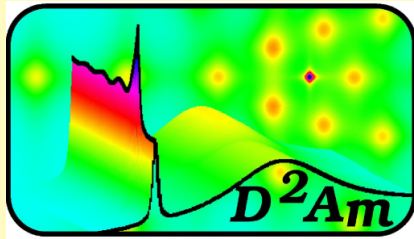


A multi elements assembly for X-ray synchrotron radiation XPAD : pixels detector for material sciences.



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Material sciences :

2d X-ray detectors for scattering

Imaging : → X-ray microscopy, X-ray topography, X-ray radiography

Spectroscopy : chemical composition (XAS), short order range (EXAFS)

Scattering by beam → $I(Q) \propto F^2(\rho)_e$

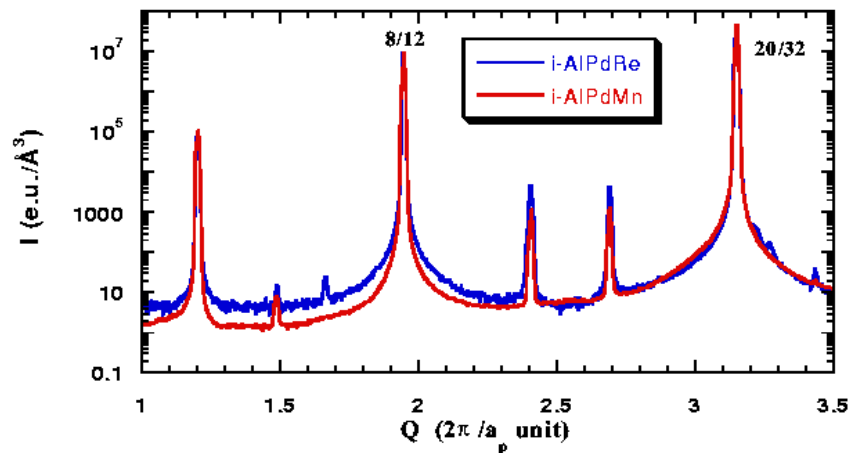
Intensity range in scattering experiments

$1 \rightarrow 10^3$	atomic structure	(biocrystallography)
$1 \rightarrow 10^6$	ordering	correlation, incommensurate
$1 \rightarrow 10^9$	SAXS	μm objects interaction, polymers

- Synchrotron → current flux on sample : $10^{12} - 10^{14} \nu/s$
- Counting rate : $10^9 \nu/s$ within $10^{-2} mm^2$
- Resolution : angular $10^{-3} \text{ }^\circ \rightarrow 100 \mu m$ linear

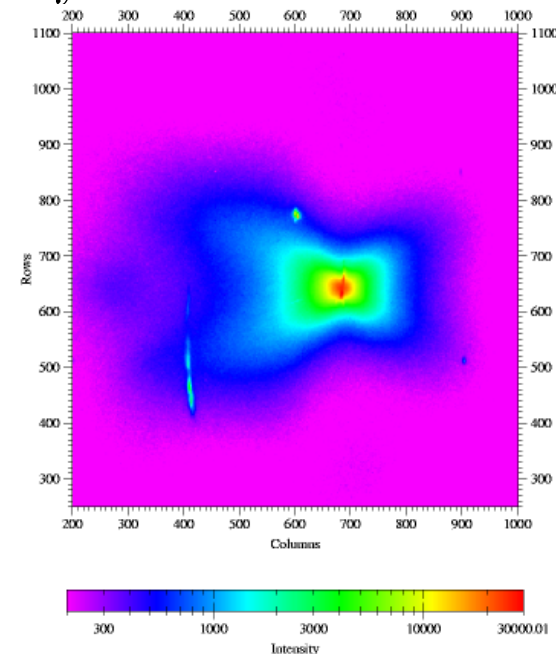
in 2004, on BM2 and other ESRF beamlines

Very demanding experiments use slits and photomultipliers to reach the required quality



Diffuse scattering in icosahedral quasi-crystals : 7 orders of magnitude are necessary to measure this signal.

In structural works, CCD cameras with indirect photon detection are commonly used



Complex shape of the diffusion around Bragg peak obtained by adding 10 frames of CCD.

