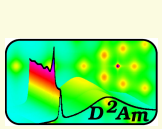
Pixel detectors in material science :

an opportunity or the future for synchrotron experiments ?



D2AM/CRG-ESRF
L. Cristallo, CNRS
Grenoble

J.-F. Bérar, N. Boudet,
J.-C. Clemens, P. Delpierre
. . .

CPPM IN2P3-CNRS
Marseille

ESRF Workshop :
New Science with new detectors,
Feb. 9-10th, 2005.

Summary.

The improvement of synchrotron sources will open new or difficult experimental domains but detectors have to follow this improvment.

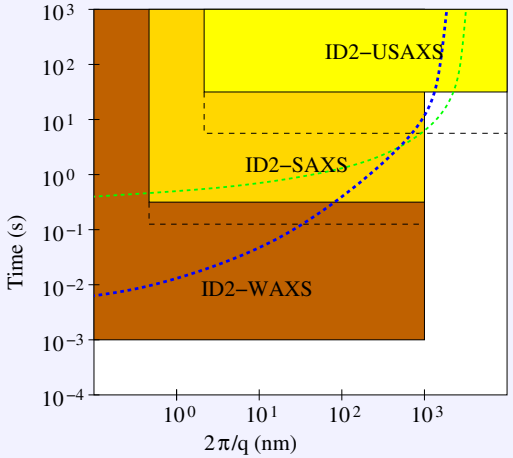
- source brillance $\nearrow$, SAXS beam photon flux $\nearrow$: $2.10^{13} \rightarrow 10^{15} \nu/s$
- photon energy used (Insertion Device) : $10 \rightarrow 20\, keV$

Detectors & material sciences scattering

Intensity range in scattering experiments $\rightarrow I(Q) \propto F^2(\rho_e)$			
$1 \rightarrow 10^4$	13b	mean structure	chemistry (biocrystallography)
$1 \rightarrow 10^6$	20b	ordering	correlation, incomensurate
$1 \rightarrow 10^9$	30b	SAXS	μm objects interaction, polymers

- Synchrotron $\rightarrow$ current flux on sample : $10^{11} - 10^{14}\,\nu/s$ (ID2, ID 9)
- Spot size at sample or detector position : $.5 \times .3 \rightarrow 0.05 \times 0.10\,mm^2$
- Diffraction : resolution $10^{-3\,o}\,\omega$ slits $10^{-2}\,mm^2$ at $0.5\,m$
- Crystal counting rate : $\gg 10^9 \nu/s$ within $10^{-2}\,mm^2$
- Powder diffraction : 10^4 points (measurement step $5\,10^{-3\,o}$)
- SAXS : 10^3 points centered on direct beam (apperture $0.01 \rightarrow 0.5\,rad$)

SAXS and Kinetics

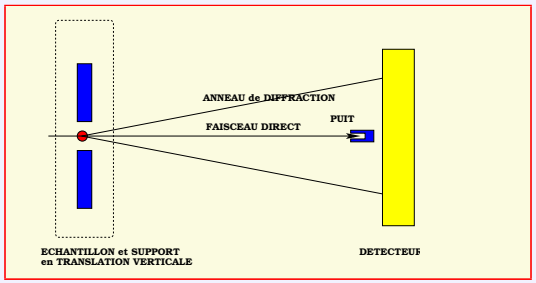


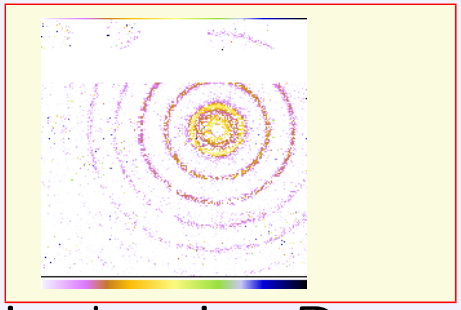
- ID2-USAXS appears limitated by the optic and will take profit from all source improvement (dashed).
- ID2-WAXS with higher signal is limited by detector exposure time and repetition rate.
- BM2-SAXS (green) is limited by flux and detector, pixels would lead to blue curve.

