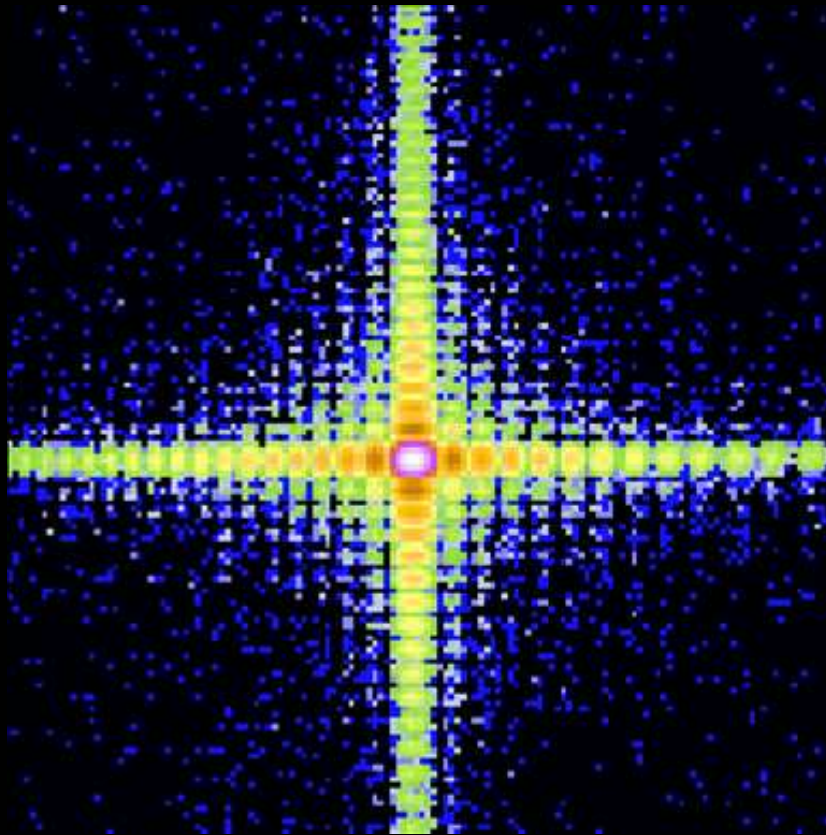


# Using coherence in diffraction



Sylvain RAVY  
ligne CRISTAL  
Synchrotron-SOLEIL

CRISTAL Beamline  
Vincent Jacques  
Frédéric Picca  
David Le Bolloc'h (LPS Orsay)  
Stéphane Labat (Marseille)  
Mourad Idir  
Detector group

For scattering/diffraction experiments in the 5-10 keV range  
**two** coherence lengths

**Transverse coherence length** (due to propagation is 3rd gen. sources)

$$L_T = \lambda \frac{R}{S}$$

U20 :  $S_H = 910 \mu\text{m}$ ,  $S_V = 19 \mu\text{m}$   
CRISTAL :  $R = 35 \text{ m et } 1 \text{ \AA}$

$L_{TH} = 4 \mu\text{m et } L_{TV} = 185 \mu\text{m}$

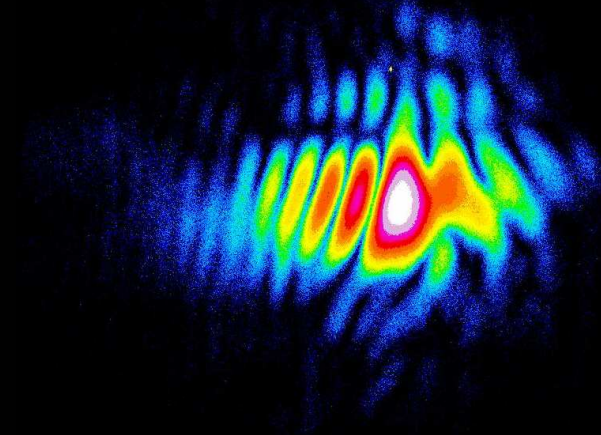
**Longitudinal coherence length** (spectral purity: monochromators)