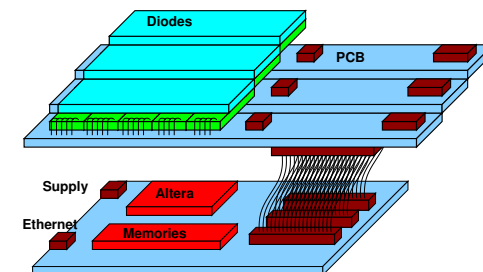
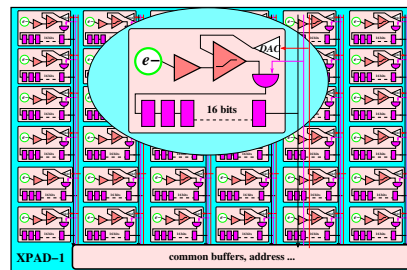
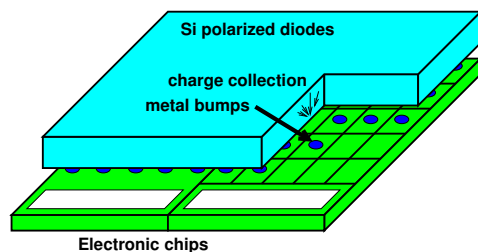
Data from M. de Boissieu, see *Phil. Mag. Let.* (2001) 81, 273-283 and (2003) 83, 1-29

The XPAD project

A pixel detector designed for D2AM-CRG/ESRF beamline experiments.

dynamic range	$> 10^9 \text{ count/pixel}$	$\Rightarrow 32$ bits architecture
saturation rate	$> 10^7 \nu/s/pixel$	$\Rightarrow \text{noise} < 0.1 \nu/s/pixel$
energy range	$5 \rightarrow 25 \text{ keV}$	from dynamic range
pixel size	$330 \times 330 \mu\text{m}^2$	mean spot size in 1995
exposure time	$1 \text{ ms} \rightarrow 1000 \text{ s}$	kinetics potentiality

Absorbed photons \rightarrow electron clouds \rightarrow charge migration \rightarrow electron bunches
 \rightarrow pixel threshold \rightarrow pixel counters \rightarrow on-board memories \rightarrow ethernet data

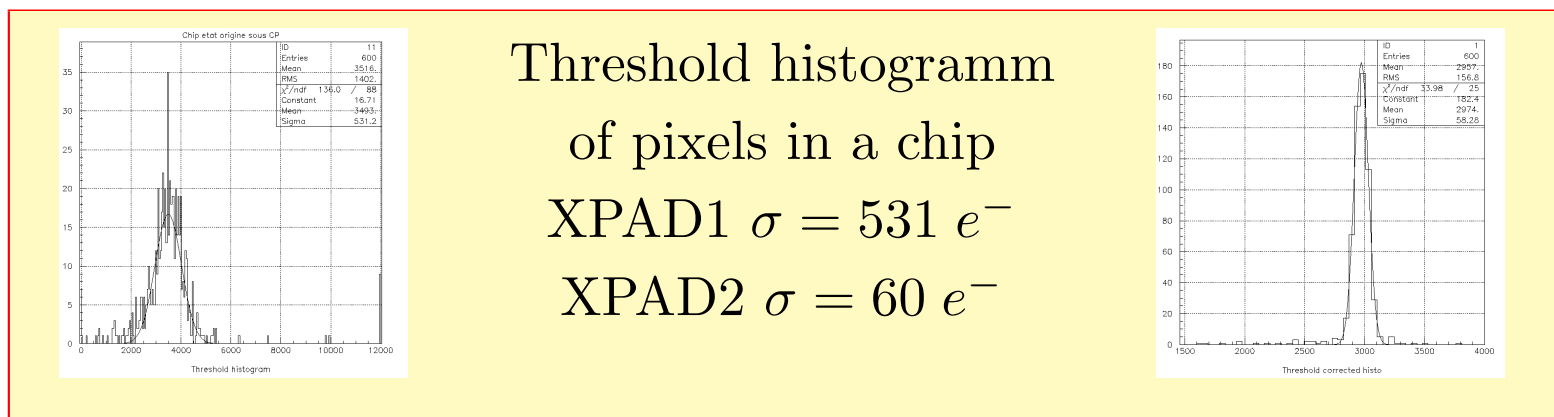


Boudet NIM A510 (2003) 41-44,

Berar J. Appl. Cryst. 35 (2002) 471-476

XPAD2 chips

- design from XPAD1 prototype : pixel size $330 \times 330 \mu m^2$
- same $0.8 \mu m$ CMOS technology (AMS) technology
- read out improved to fit the project requirements
- amplifier modified \rightarrow reduce the wide distribution of the threshold level
- smaller bounding pads : whole electronic noise $\approx 120 e^-$ reached