Improving Stopped Flow solution chemistry down to the msec timescale is more a question of detector than source both on IDs and BMs.

XPAD2 200 × 200 pixels

- exposure : $1ms \rightarrow 8300s$
- reading : $2ms$
- images : $423 < 10ms$
  $233 >= 10ms$



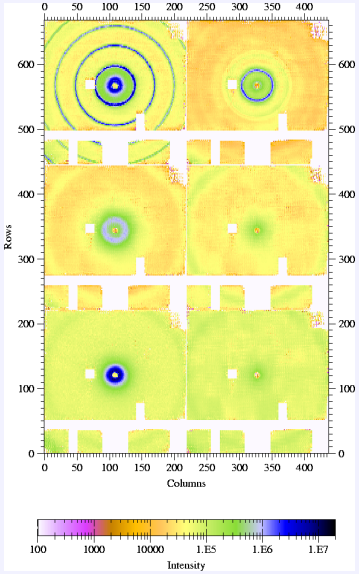


10ms images of a 2s movie showing Bragg rings while the sample crosses the beam at D2AM SAXS camera.

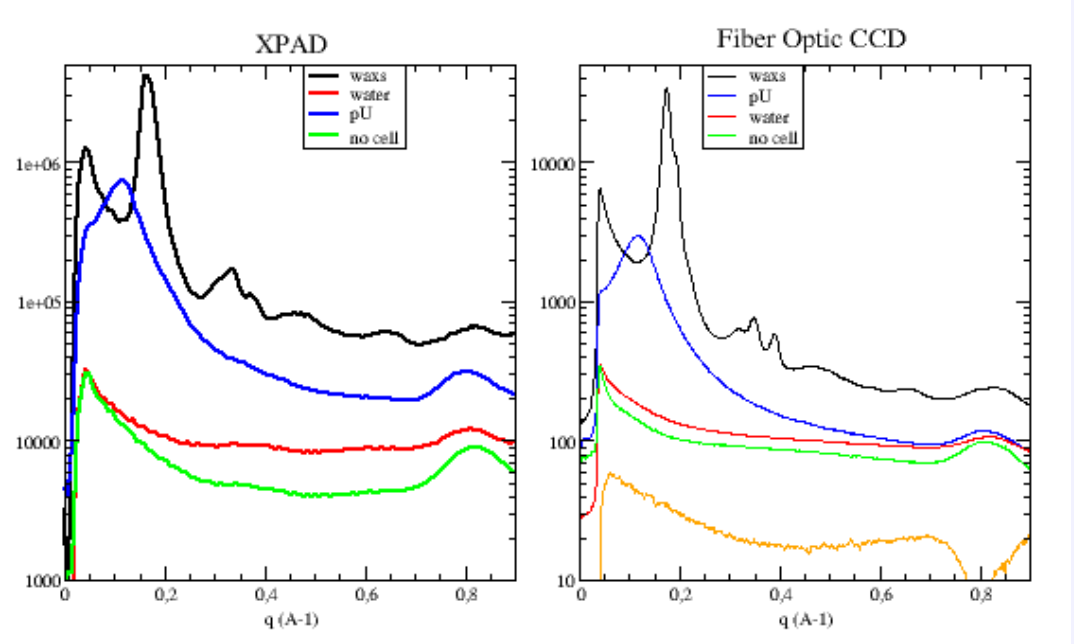
SAXS and weak scatterers

Samples recorded at BM2 SAXS camera using XPAD detector at $20\,keV$.

- Ag Behenate
- Bee waxs
- Polyurethan
- Empty cell
- Teflon
- Water



Data have been compared with FOB CCD* ones using the same setting.



