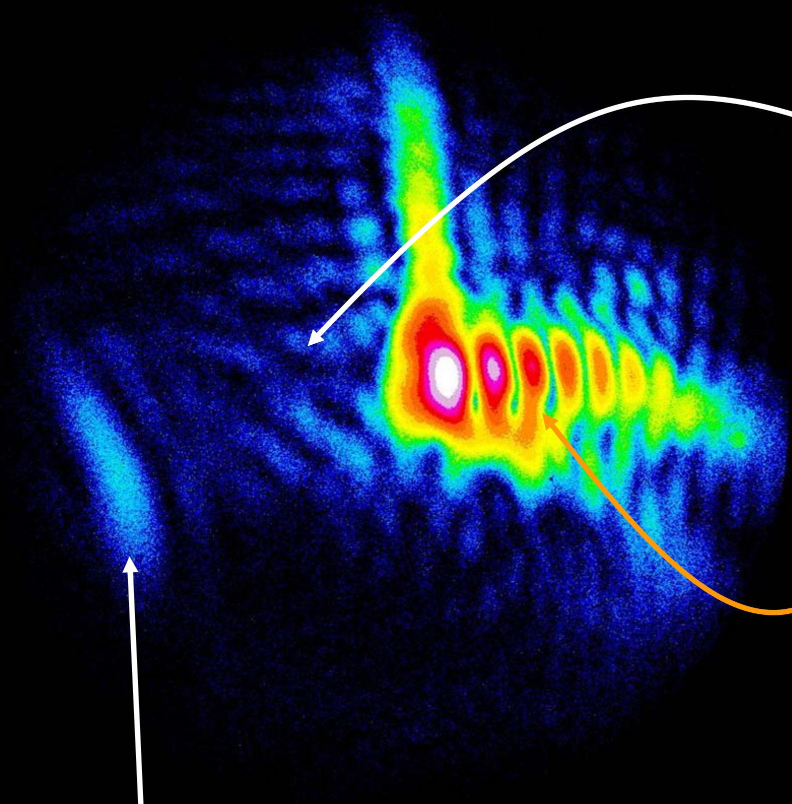
The Au block consists in 133 grains,
mainly (111) with a few (100)
(checked by μ-diffraction at ESRF-BM32)





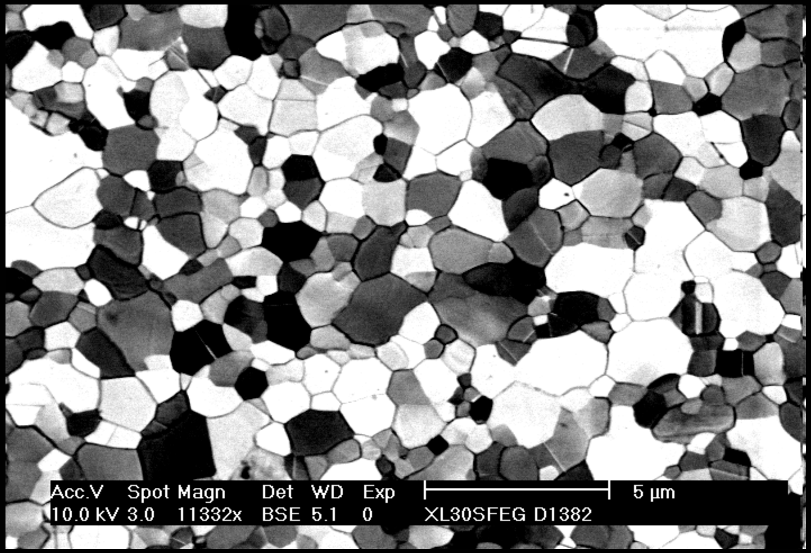
Finding the sample with Au fluoresence

Selecting the grain…



Asymmetry of fringes due to strains

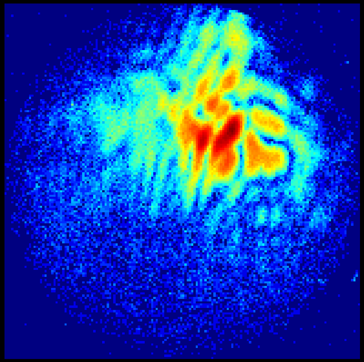
Contamination by a second grain!!