$$L_L = \frac{\lambda^2}{2\Delta\lambda}$$

Si(111) mono:  $R \sim 10^{-4}$

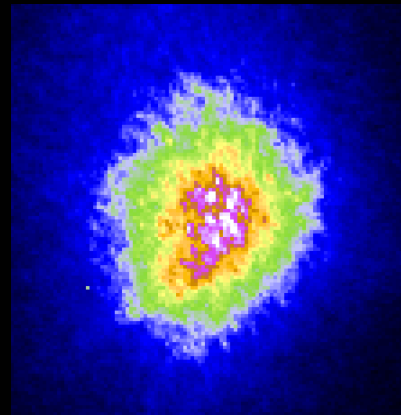
$L_L \sim 1 \mu\text{m}$

Using coherence allows one to study  
**interference fringes,**



**Small objects  
Nano-crystals**

or **speckle patterns,**



**Disordered systems**

which are **smoothed** otherwise

To use **coherent diffraction** methods we need **far field regime**

Fresnel-----Fraunhofer

$$\lambda = 0.1 \text{ nm}$$

$$ff \cong 2 \frac{a^2}{\lambda}$$

$$a = 1 \mu\text{m}$$

$$ff = 2 \text{ cm}$$

$$a = 100 \mu\text{m}$$

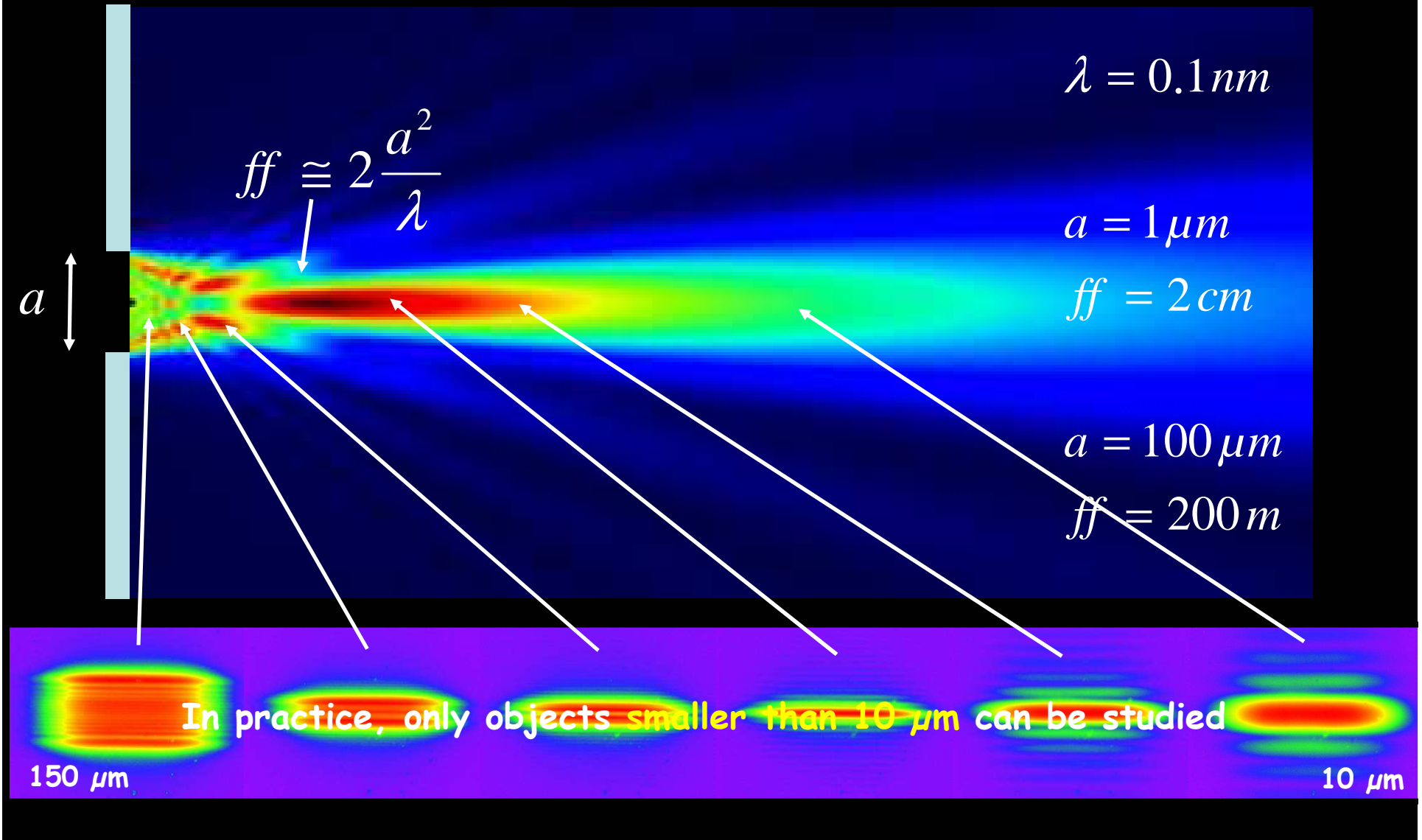
$$ff = 200 \text{ m}$$

$a$

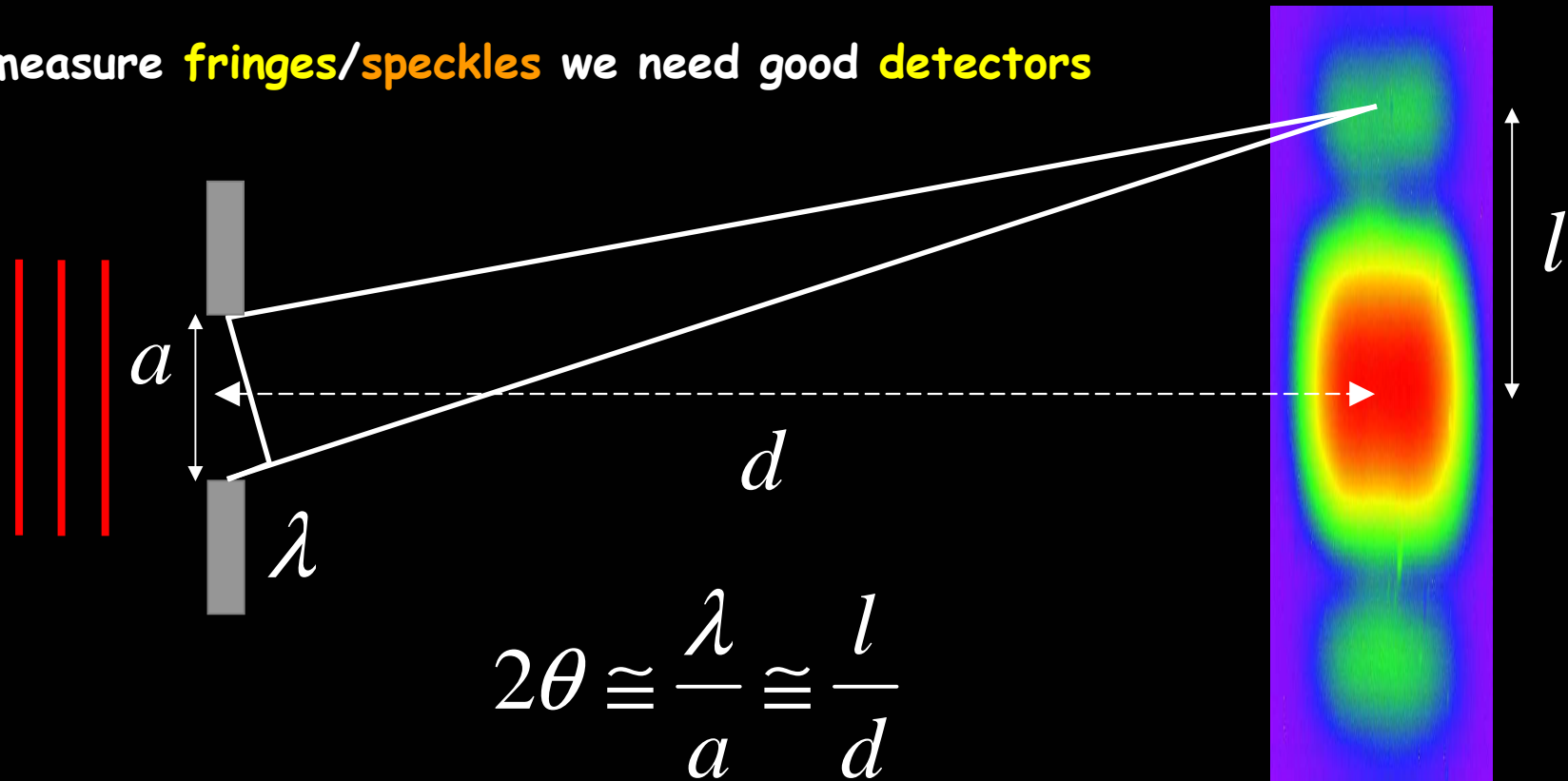
In practice, only objects **smaller than 10  $\mu\text{m}$**  can be studied