The low noise achieved with pixel detectors allows to improve the measurement of weak scatterer like water : the signal observed without sample is really lower with XPAD than with the CCD (due to PSF tails† of beam)

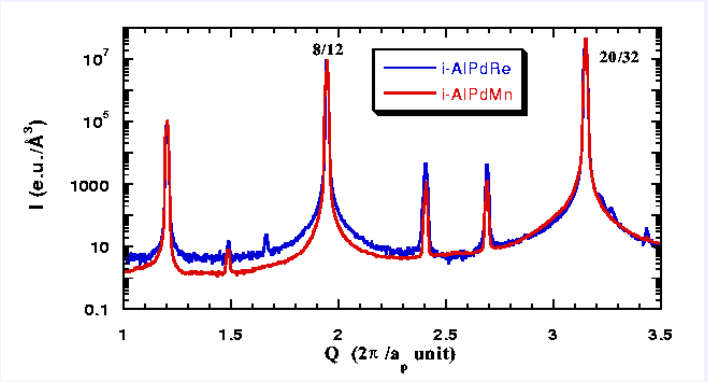
† CCD and Pixel detector comparison : *C. Ponchut et al., SRD-IEEE Conference, Rome 2004*
* PI-SCX-1300, Roper Scientific (EEG 1340x1300, $50\mu m$ pixel size, dark corrected)

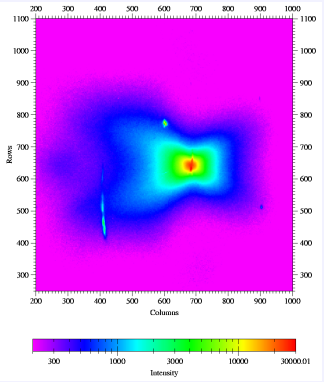
Ordering/Defects in crystal

Diffuse scattering in icosaedral quasi-crystals :

Counts range on 7 orders of magnitude

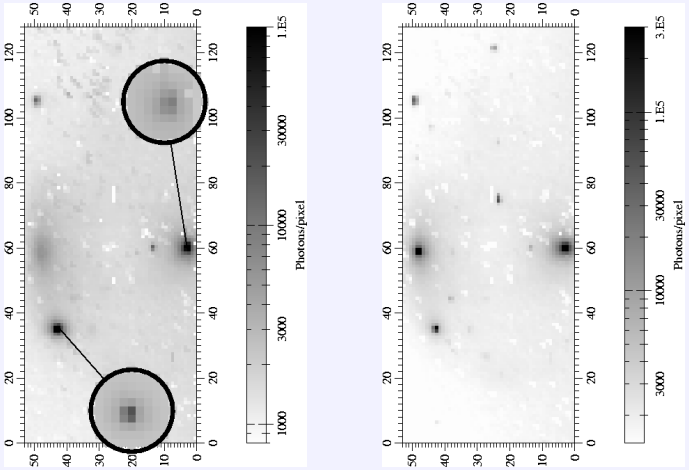
Dynamic extended by filters, time consuming mapping





Complex shape of the diffusion around a Bragg peak obtained by adding 10 (1000) frames on CCD. Out of peak to avoid blooming effects.

Diffusion measurement using a pixel detector using a fixed image.



On the associated rotation image (right) the higher peak ($\gg 10^6\,\nu/s$) saturates while crossing the Ewald sphere.

Data from M. de Boissieu, see Phil. Mag. Let. (2001) 81, 273-283 and (2003) 83, 1-29

Ordering/Defects in crystal (2)

The brillance of new sources will make, in principle, 2D detectors experiments recording "orbital ordering", magnetic superstructure . . . achievable.

But recording such very weak signals is not only a problem of intensity* but also of a clean experimental setup.

This kind of lines can easily be confused with spurious surface lines.