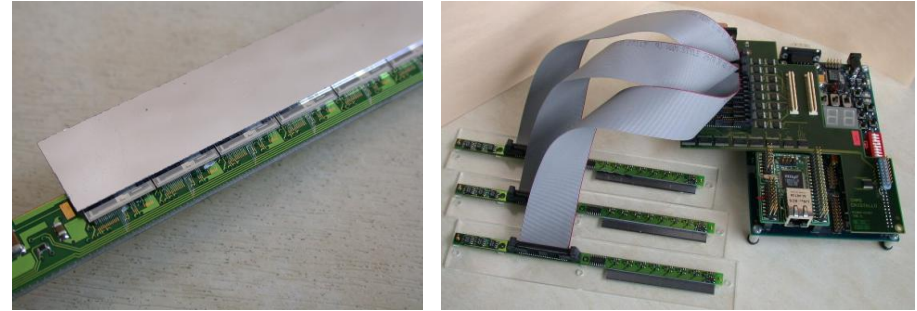
Electronics characteristic of the XPAD2 chips are very similar to XPAD1 ones
 \Rightarrow building of a large area detector.

XPAD1-2 : Delpierre IEEE-TNS 49 (2002) 1709

XPAD2 detector : 8 modules \times 8 chips

New Si diodes : $500\mu\text{m}$ thick \rightarrow efficiency 78 % @15keV, 21% @25keV

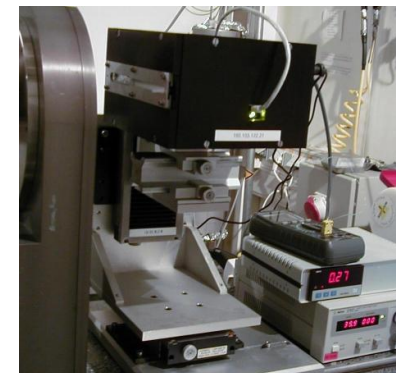
Diode \Rightarrow 8 chips of 24×25 pixels
PCB card : drivers and regulators.
Modules \Rightarrow acquisition card
Alterra Nios kit + ethernet



Tiled as close as possible
 \rightarrow reduce shading, dead zones.
Metallic holder \rightarrow few μm .
 200×192 pixels $\approx 68 \times 68\text{mm}^2$.

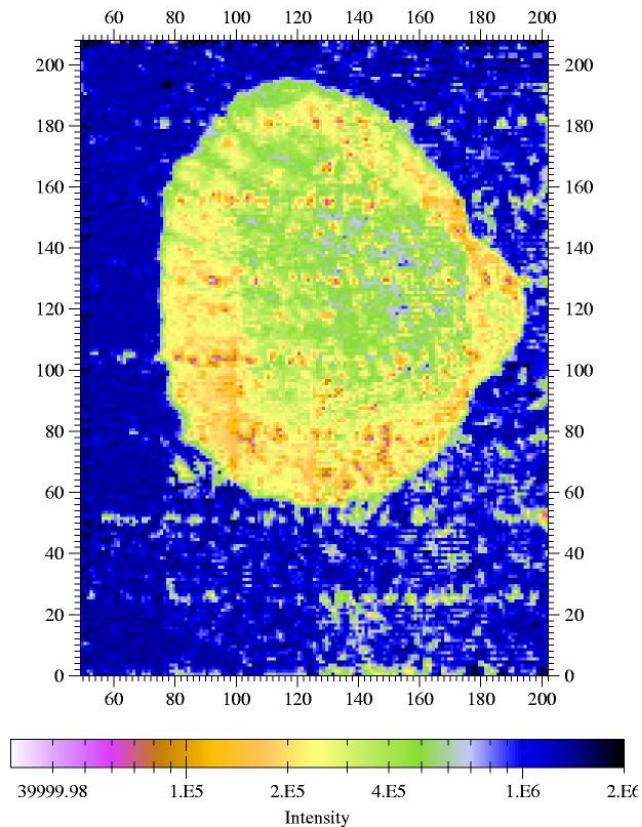
Interface software

developed using LabWindows/CVI
application software moves to Linux.