150  $\mu\text{m}$

10  $\mu\text{m}$



To measure **fringes/speckles** we need good **detectors**



$$2\theta \cong \frac{\lambda}{a} \cong \frac{l}{d}$$

With  $\lambda=1 \text{ \AA}$  and  $d=1\text{m}$ , for  $a = 10 \mu\text{m}$  fringes spacing is  $l = 10 \mu\text{m}$   
Pixel size has to be small  
**CCD cameras are needed** (ex : ANDOR  $13 \mu\text{m}$ )

For smaller (nano-) objects  $a < 1 \mu\text{m}$  ,  $l > 100 \mu\text{m}$   
New **pixel detectors** can be used (ex : XPAD pixel size is  $130 \mu\text{m}$ )

CCD cameras are **commonly** used for coherent diffraction:



- Small pixel size
  - Low noise (with appropriate data analysis)
- but
- Very low dynamics (long acquisition time)
  - Too small and too fragile

New **hybrid pixel detectors** (XPAD, Pilatus) are much better:



- Excellent dynamics
- Photon counting

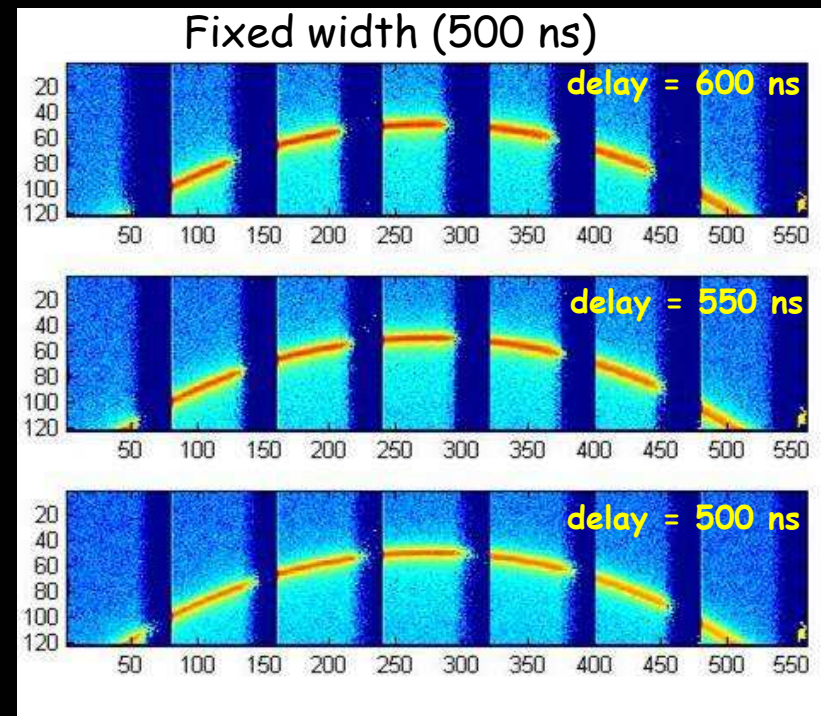
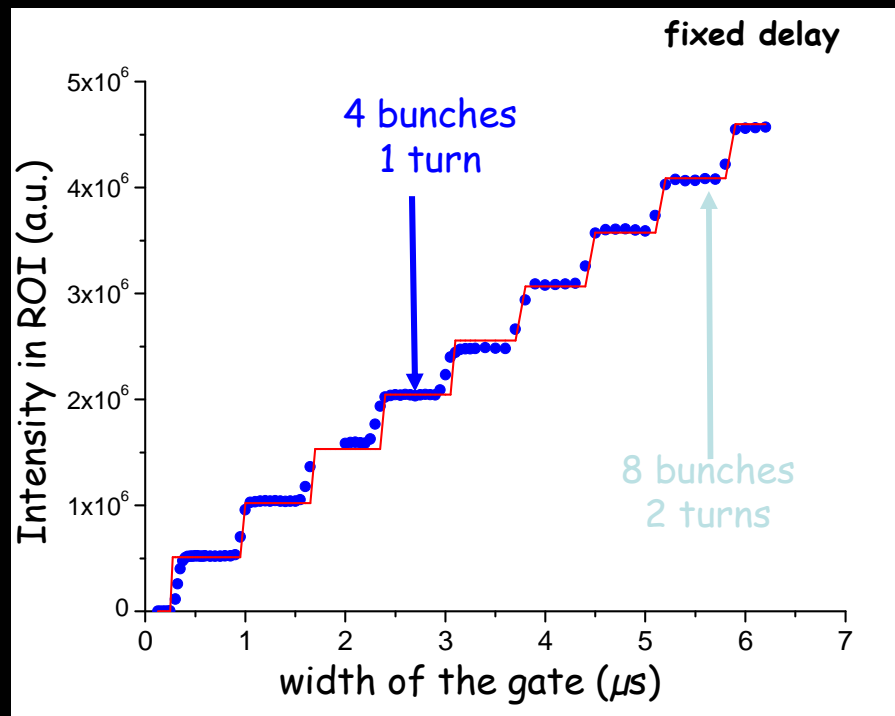
but

- Pixel size (a bit) **too large** (130  $\mu\text{m}$ )

It can perform time-resolved study...

XPAD can be 'gated' to detect one bunch:

