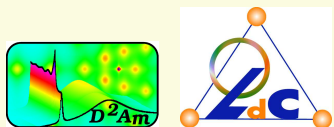


Pixel detectors in material science : an opportunity or the future for synchrotron experiments ?



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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 improvement.

- source brilliance \nearrow , SAXS beam photon flux \nearrow : $2 \cdot 10^{13} \rightarrow 10^{15} \nu/s$
- photon energy used (Insertion Device) : $10 \rightarrow 20 \text{ keV}$

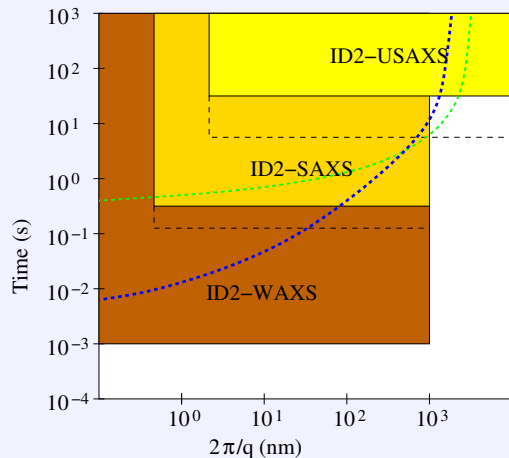
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Detectors & material sciences scattering

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, incommensurate
$1 \rightarrow 10^9$	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 : $.5 \times .3 \rightarrow 0.05 \times 0.10 \text{ mm}^2$
- Diffraction : resolution 10^{-3} ° 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 $5 \cdot 10^{-3} \text{ }^\circ$)
- SAXS : 10^3 points centered on direct beam (aperture $0.01 \rightarrow 0.5 \text{ rad}$)

SAXS and Kinetics

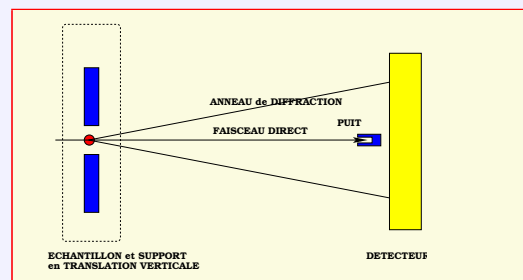


- ID2-USAXS appears limited by the optic and will take profit from all source improvement (dashed).
- ID2-WAXS with higher signal is limited by detector exposure time and repetition rate.
- BM2-SAXS (green) is limited by flux and detector, pixels would lead to blue curve.

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 → 8300s
- reading : 2ms
- images : 423 < 10ms
 233 ≥ 10ms

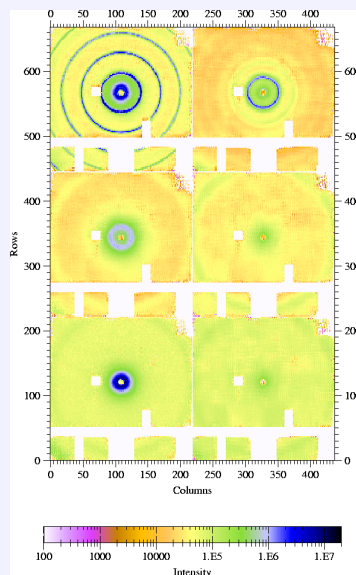


10ms images of a 2s movie showing Bragg rings while the sample crosses the beam at D2AM SAXS camera.