Short results

It is quite easy to present images even if numerous pixels are not well tune.
With 75% of pixels counting in a reasonable range, by overlapping 2 (or more) images obtained moving the detectors, one can reasonably get :



log scale



Absorption

Seashell

•

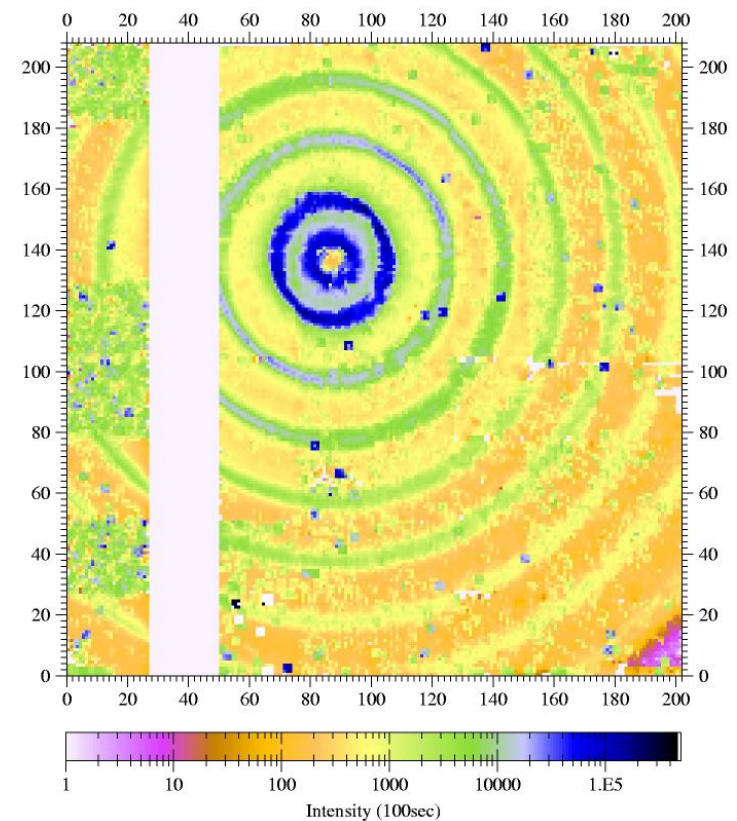


SAXS

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Ag. behenate

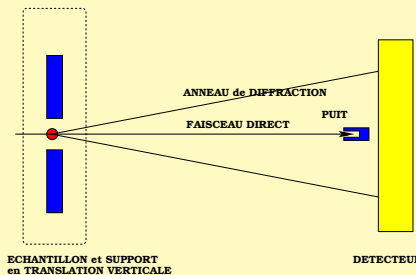
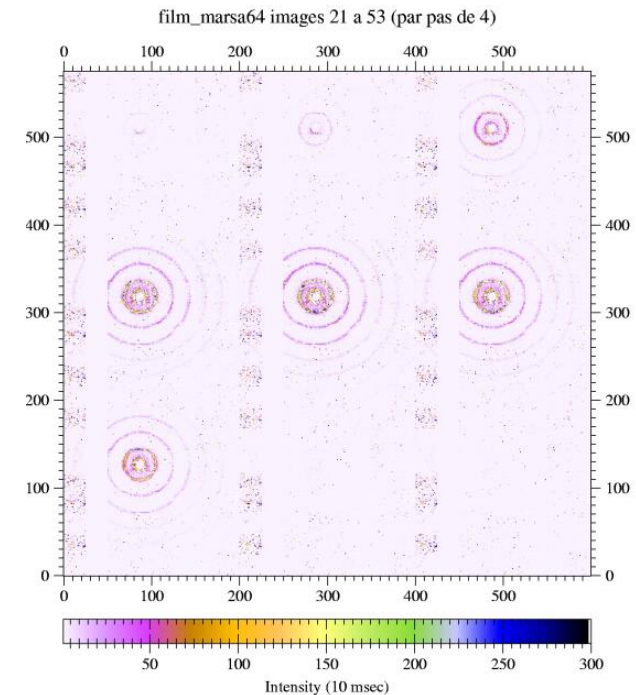
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Kinetics potentiality of XPAD2