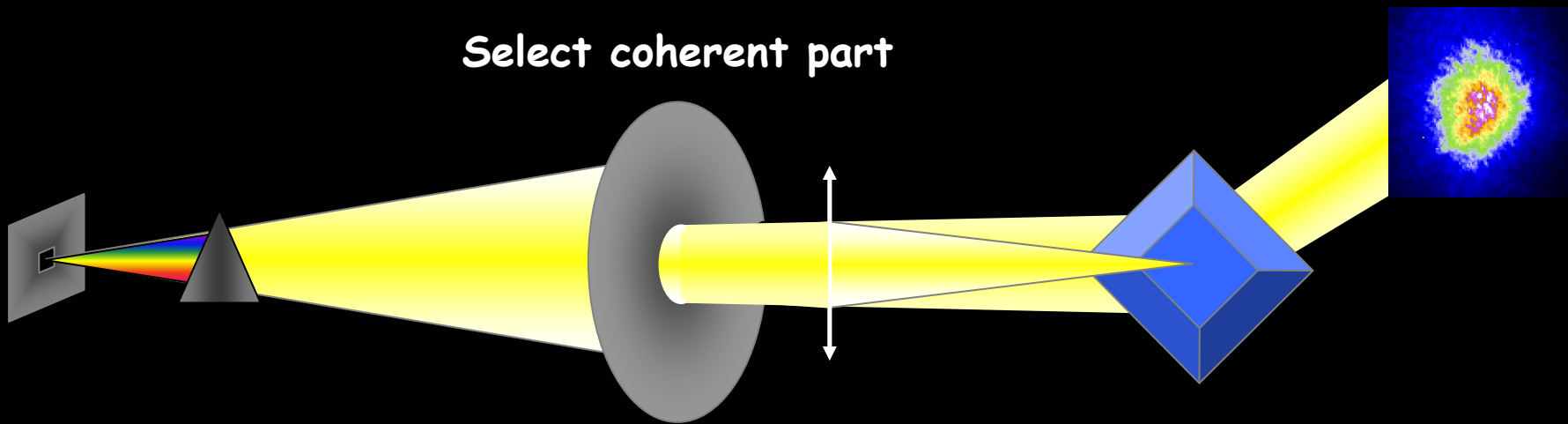
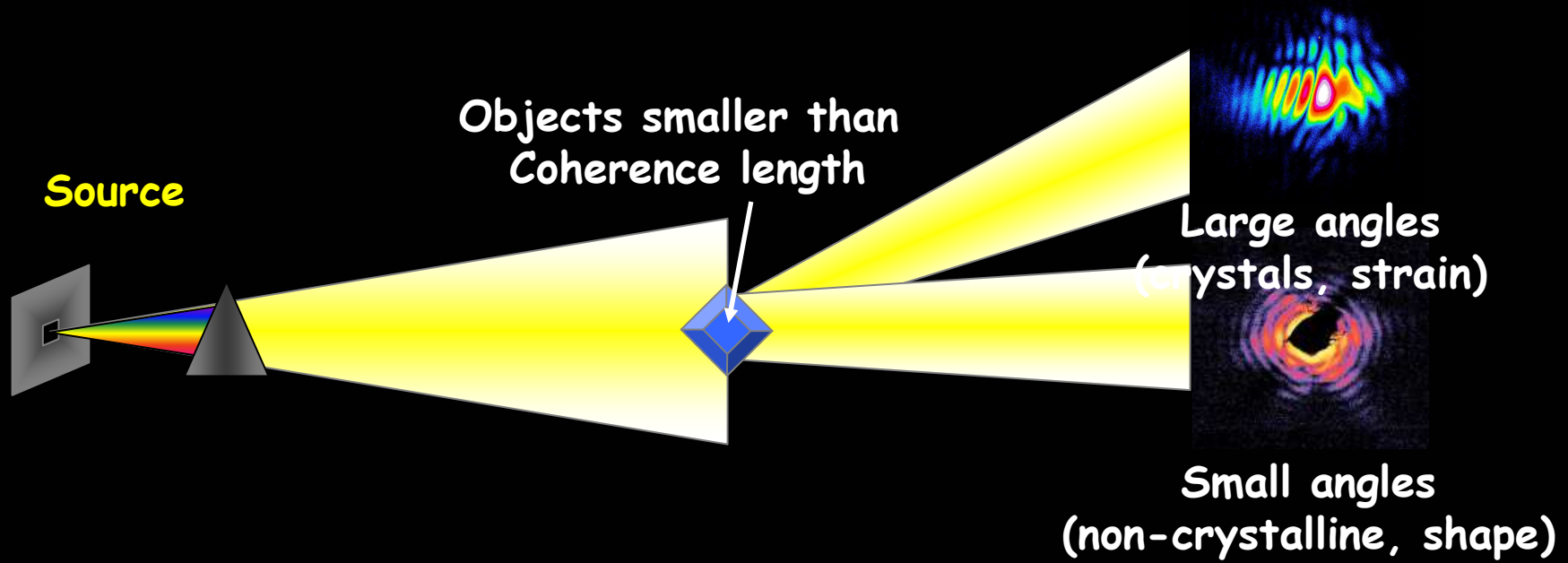
**It works** at ESRF 4-bunches mode (700 ns between bunches) and soon in "any" few bunch modes (150 ns) for pump-probe experiments



Could be used for **pump-probe experiments** on 4th generation sources

Possibility to use a buffer and obtain **two images** separated by **150 ns**.

# Different ways to perform coherent diffraction



Diffracted amplitude :

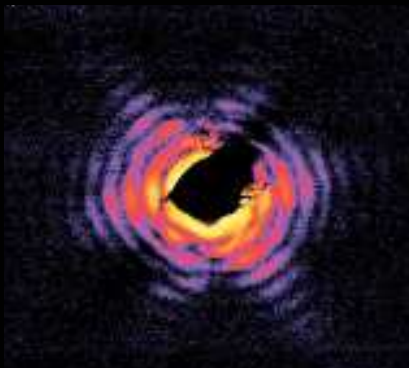
$$A(\mathbf{q}) = \sum_j f_j e^{i\mathbf{q}\cdot\mathbf{r}_j}$$

Same at small angles and large angles  
EXCEPT if strains  $u_j$

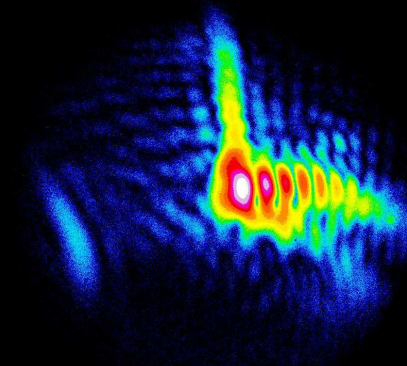
$$A(\mathbf{q}) = \sum_j f_j e^{i\mathbf{q}\cdot\mathbf{r}_j} e^{i\mathbf{q}\cdot\mathbf{u}_j}$$