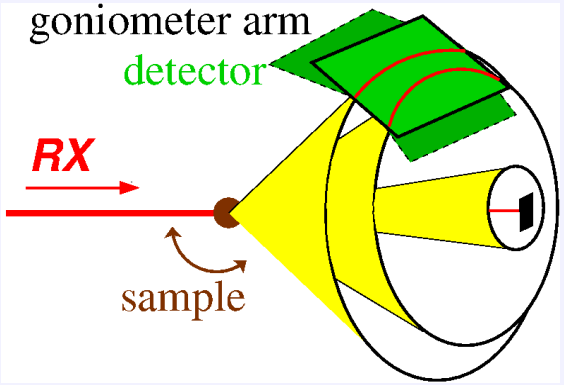
Diffusion is more difficult to reduce with 2D detectors, this has to be considered in building new detectors to take a real profit of source improvement.

magnetic structure of NiO *F. de Bergevin and M. Brunel, Phys. Lett. 39A, 141 (1972)*

High resolution powder crystallography

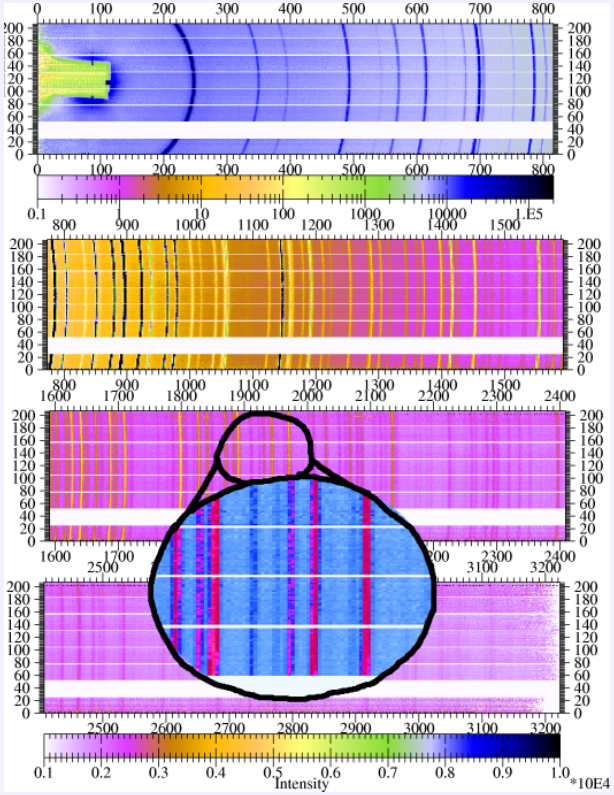
A slightly lower resolution than the analysor one is achieved with slits far from sample. With oscillating reactor the time/step is not only defined by flux but also by sampling period.