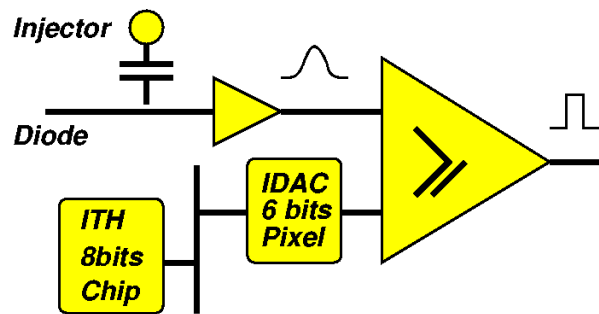
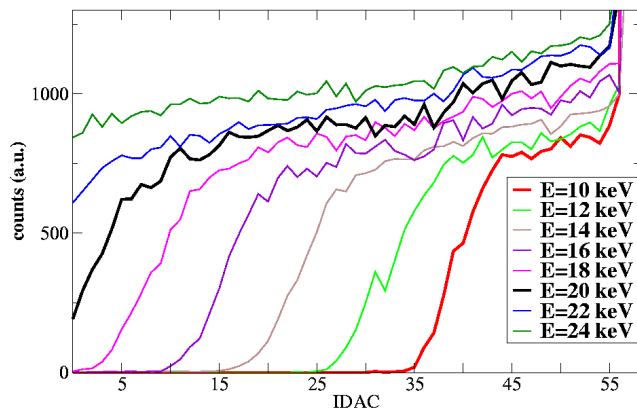
Whole electronic designed to allow kinetics studies (ms range)

- chips register 16bits + overflow
- on-board memories 32 bits
- exposure time : $1ms \rightarrow 8300s$
- dead time for reading :
 - whole image $2ms$
 - overflow $16\mu s$ each $10ms$
- on-board storage :
 - 423 images $< 10ms$
 - 233 images $\geq 10ms$



Images of 10 ms each taken off a 2s movie showing diffraction while the sample crosses the beam at D2AM SAXS camera.

XPAD2 calibration and dispersion



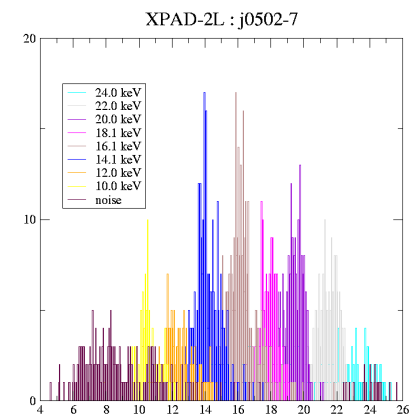
- *beam* E_x : monochromatic flat scattering (amorphous), noisy, time expensive
- *injection* E_{inj} : simulate the beam, quick and easy but need calibration
- Each pixel is described by : $C, \alpha, \beta, E_{inj}(noise)$

$$E_x = CE_{inj} = \alpha(I_{th}) + \beta(I_{dac})$$

$$E_x(noise) = CE_{inj}(noise)$$
- $\approx 4 \cdot 10^4$ pixels \Rightarrow automatic configuration/calibration procedure.

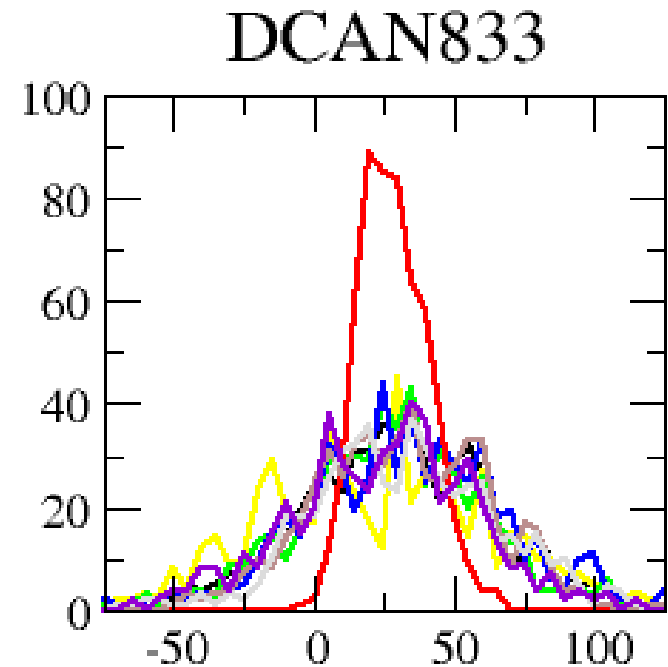
Knowing then these characteristic :

the setup of each chip at a given energy E can be defined as the value of the chip common threshold level I_{th} for which most of the pixels can be fine tune, $I_{dac} \in [0, 63]$.