Negligible at small angle  
because  $u_j \ll r_j$

Strong effect  
on Bragg reflections  
Asymmetry



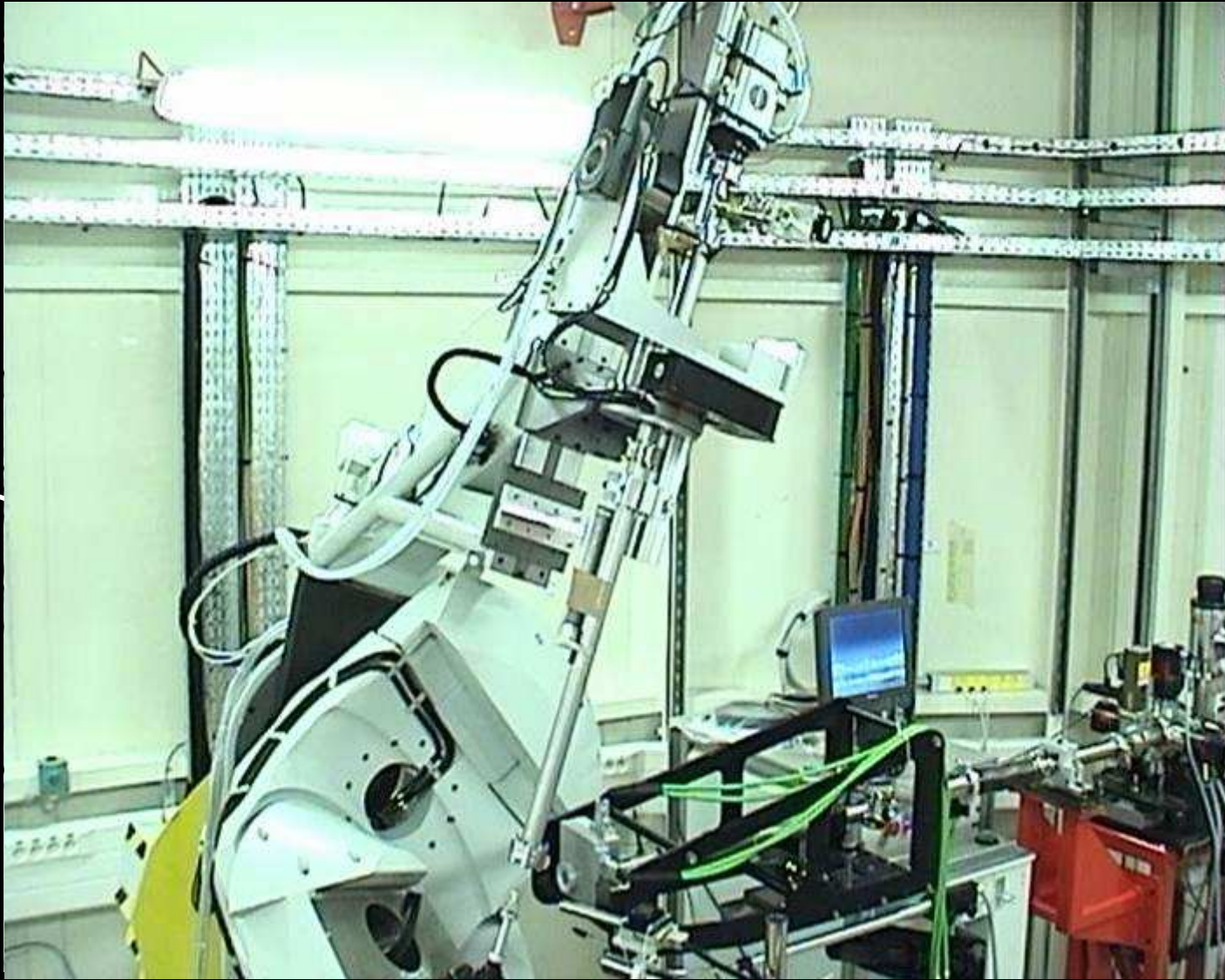
Reconstruction, Shape  
Resolution is  $(\Delta q)^{-1} \sim 10\text{nm}$



Projection of  $u$  along  $q$

6-circles

CCD  
Camera



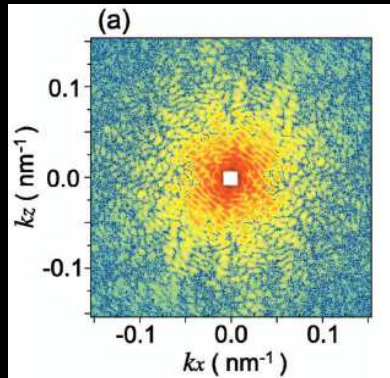
Slits  $\mu\text{m}$   
10 cm

Vacuum fly-path tube 5.2 keV

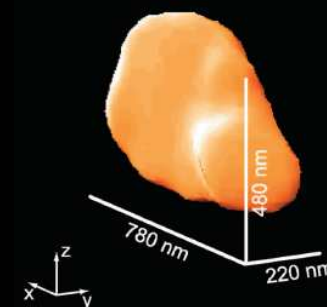
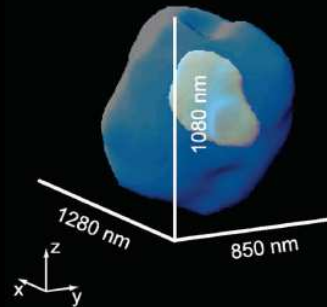
Very few examples of successful reconstructions: it's the beginning...  
Small angles :

**Al<sub>2</sub>CuMg precipitate in Al  $\mu$ -grain.**

Takahashi et al. J.Appl.Phys. 90, 184105(2007)

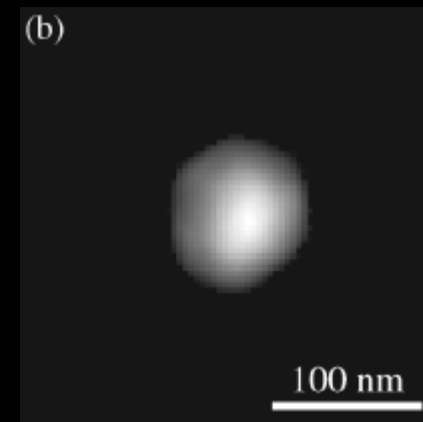
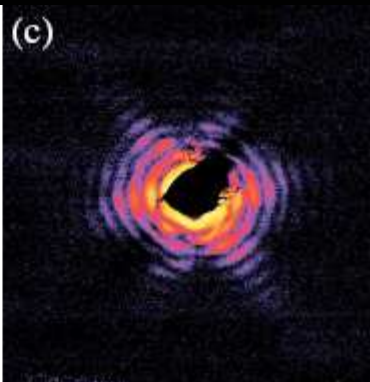
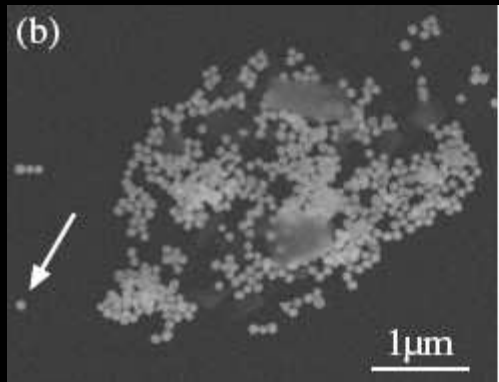