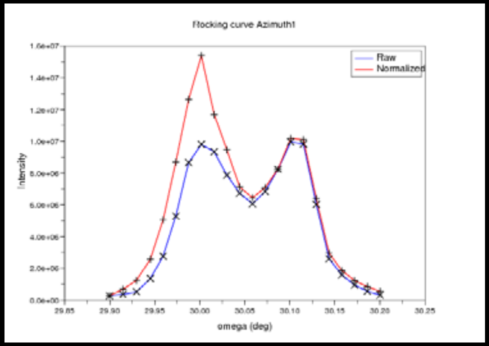


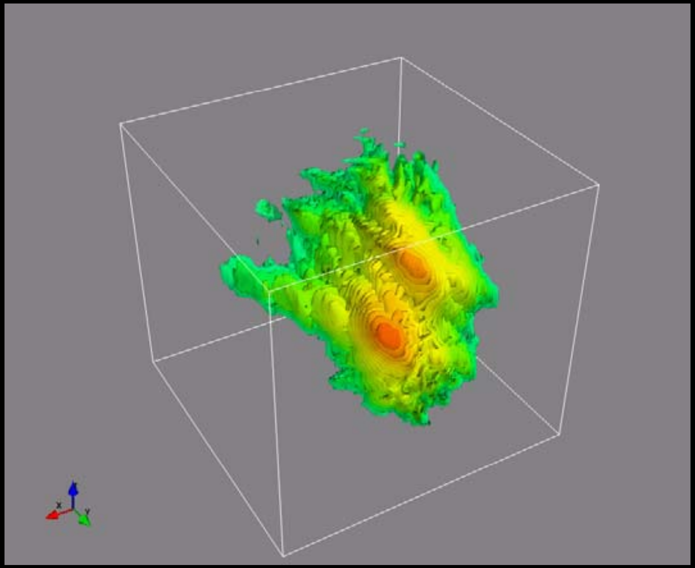
Experimental cross maps

Rocking Curves of the same (111) reflection for different azimuths

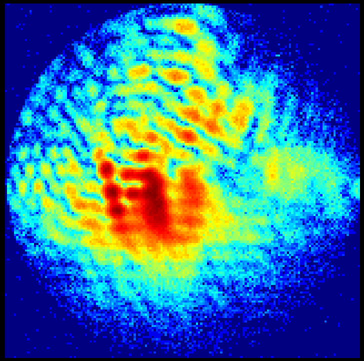
Azimuth phi=90°

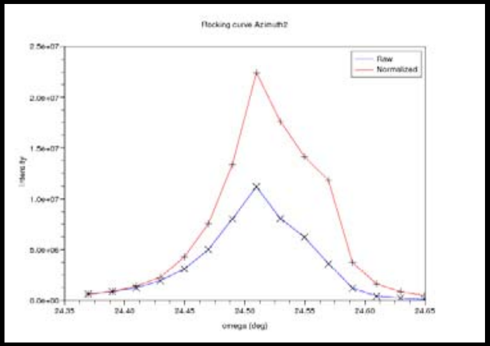


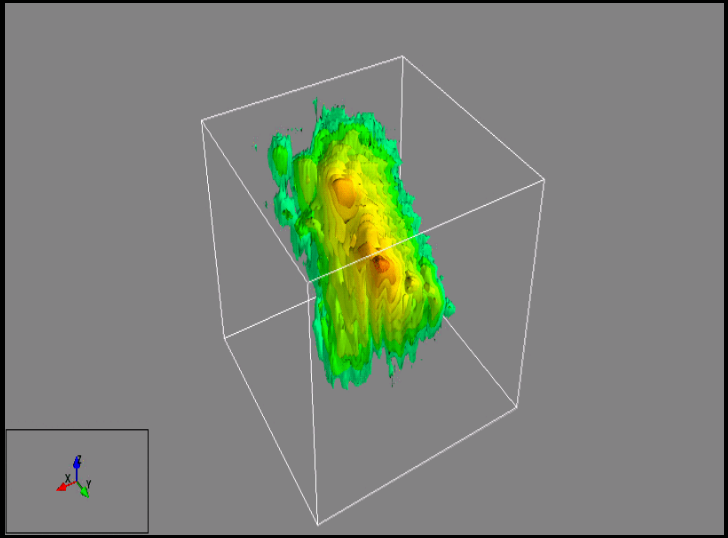




Azimuth phi=0°

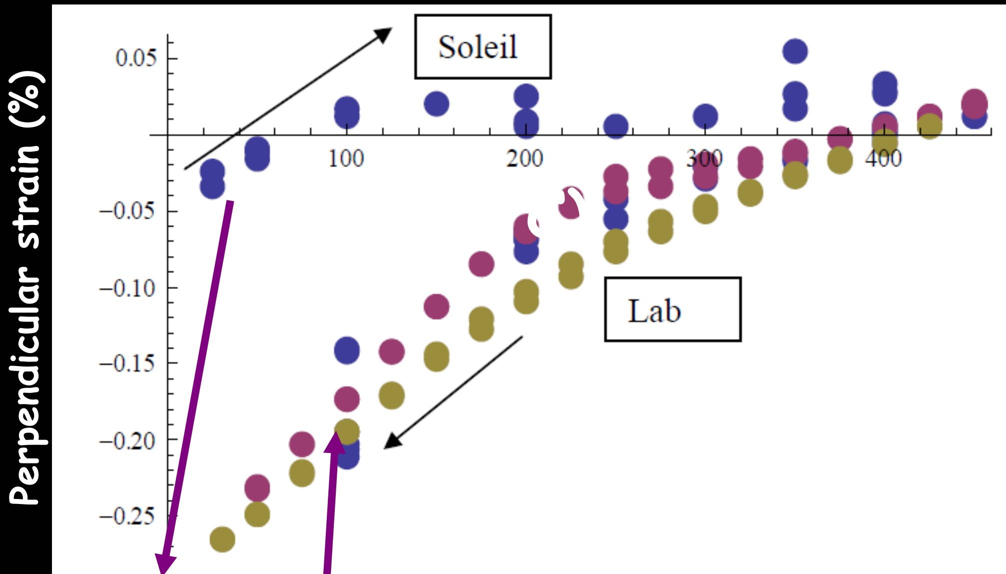






Not possible to reconstruct with current algorithms (N. Vaxelaire, Tomorrow)

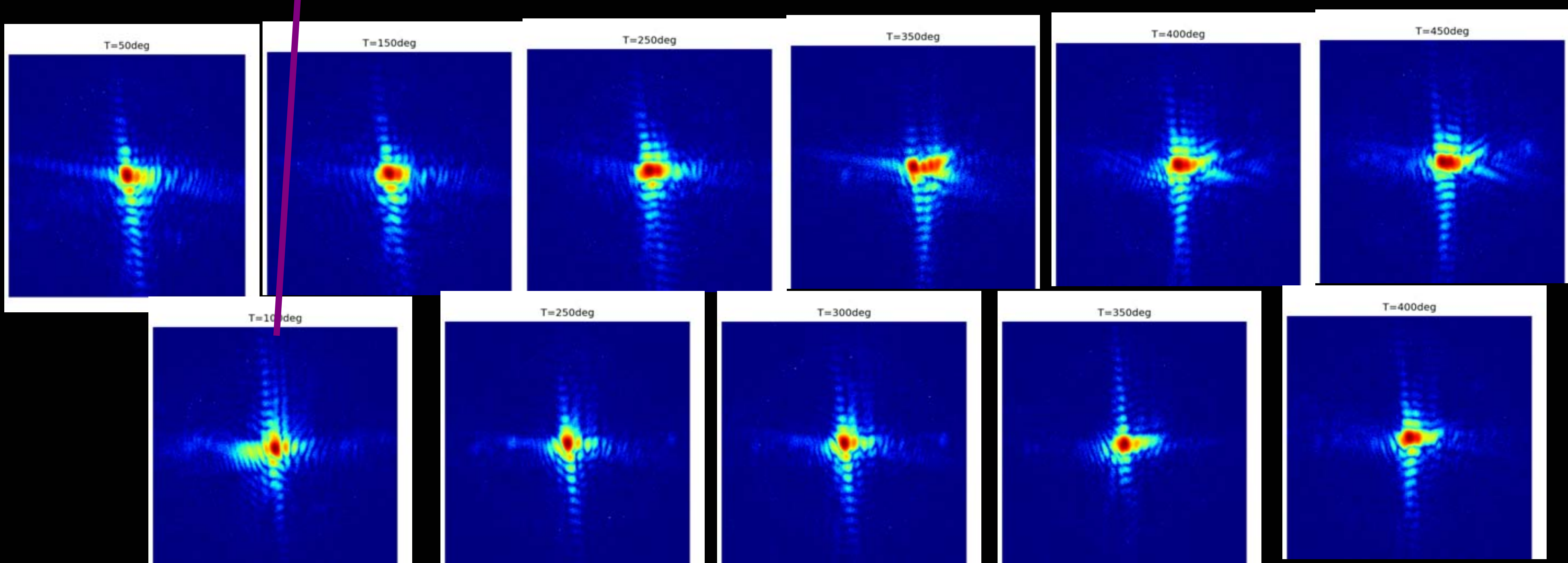
Plastic behaviour of a single grain during a thermic cycle