XPAD2 calibration and dispersion

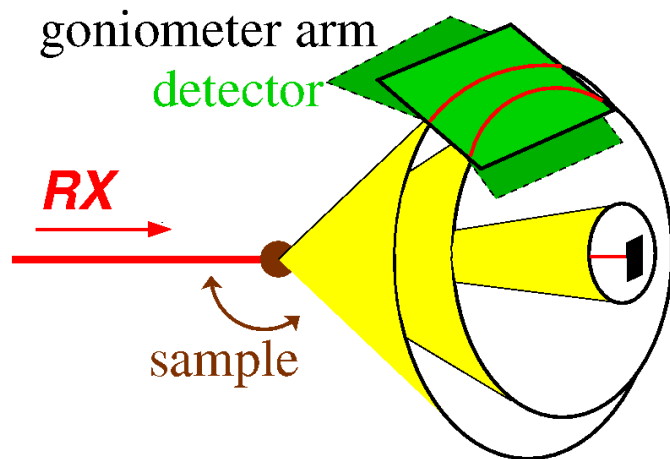
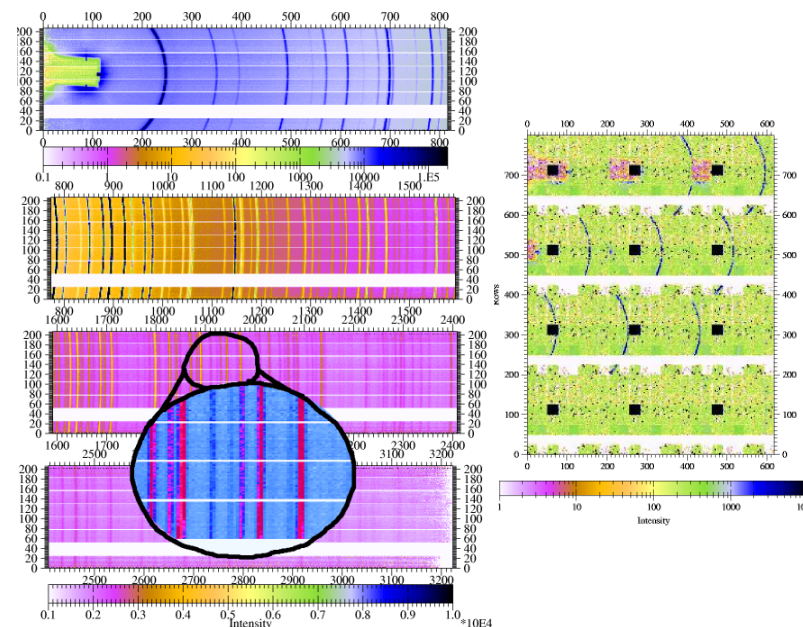
- XPAD2 initial threshold dispersion $60 e^-$
⇒ pixels not tuned $< 3\%$
- manufacturing problems :
leakage in bumping process
⇒ new foundry using the same masks
- threshold dispersion increase strongly
 $\approx 120 e^-$ on most chips
⇒ pixels not tuned $< 15\%$



However, even if all the pixels are not perfectly set, the XPAD2 detector appears as a useful tool for recording new data in SAXS and diffraction on a synchrotron beamline in the range 15 - 25 keV.