70 frames  
2h/frame  
CCD



**Imaging with nano-focused illumination**

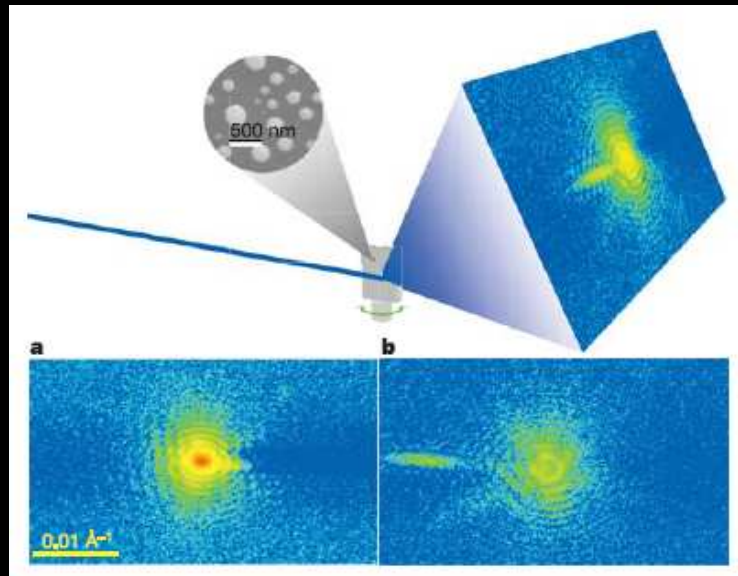
Schroer et al. PRL 101, 090801 (2008)



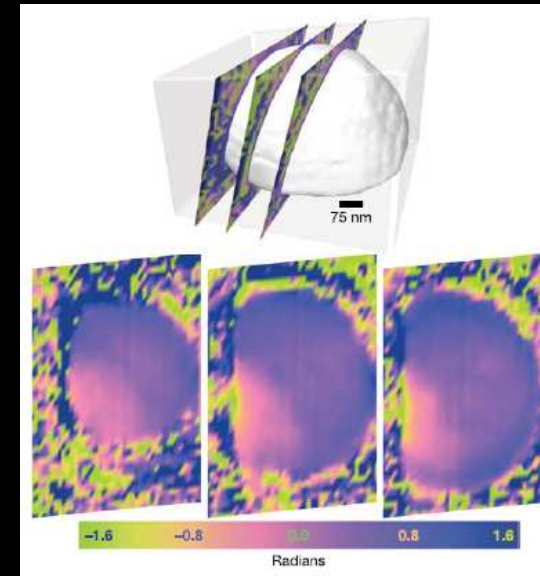
ESRF ID13, 100nm $\times$ 100nm beam @15keV (10 mn)

# Bragg diffraction

## Strain in a Pb 750 nm droplet M. Pfeifer *et al.* Nature 442, 63(2006)

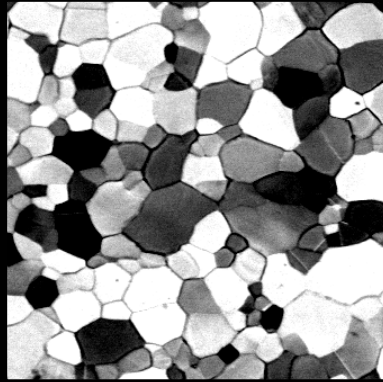


3 hours experiment, 50 frames  
Asymmetric Bragg spots



Mapping of the  $q.u$  phase

Study of **strain** in Au thin films (S. Labat, N. Vaxelaire, IM2NP Marseille)  
**CRISTAL** beamline, SOLEIL