Thermo-elastic behavior

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

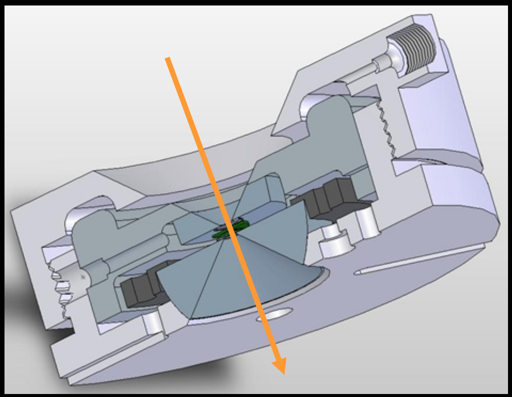
2D slice at the maximum of rocking curve



5. CXD under Pressure:
It's feasible

Coherent diffraction under pressure

D. Le Bolloc'h, J.-P. Itié, A. Polian S. Ravy, High Pressure Research (2009)

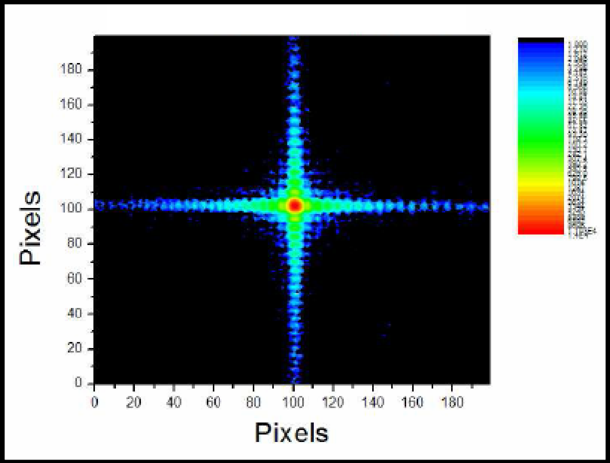


1.2 mm thick diamonds

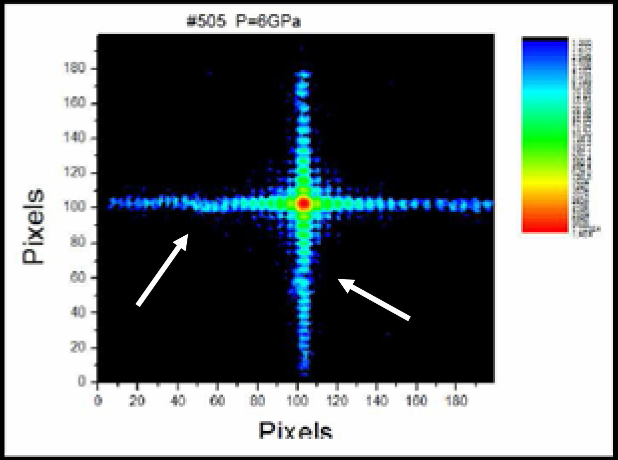
Opening 2 x 40 °

Diffraction through the diamonds

10 keV -> 12 keV



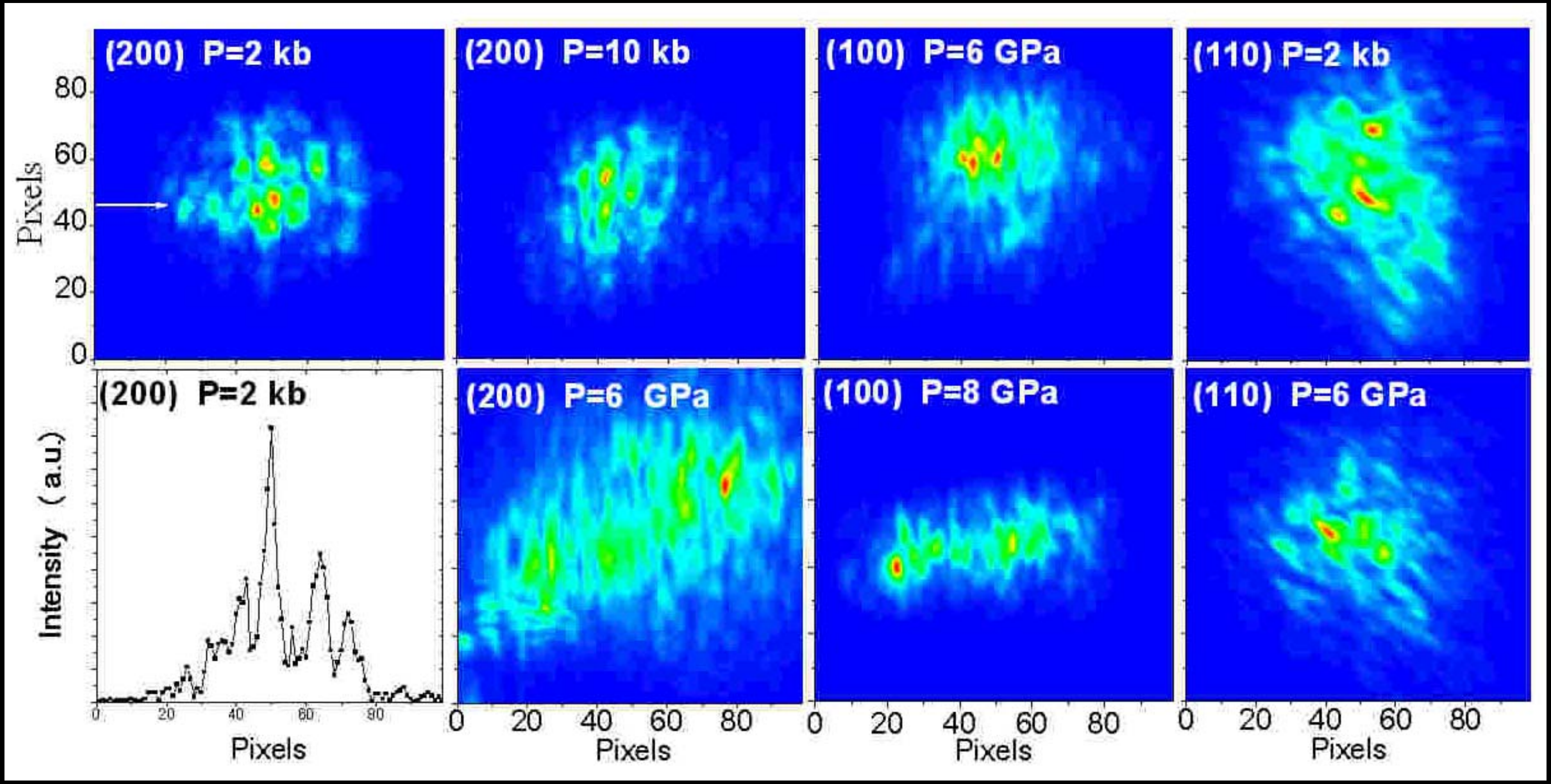
Diffraction by
5 μm x 5 μm slits



Through the cell
at 6 GPa (contrast β > 70%)