- Diffraction along cones
- Data redondancy with 2D detector

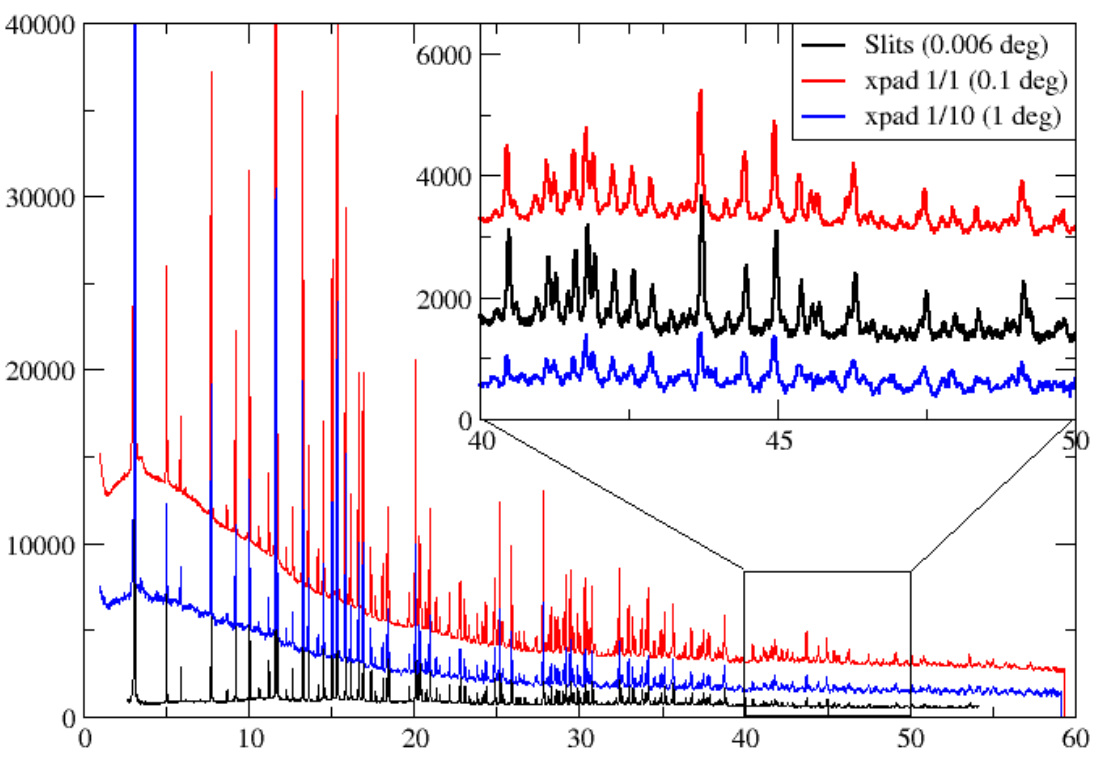


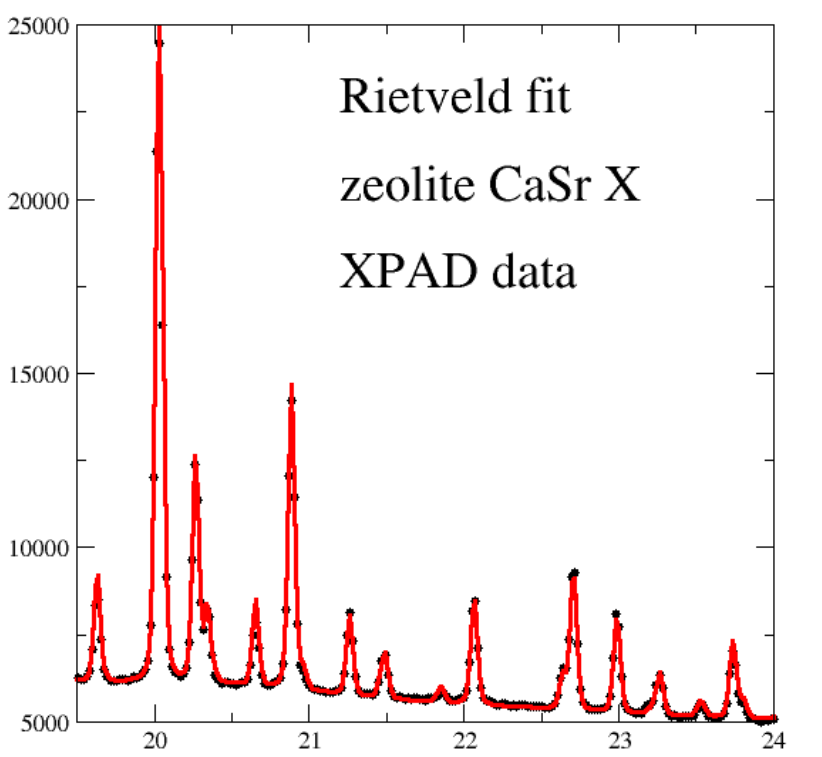
Wide dedicated powder detectors (10^4 steps) can be considered or moving smaller ones to reduce acquisition time.

- Complex pattern of a Zeolite
- 60^o collected at high resolution
- XPAD apperture 4^o at $1m$
- Debye-Scherrer film



High resolution powder crystallography (2)





Rietveld method : R_{wp}=8.8%
R_{exp}=4.1% and R_{bragg}=4.4%
Atomic parameters same as conventional
Whole experiment time $\rightarrow$ 1/20.

Data recorded in 1/200 time will lead to very similar results but extracting procedure needs improvments.

S. Basolo et al., IEEE-TNS (2005) accepted)

Low resolution powder crystallography

Very quick reactions, for solid state scientists, do not require such a high resolution but more flux and better detectors.