Powder diffraction application

- Powder diffraction : along cones
- Data redundancy with 2D detector
- Complex pattern of a Zeolite
- 60° collected at high resolution
- angular apperture 4° at 1m
- Reconstructed Debye-Scherrer film →



Resulting Y counts on pixel p :

$$Y_p = N_p^{-1} \sum f_q y_i(q(p, i))$$

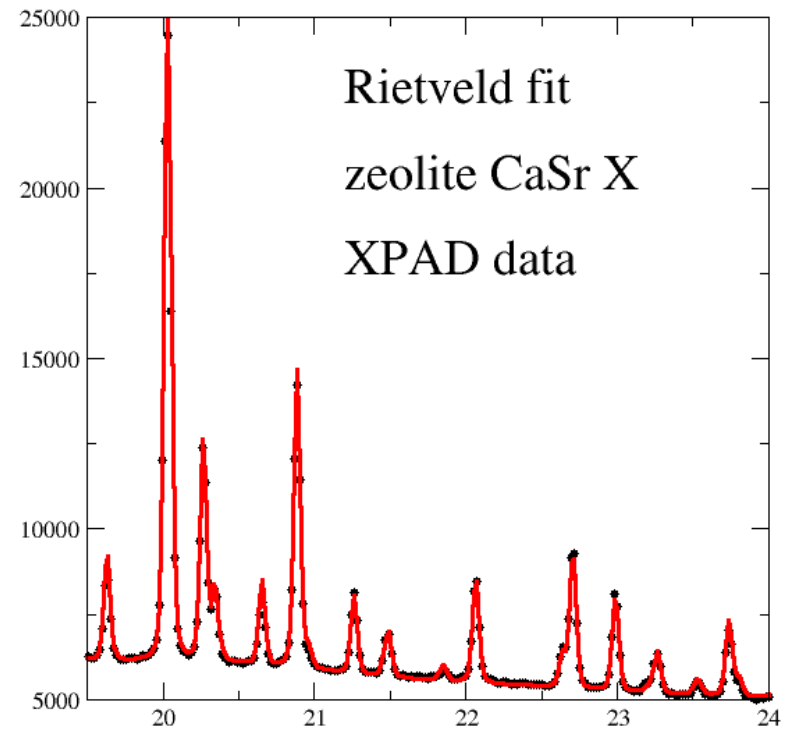
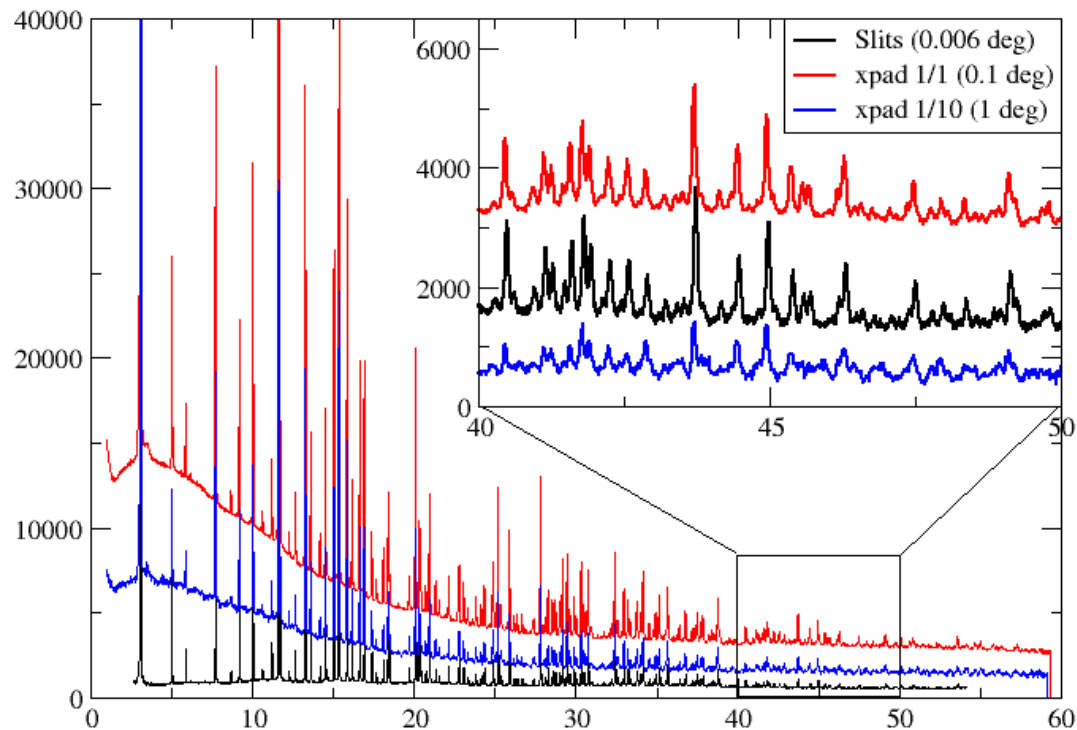
$y_i(q(p))$ counts on image i of pixel $q(p, i)$