Observation of Speckles up to 8 GPa on $SrTiO_3$

D. Le Bolloc'h, J.-P. Itié, A. Polian S. Ravy, High Pressure Research (2009)



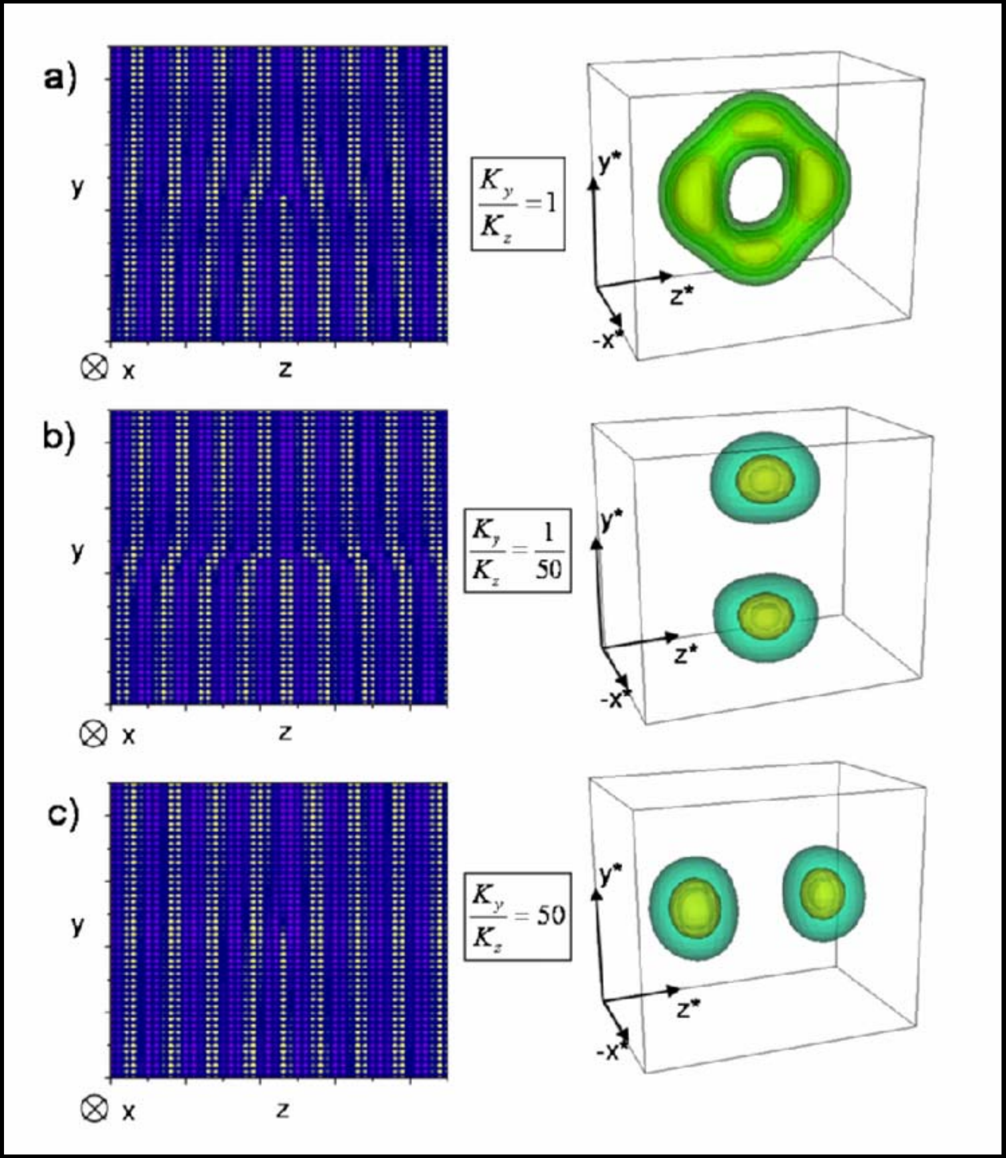
Main problem mosaicity increased under pressure!

-> Small crystals

These experiment would be easy(!) to performed on XFEL

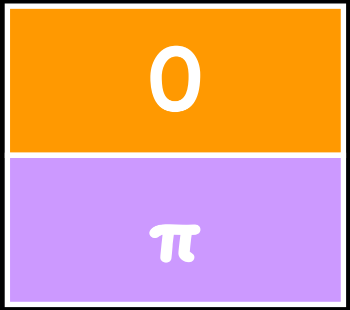
5. Topological defects
Watching dislocations

Case of edge-dislocations (easiest to understand!):



First effect:

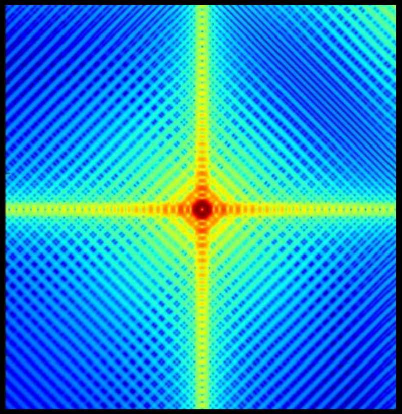
Bragg reflections corresponding to the order (lattice, DW) are zero at the center, because there is a π phase shift



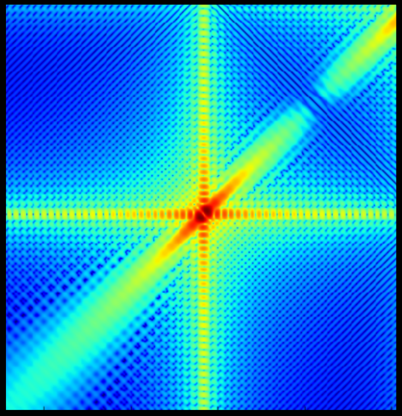
Strongly depends on the force constants K_x, K_y