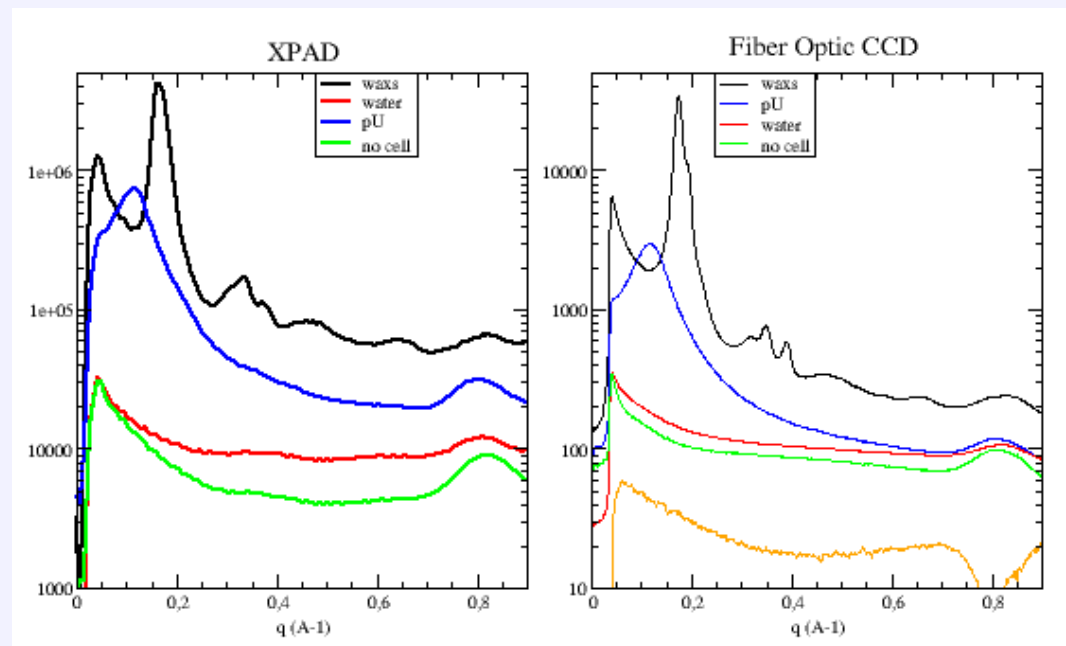
SAXS and weak scatterers

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

- Ag Behenate
- Bee waxes
- 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)

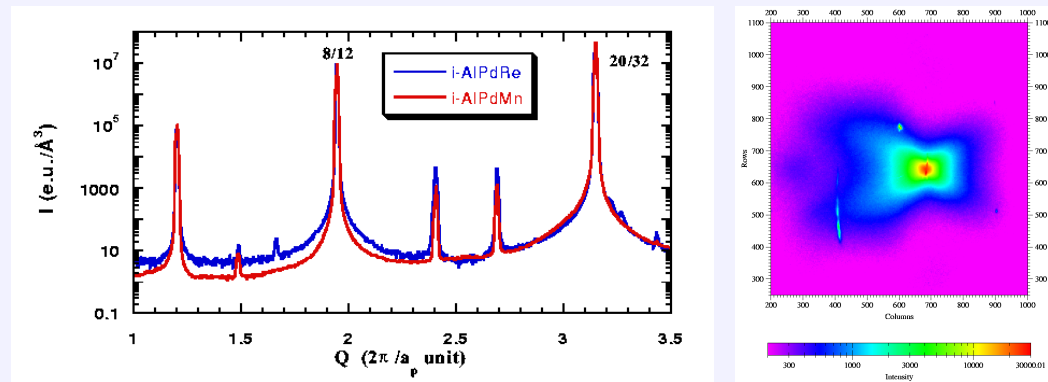
[†] CCD and Pixel detector comparison : *C. Ponchut et al., SRD-IEEE Conference, Rome 2004*
* PI-SCX-1300, Roper Scientific (EEG 1340x1300, 50 μ m pixel size, dark corrected)

Ordering/Defects in crystal

Diffuse scattering in icosahedral quasi-crystals :

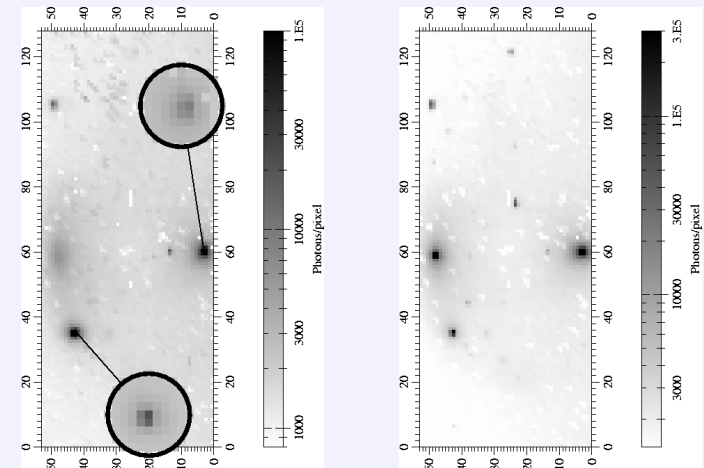
Counts range on 7 orders of magnitude

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 brilliance 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.