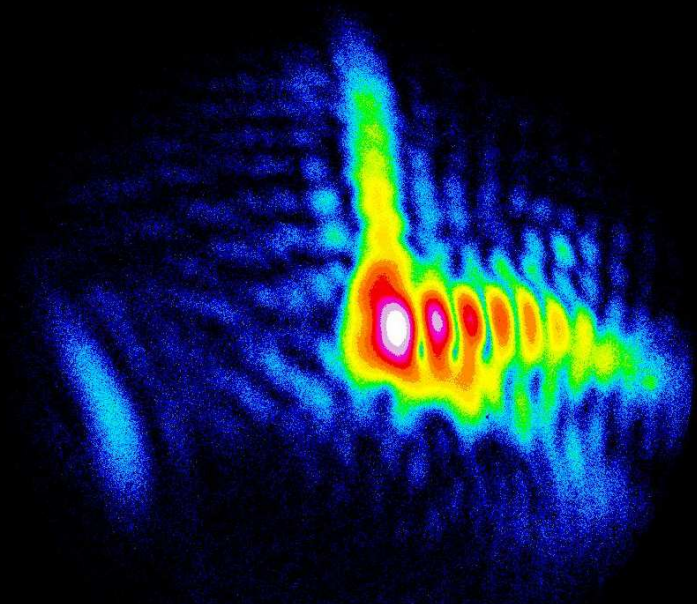


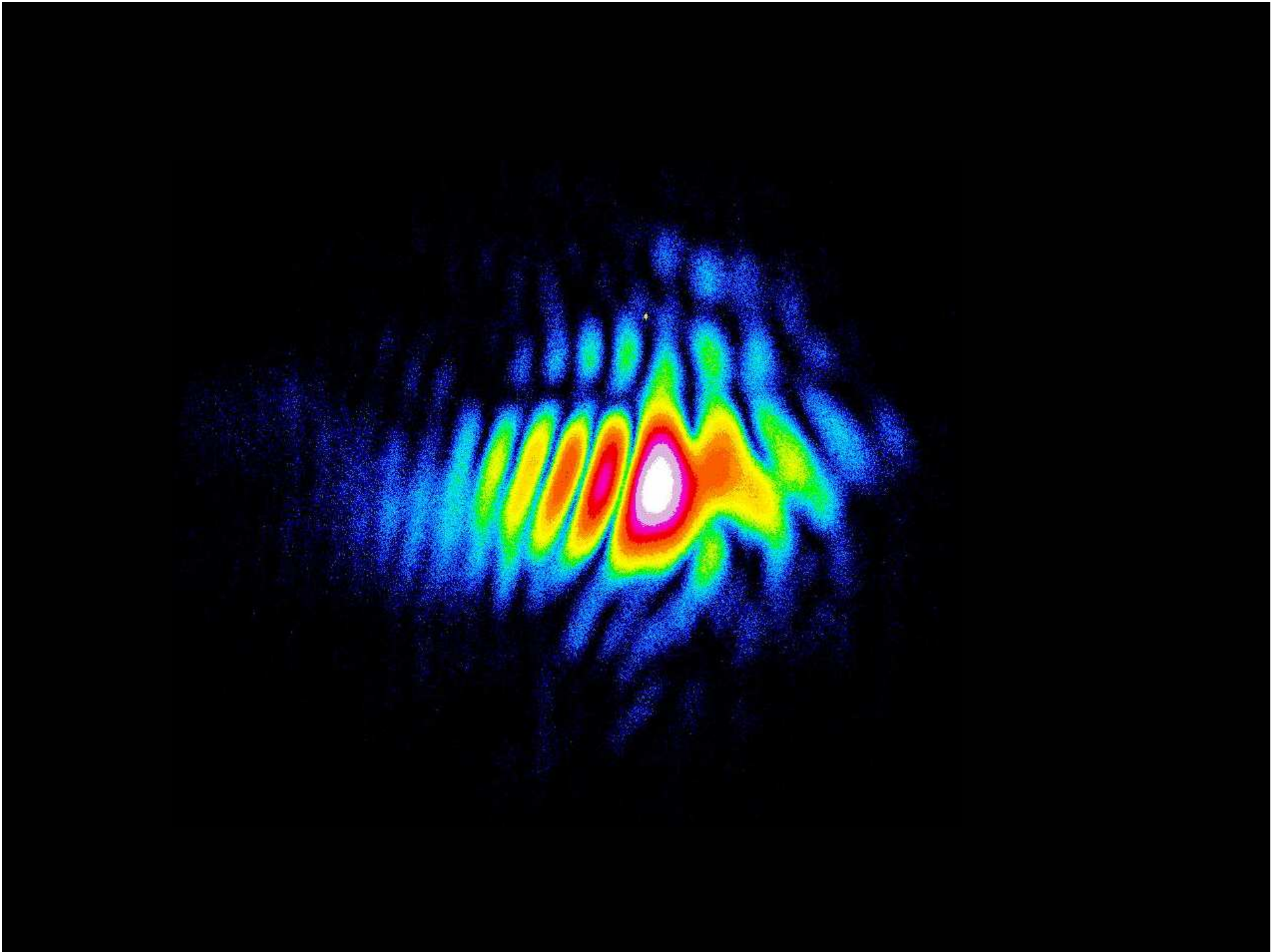
10  $\mu\text{m}$



Main difficulty:  
**Select only one grain!**

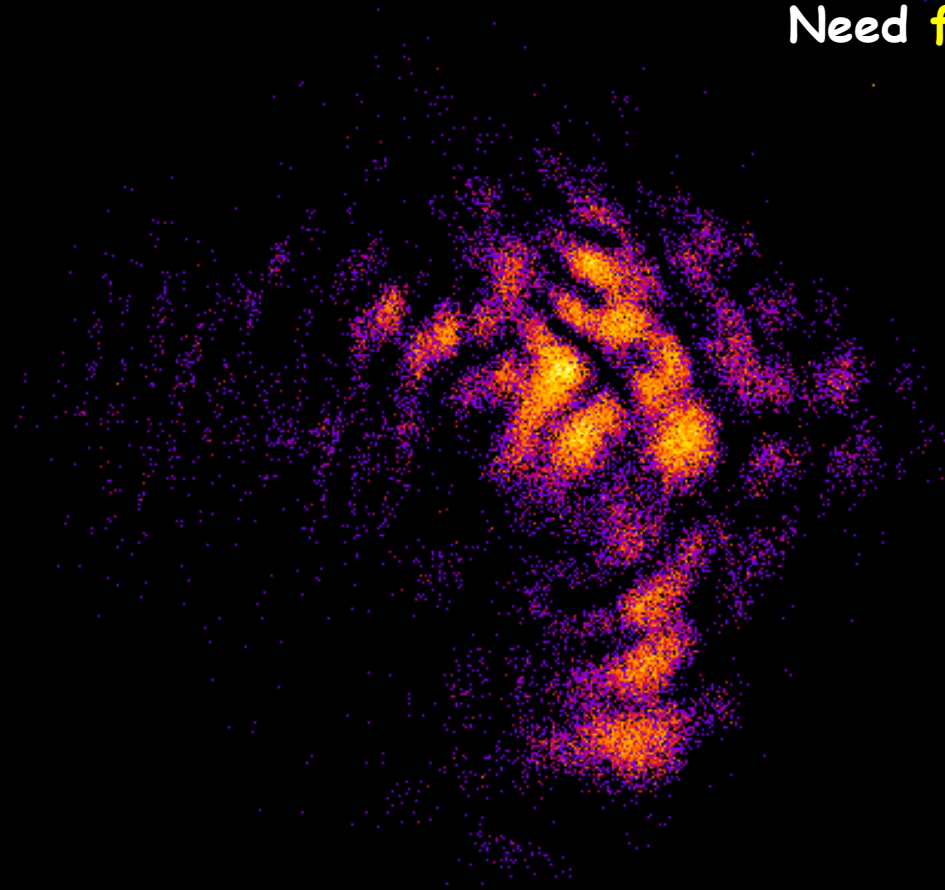
**Asymmetry** of fringes  
due to **strain**





(111) « rocking curve » ( $20 \times 0.015^\circ$ ) @5.2 keV  
of a  $\sim 800 \text{ nm} \times 800 \text{ nm} \times 200 \text{ nm}$  grain  
Selected by  $10 \mu\text{m} \times 10 \mu\text{m}$  slits

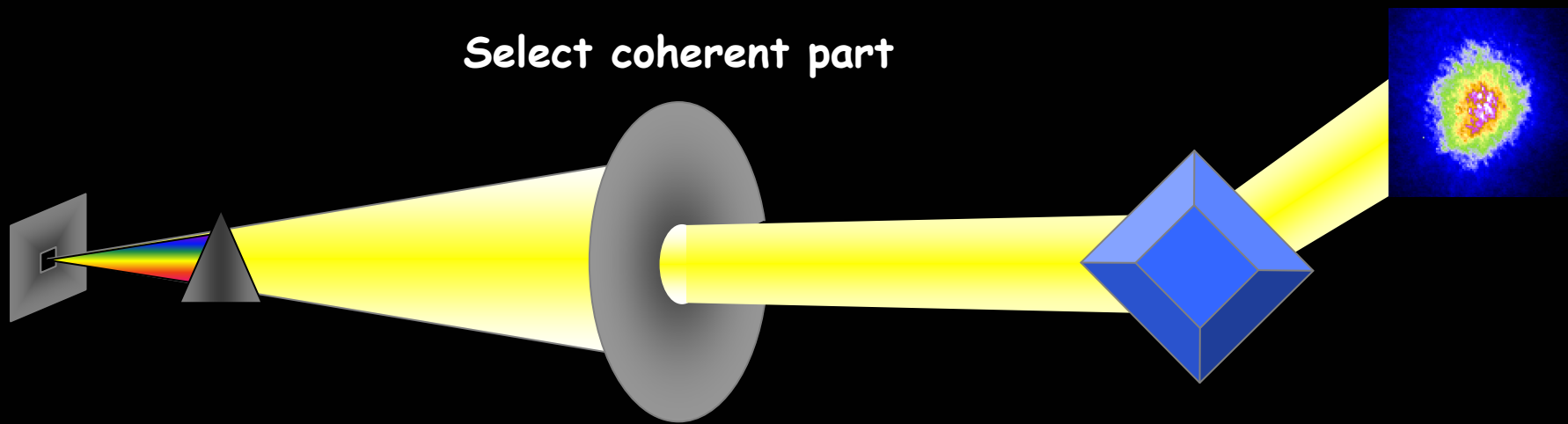
Measured with CCD  
Pixel detectors would work  
Need flux (focusing?)