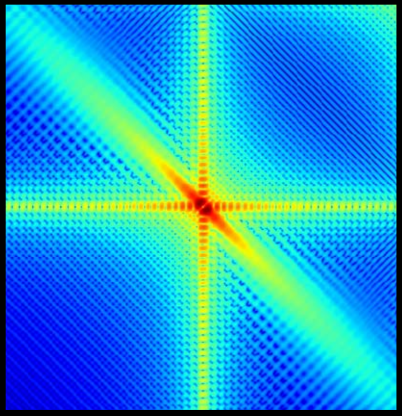
Case of edge-dislocations II

$$\varphi(x',y') = \frac{\pi}{2} * \mathrm{sgn}(x'-x_0') + \tan^{-1}\left(\sqrt{\frac{K_{x'}}{K_{y'}}}\,\frac{y'-y_0'}{x'-x_0'}\right)$$



$$\frac{K_{x'}}{K_{y'}} = 1$$





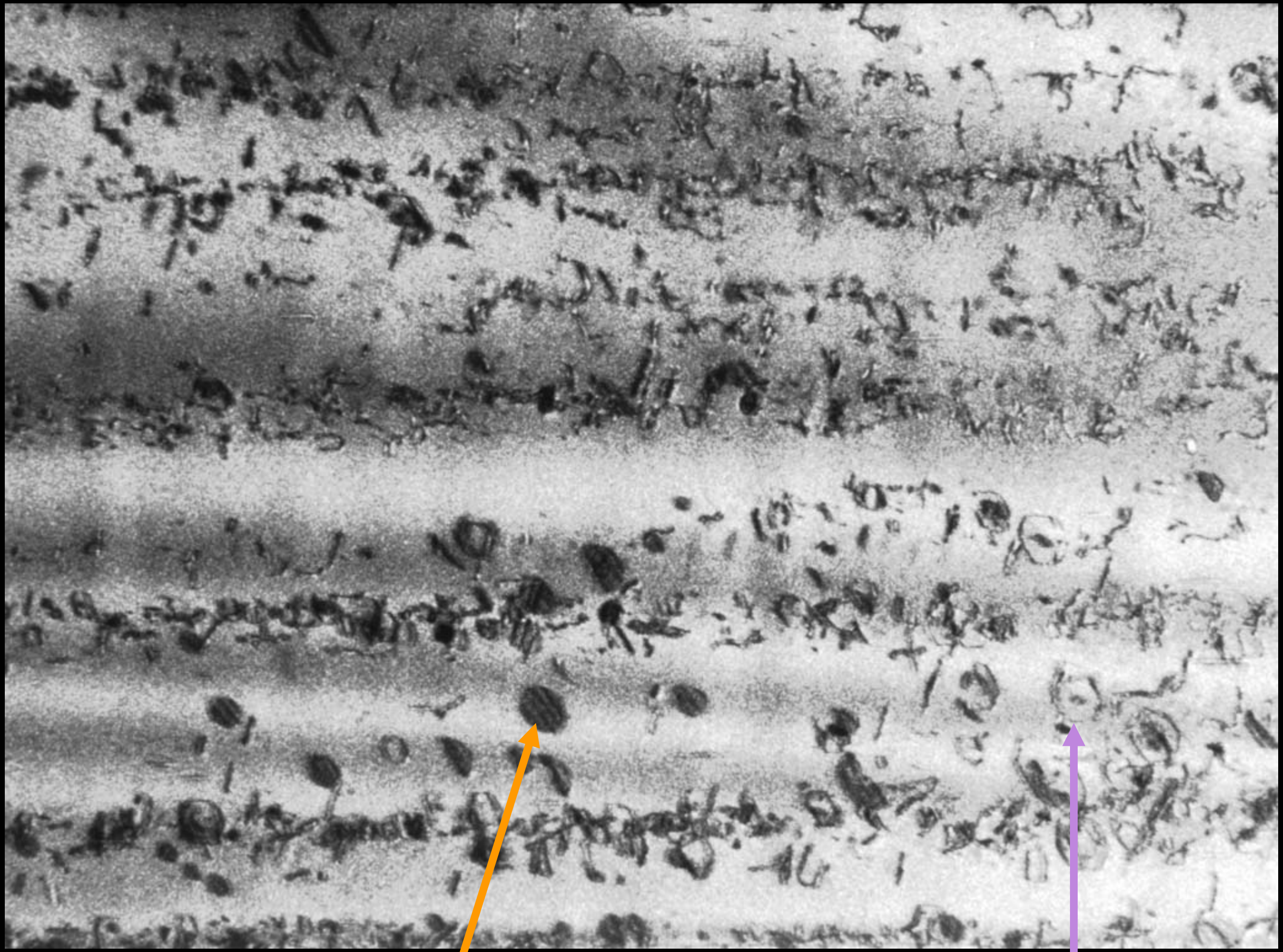
$$\frac{K_{x'}}{K_{y'}} = 100$$

$$\frac{K_{x'}}{K_{y'}} = 0.01$$

Testing these ideas on

Silicon dislocations

Si annealed at 1100 °C (Guy Rolland, CEA)
Transmission topography reveals two types of defects



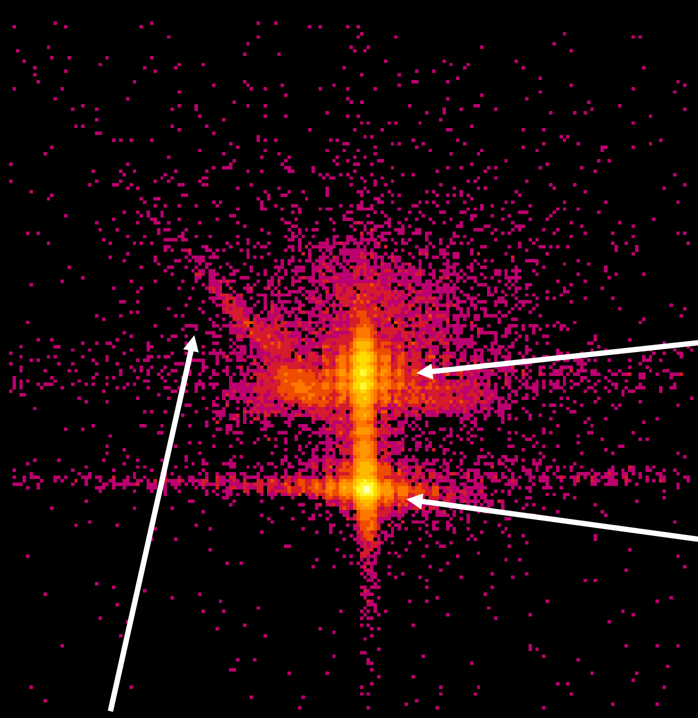
Partial dislocation loops:
Loops + stacking faults
Burger vector = (111)/3

Prismatic loops:
Burger vector = (110)

Coherent diffraction on dislocations in Si : New effects to understand

V. Jacques, D. Le Bolloc'h (LPS, Orsay)

Position scans (3 µm steps) off the (220) Bragg reflection with a 7 µm x 7 µm beam



Bragg tail

Truncature rod

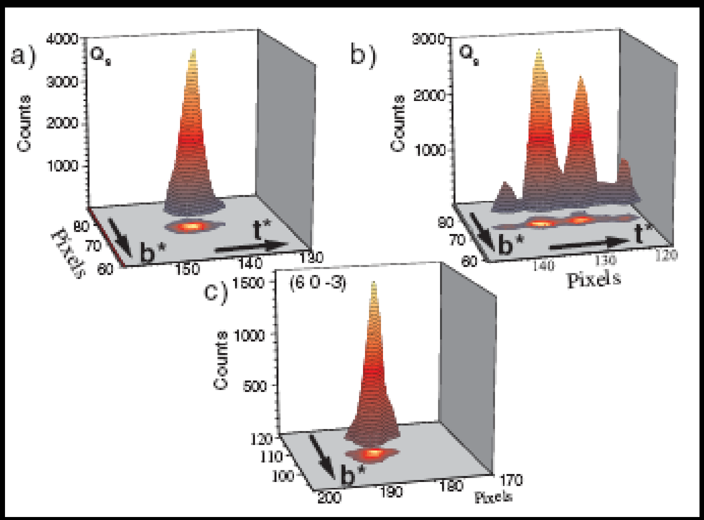
Diffuse streaks

Towards a ptychography of topological defects?