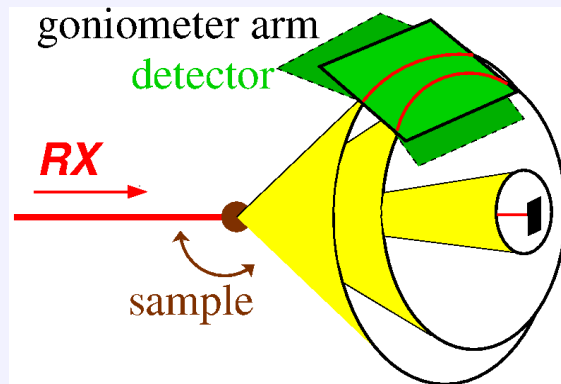
Diffusion is more difficult to reduce with 2D detectors, this has to be considered in building new detectors to take a real profit of source improvement.

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

High resolution powder crystallography

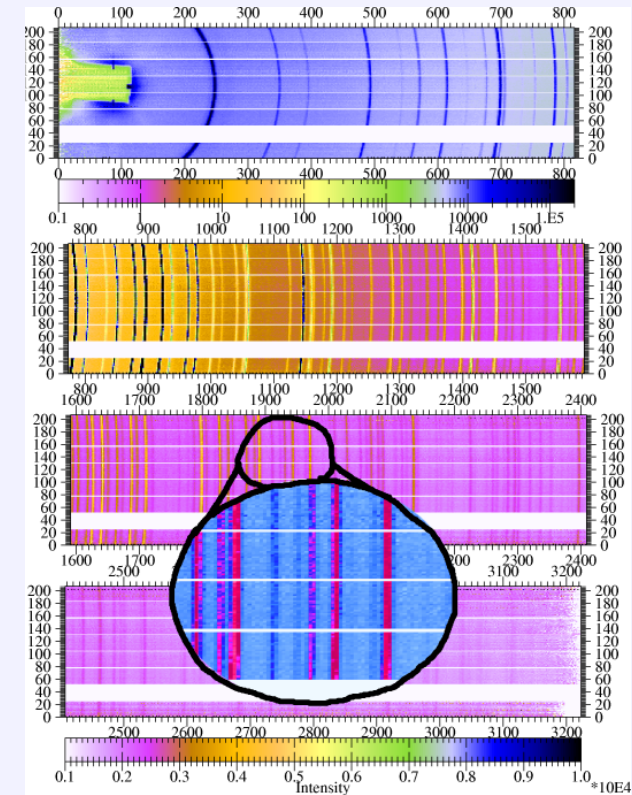
A slightly lower resolution than the analyser one is achieved with slits far from sample. With oscillating reactor the time/step is not only defined by flux but also by sampling period.