f_q flatfield of pixel q :

minimisation of $\sum (Y_p - N_p^{-1} \sum f_q y_i(q(p, i)))^2$

Powder lines : $Y_{p \in Bragg} \rightarrow Y_{Bragg}$

Powder diffraction application



Rietveld method : $R_{wp}=8.8\%$

$R_{exp}=4.1\%$ and $R_{bragg}=4.4\%$

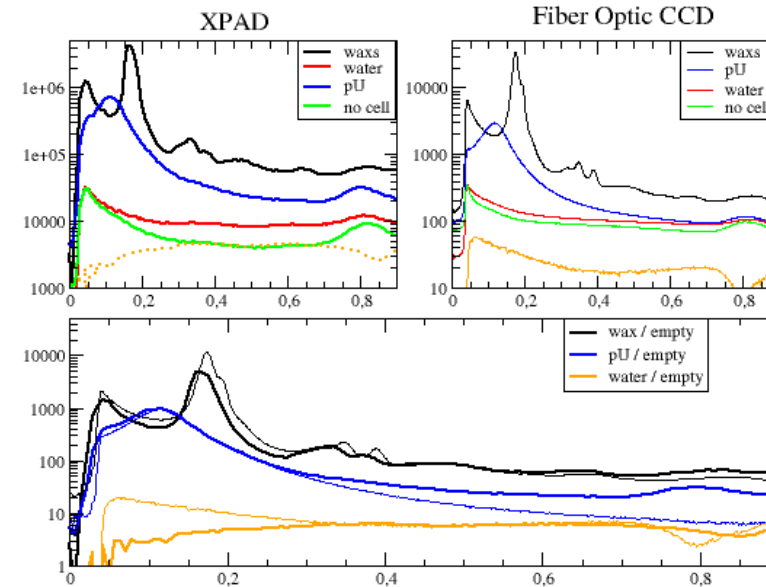
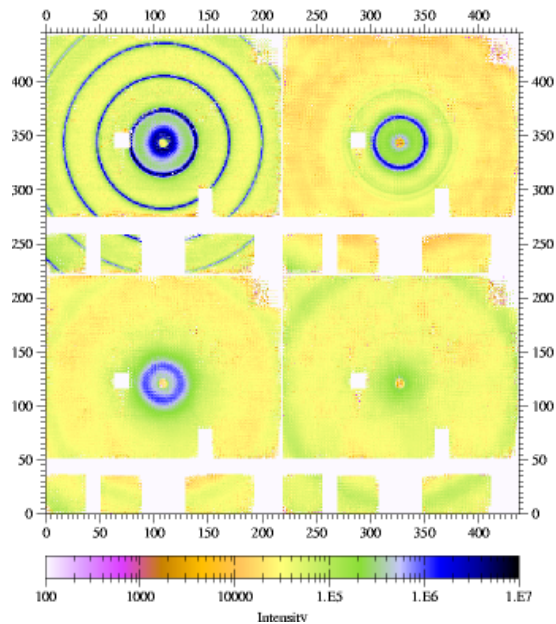
Atomic parameters same as conventional

Whole experiment time $\rightarrow 1/20$.

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

SAXS application

Scattering of some samples on BM2-SAXS camera using XPAD detector.



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

The low noise achieved with the XPAD detector allows to improve the measurement of weak scatterer like water : the signal observed without sample is really lower with XPAD than with the CCD (fluorescence, PSF tails ...)

* PI-SCX-1300, Roper Scientific (EEG 1340x1300, 50 μ m pixel size, dark corrected)

XPAD3

Setting up large array detectors

Our goal is to develop a real detector with more than 10^6 pixels. Such a detector will consist of several modules to be tiled together. This step has recently been validated by assembling 8 modules of 8 chips each.

Future developments

To achieve the number of pixels required in numerous experiments, we have already begun the design of a new chip XPAD3. It will use radiation-hard submicronic technology ($0.25\mu m$), which will allow the pixel size to be reduced to $100 - 150\mu m$ with similar or enhanced performance.

Thanks for your attention

and to colleagues working on (or close to) the beamline,
⇒ *without them no experiment could be done*