The same with XFEL bunch trains: 10 m/s

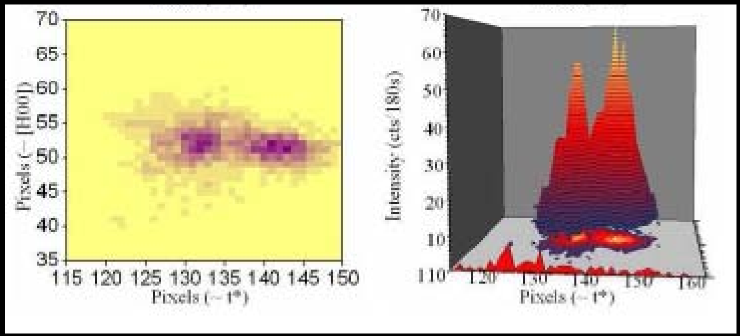
Study of topological defects of complex order parameters in the bulk

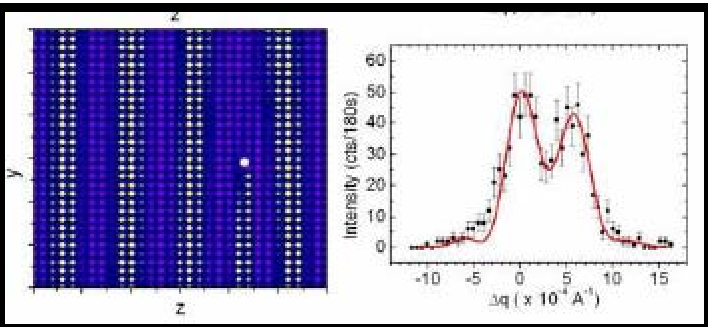
Charge Density Waves dislocation in $KMoO_3$



Le Bolloc'h PRL95, 116401(2005)

Spin Density Wave dislocation in Cr

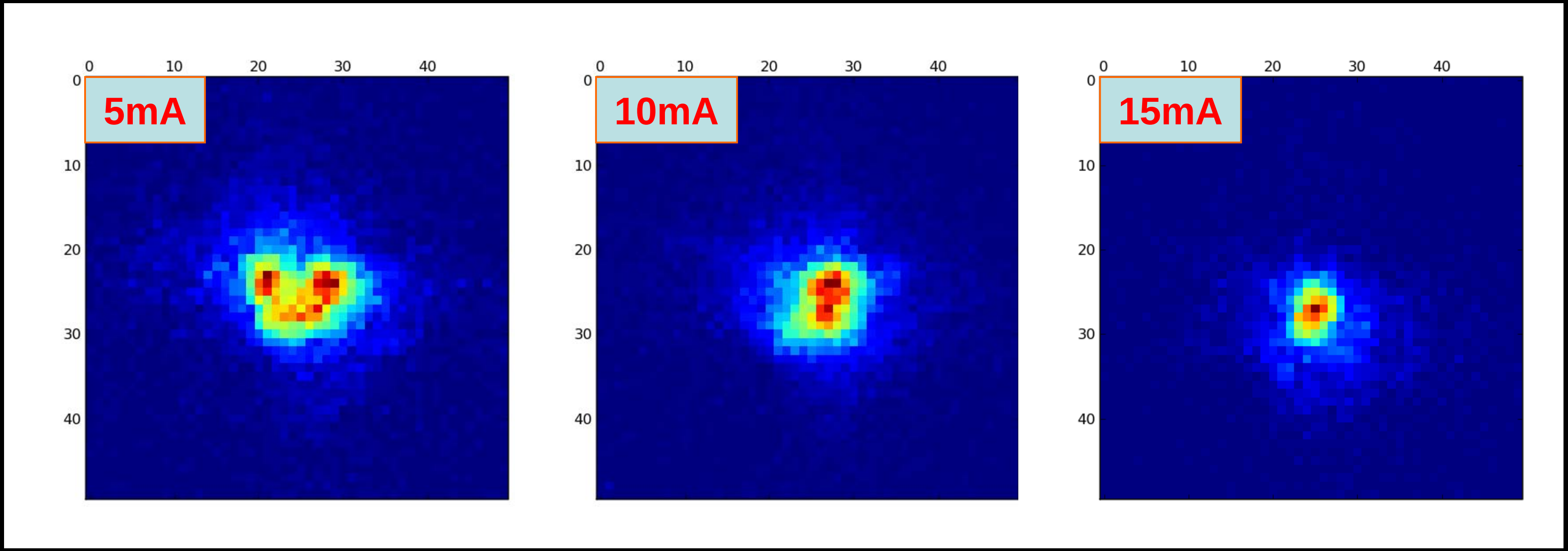




V. Jacques, EPJB70, 317 (2009)

How dislocations emerge, move, deform or disappear?

Ex: Disappearance/movement of CDW defects upon sliding
$K_{0.3}MoO_3$ CDW satellite reflection (CRISTAL, 7 keV):
Motional ordering