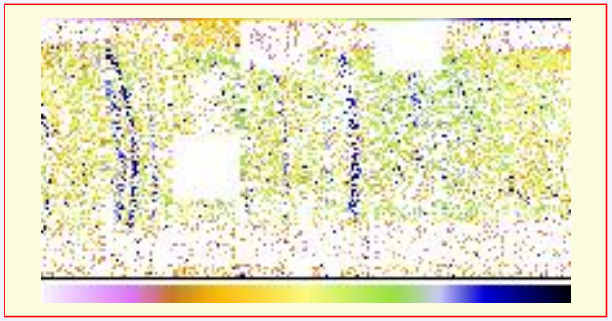
- Laser melting and solidification of oxides.
- Magnetic induction melting and solidification of metals.
- Self-Propagating High-Temperature Synthesis of alloys.
- ...

These systems are badly triggered and memory buffers must ensure to record the short phenomenon, most transformations occur in $\approx 100\,ms$ but the whole process takes a few seconds ($\approx 10\,s$).

Low resolution powder crystallography (2)

To study such phases diagram, various setups can be considered.

- Moving films (IP) are often too slow.
- Gas detectors with dedicated acquisition electronics (256 frames of 10 ms, no read out time) : limited detector counting rate (JF Javel, Thesis, Nancy 1998)
- X-Ray Intensifier and Frelon CCD camera (25ms frames but readout time 110ms/frame) : (C. Curfs, Thesis 2002, ID11 $2\,10^{12}\,\nu/s$)
- Pixel or strip detectors.
  the way to succes if they reach the angular apperture needed,
  limited by windows of cell and the prototype number of pixels.
- ...

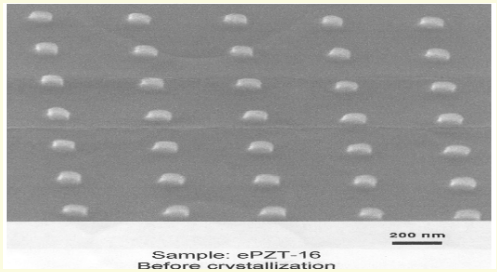


20 ms images of a 4s movie showing recrystallisation of $CaAl_4O_7$ during quench.

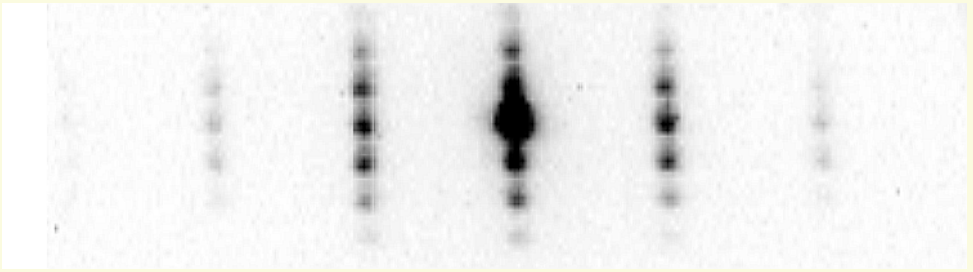
Quantum objects crystallography

These new materials appears as layers grown on dedicated surfaces. They need both GI-SAXS characterisation and GI-crystallography.

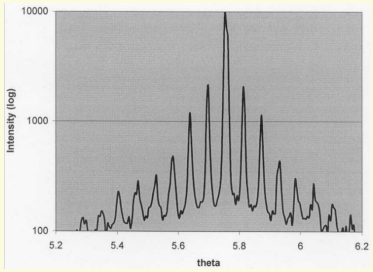
- GI-SAXS leads to detector requirements similar to SAXS.
- GI-crystallography using slits is time consuming.



Edched lattice



diffraction recorded with CCD



slits section

2D detectors will not waste such beam time but a very high dynamical range is required.
Non linearity of reciprocal space may require post processing of numerous images.

Thank you for your attention

and to all unlisted colleagues

- involved in detector development
- requiring new development for studying their materials
- participating to beamline improvments
- . . .