8 h data acquisition

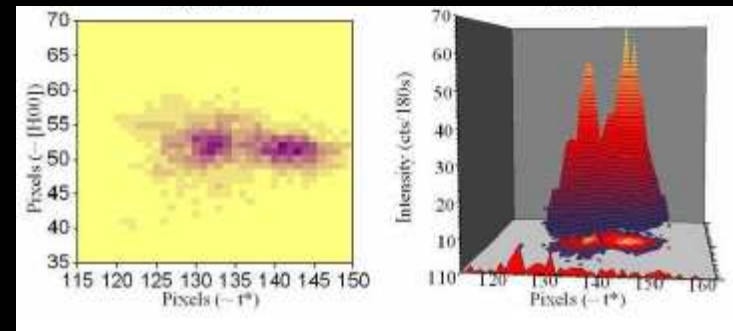
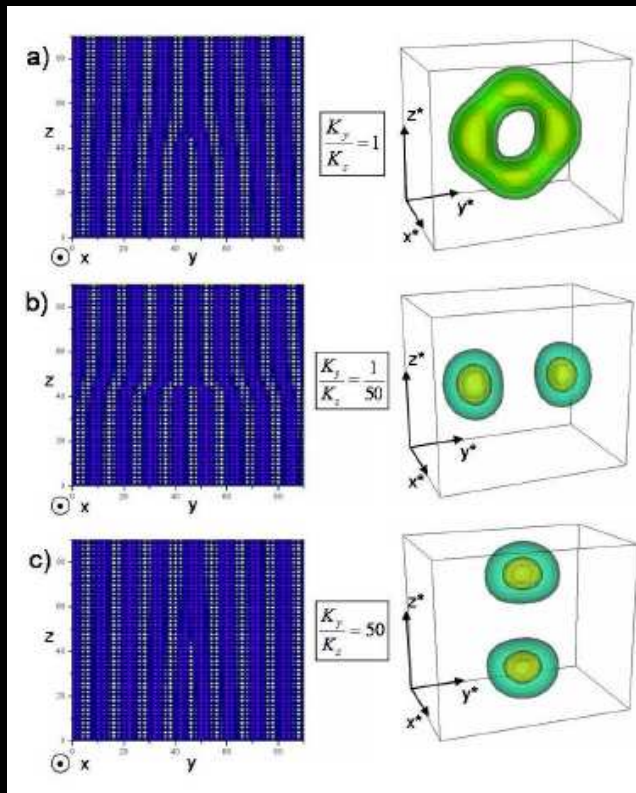
To be reconstructed!

# The other way...

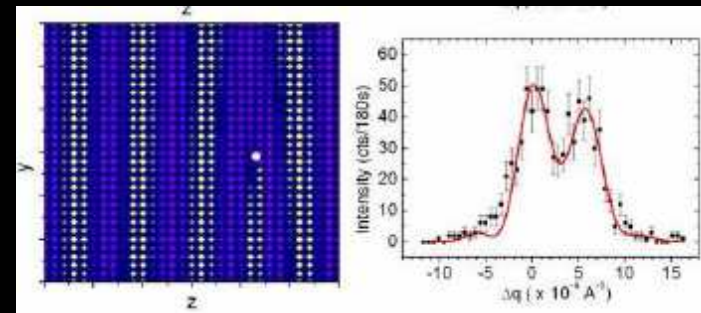


# Study of topological defects in the bulk

Dislocations make Bragg reflections **split**

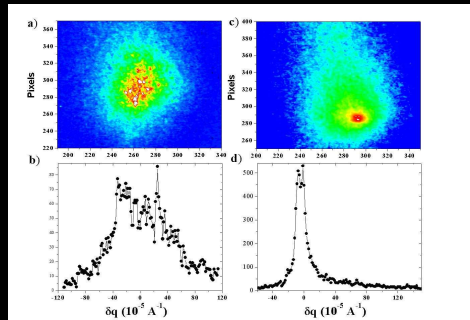


Dislocation in Cr **Spin Density Wave**  
V. Jacques et al. submitted



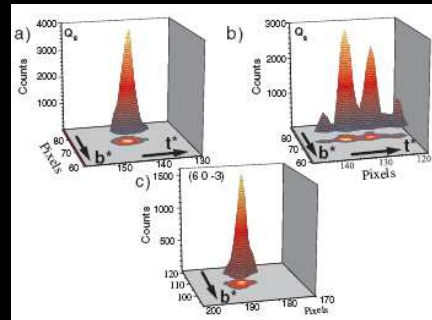
This effects allows to **image topological defects** of complex orders  
For magnetic reflections: the intensity is very small

## Surface vs bulk phase transition in SrTiO<sub>3</sub>



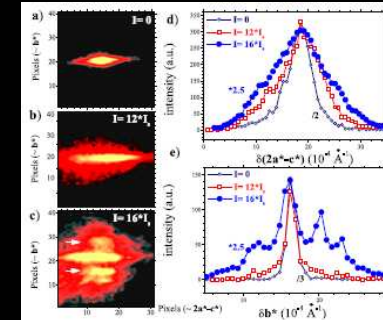
Ravy PRL98, 105501(2007)

## CDW Dislocation in KMoO<sub>3</sub>



Le Bolloc'h PRL95, 116401(2005)

## Micrometer structuration of a CDW under electric field



Le Bolloc'h PRL100, 96403(2008)

Provided the materials or the phases **resists irradiation damages...**  
(metal, perovskite but not all organic materials or complex oxides)

...this type of studies will benefit from **a higher coherent flux**  
(so far acquisition times are hours)

New exciting topics:

Study of phase transitions in nanomaterials  
Phase transformations upon electric fields, laser irradiation

# Conclusion

For coherent diffraction,  
there is a need for **higher coherent flux** in the 5-10 keV range

We already see progress in methods, algorithms, detectors and optics

- Reconstructions of **nano-crystals** (shapes and strains)  
free or embedded in a poly-crystal
- Phase transitions and **field-induced** phase transformations.

Thank you!

MERCI !