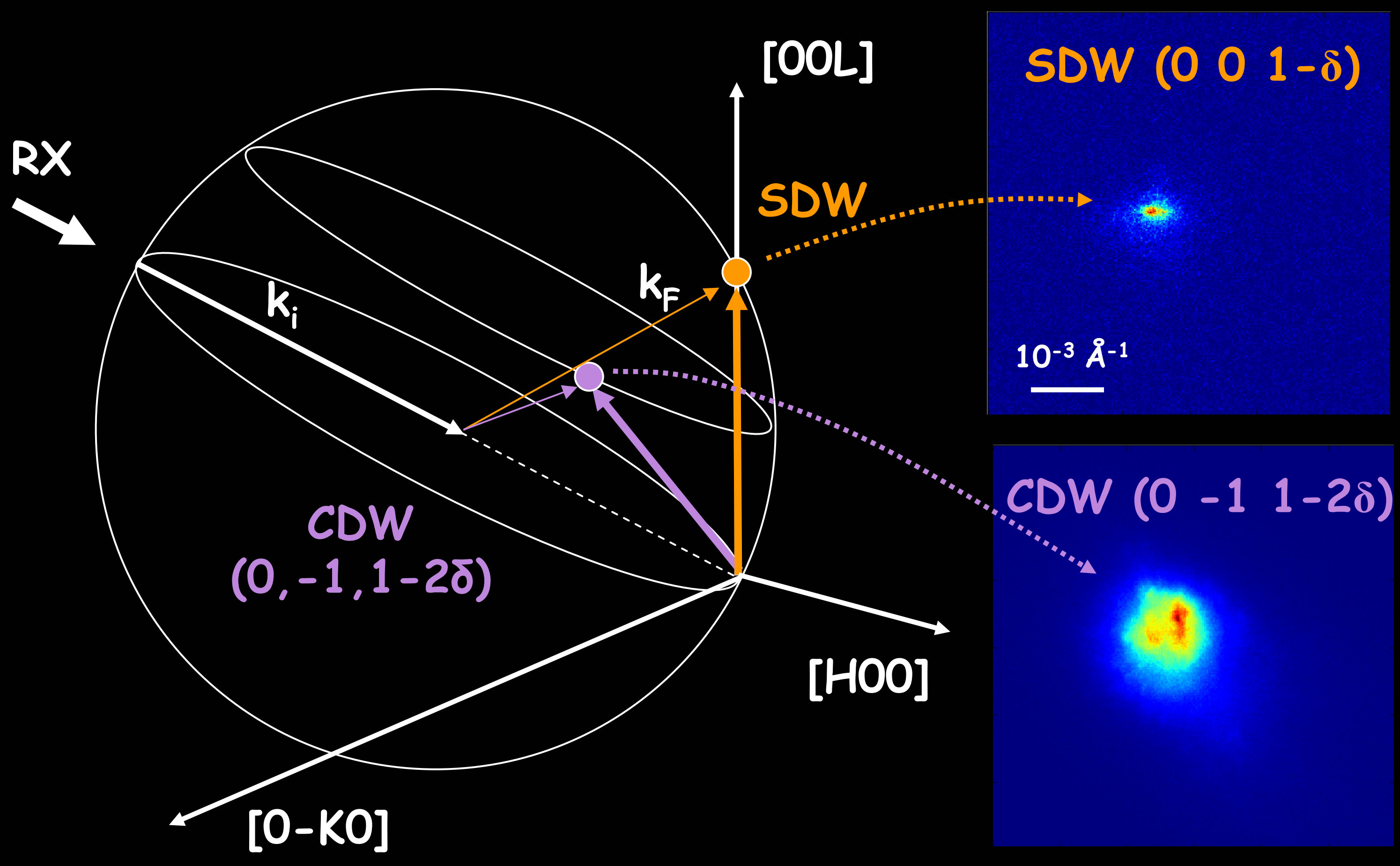


Vincent Jacques PhD Thesis.

Pump-probe!!!

Back to diffractometer:

"Double diffraction" geometry: two informations in one shot
Example SDW/CDW in Chromium (Vincent Jacques Thesis))



5. Soft surfaces:

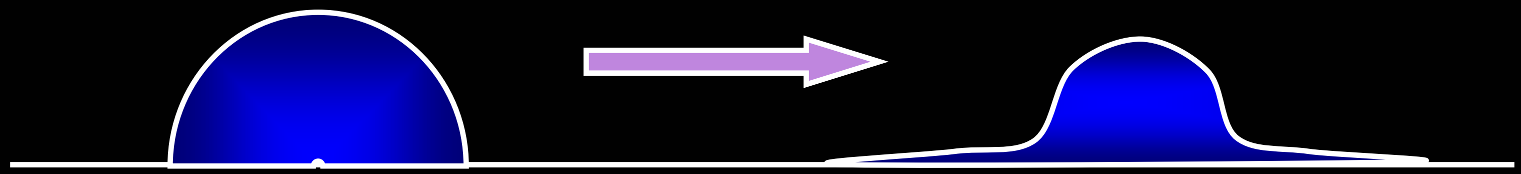
Imaging a spreading droplet

Grazing incidence CXD of a micro-droplet of PDMS

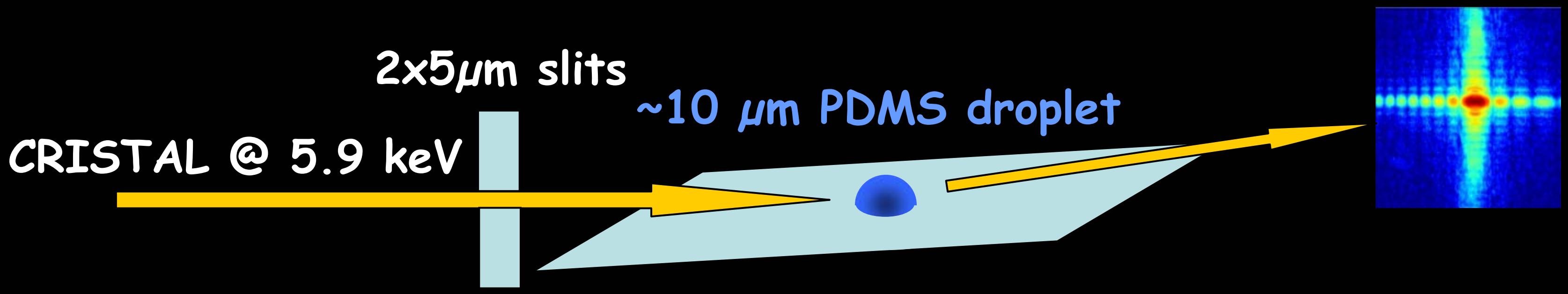
(Ph. Fontaine (SOLEIL), J. Daillant (CEA-Saclay))