- Diffraction along cones
- Data redundancy with 2D detector

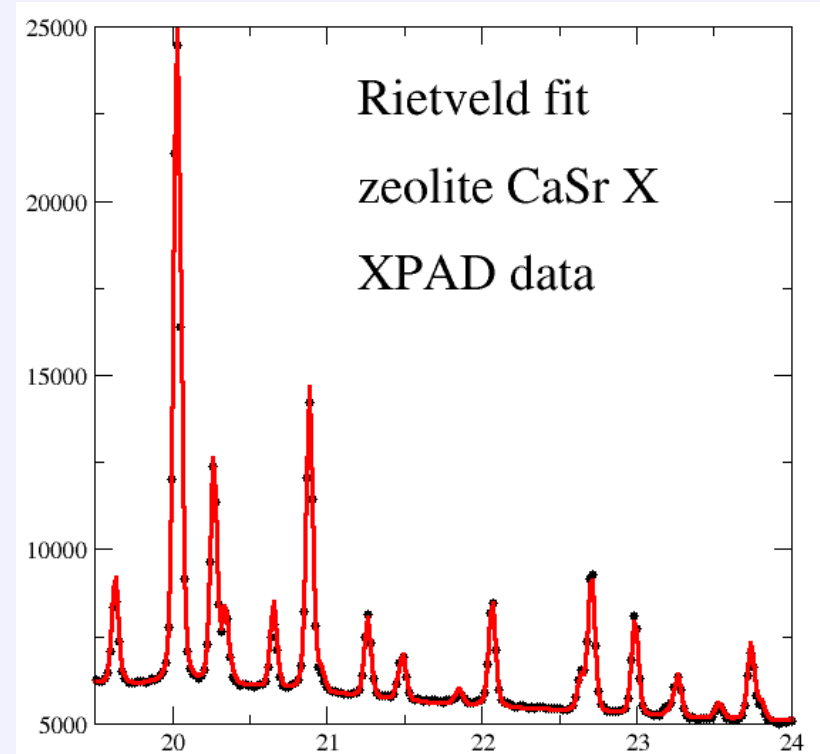
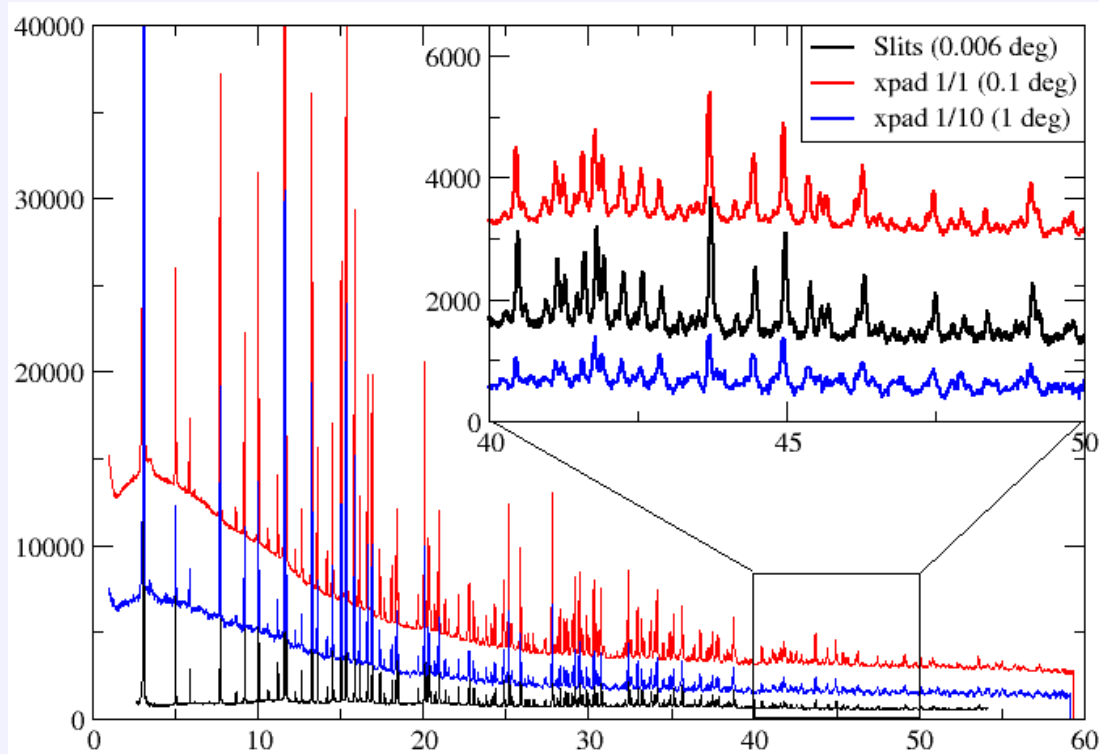


Wide dedicated powder detectors (10^4 steps) can be considered or moving smaller ones to reduce acquisition time.

- Complex pattern of a Zeolite
- 60° collected at high resolution
- XPAD apperture 4° 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
improvements.

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.

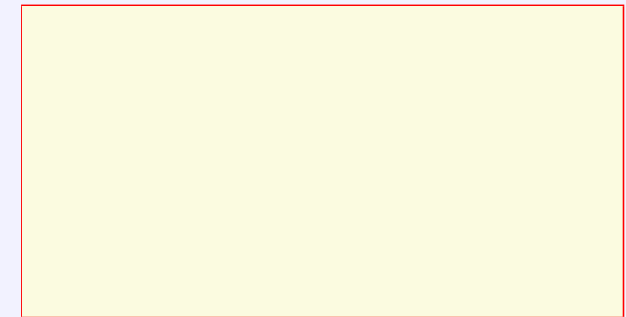
- Laser melting and solidification of oxides.
- Magnetic induction melting and solidification of metals.
- Self-Propagating High-Temperature Synthesis of alloys.
- ...

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

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 $2 \cdot 10^{12} \nu/s$)
- 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.



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 improvements
- ...