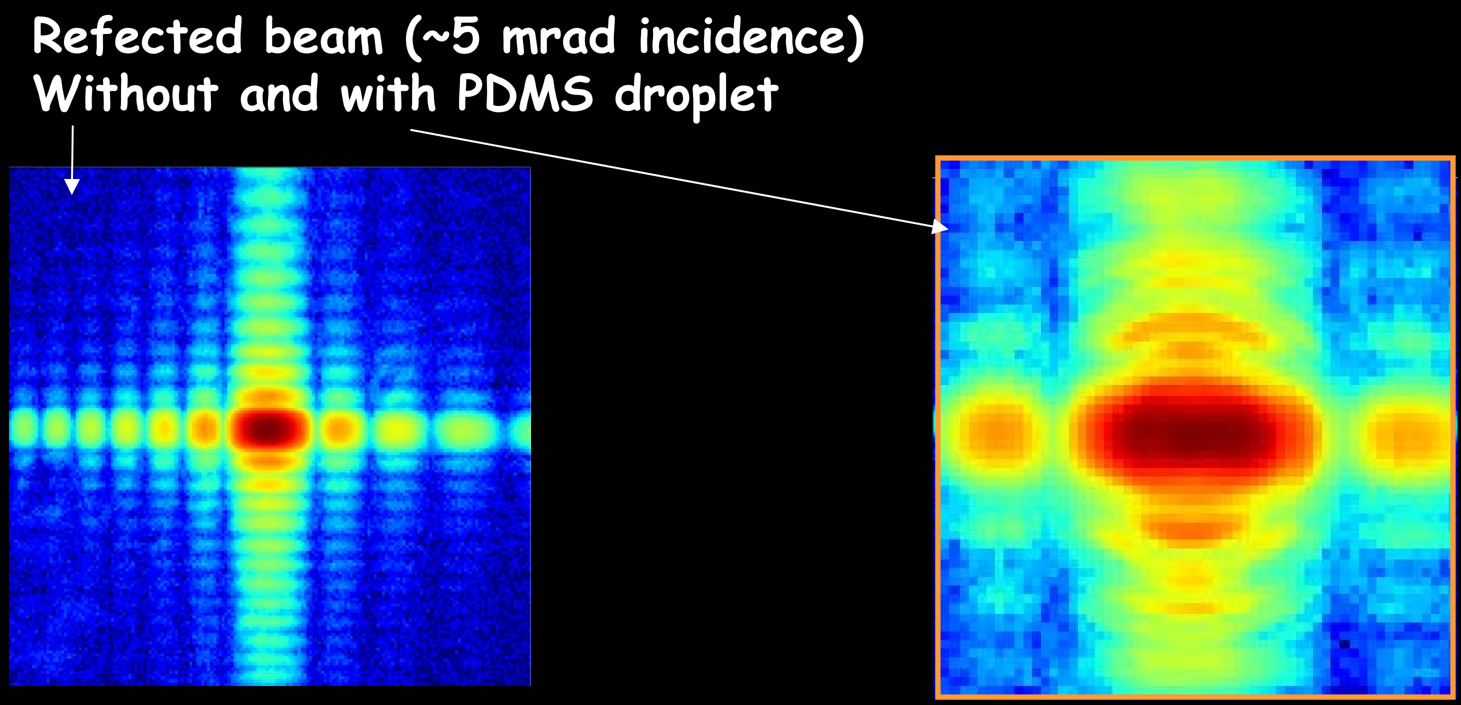
Soft surface are difficult to image
by methods like AFM or optical microscopy.

Use of CXDI to
reconstruct the shape of a
PDMS droplet
while it is spreading

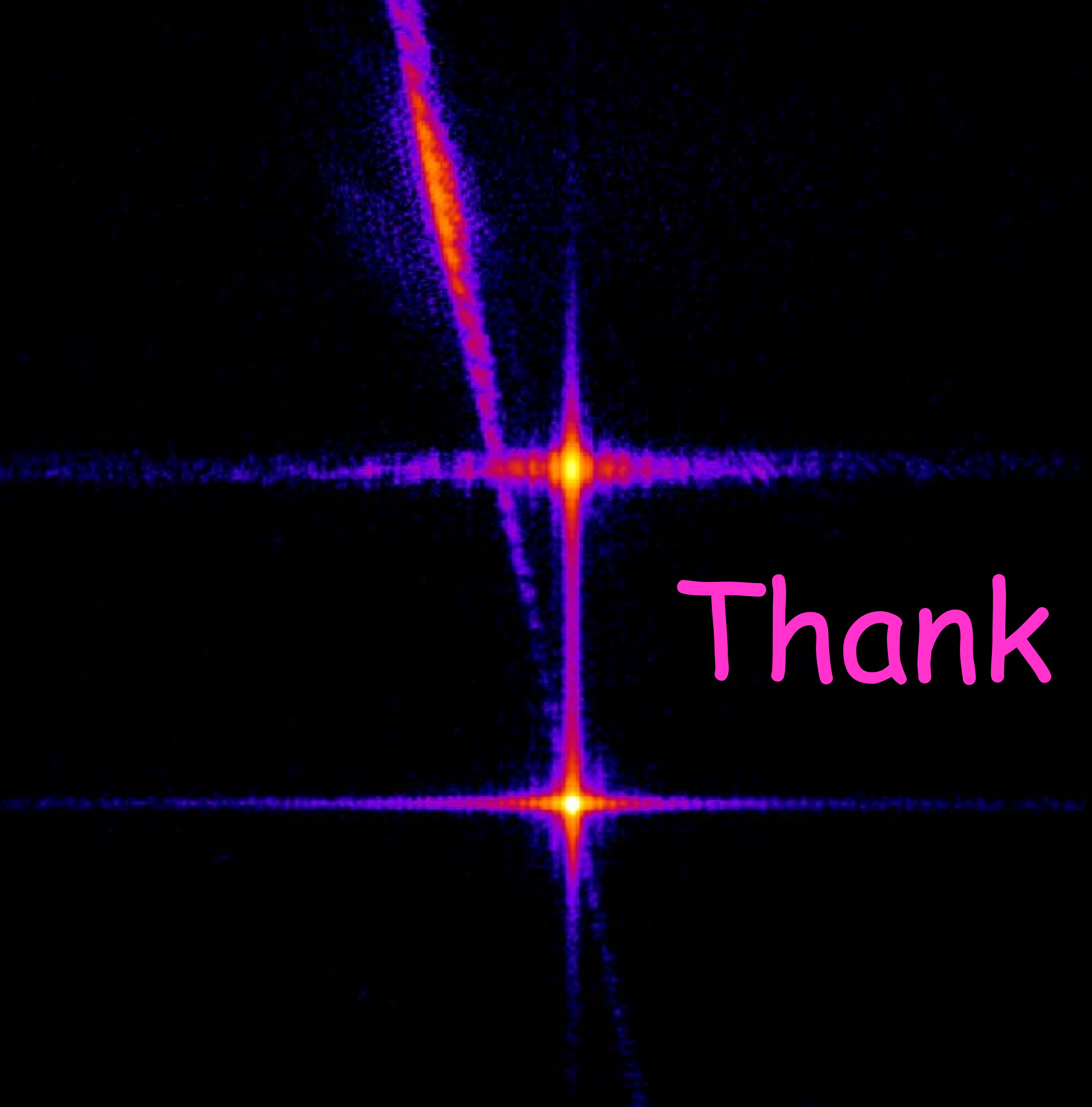


Grazing incidence coherent diffraction of a micro-droplet of PDMS





Feasible with 3rd generation source with viscous fluids (PDMS), and liquid with XFELs



Thank you