A multi elements assembly for X-ray synchrotron radiation measurement

XPAD : pixels detector for material sciences.

D2AM/CRG-ESRF
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Summary.

Detectors & material sciences scattering

Imaging : $\rightarrow$ X-ray microscopy, X-ray topography, X-ray radiography
Spectroscopy : chemical composition (XAS), short order range (EXAFS)
Scattering by beam $\rightarrow I(Q) \propto F^2(\rho)_e)$

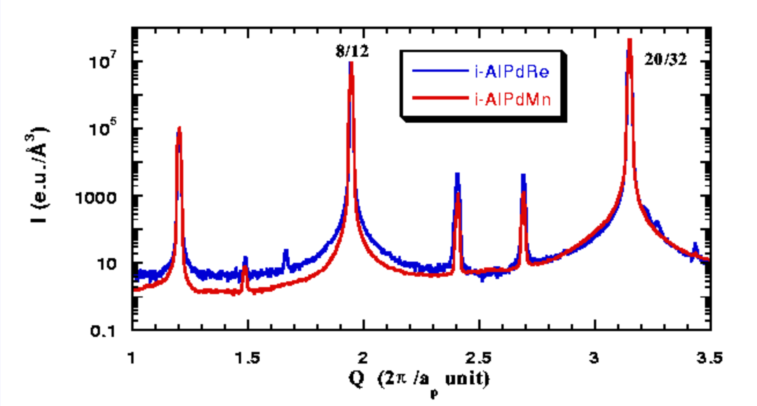
Intensity range in scattering experiments

$1 \rightarrow 10^4$	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 : $10^{11} - 10^{14}\,\nu/s$
- Spot size at sample or detector position : $1 \times 5 \rightarrow 0.05 \times 0.10\,mm^2$
- Counting rate : $10^9\nu/s$ within $10^{-2}mm^2$
- Resolution : angular $10^{-3\,o} \rightarrow 100\,\mu m$ at $0.5\,m \approx 0.01^{\,o}$

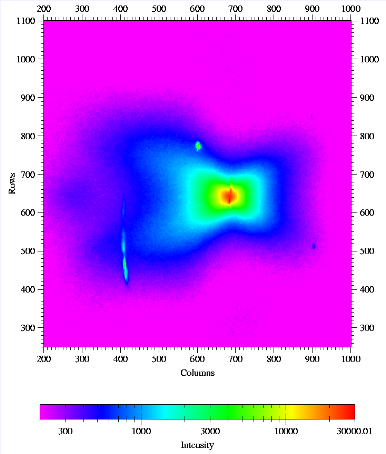
On BM2/ESRF beamlines in 2004

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



Diffuse scattering in icosaedral quasi-crystals : 7 orders of magnitude are necessary to measure this signal.
Dynamic extended by filters, time consuming mapping

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



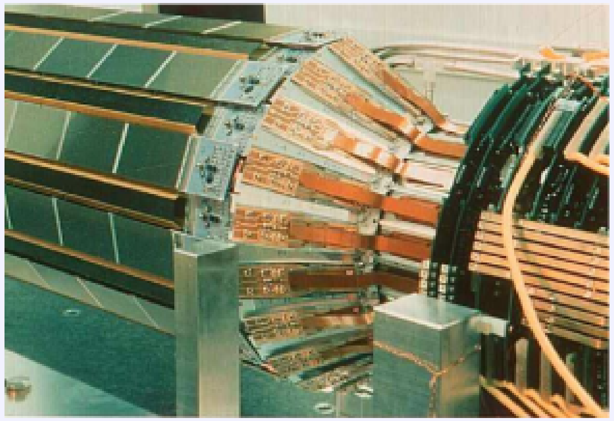
Complex shape of the diffusion around Bragg peak obtained by adding 10 (1000) frames.
Out of peak to avoid blooming effects

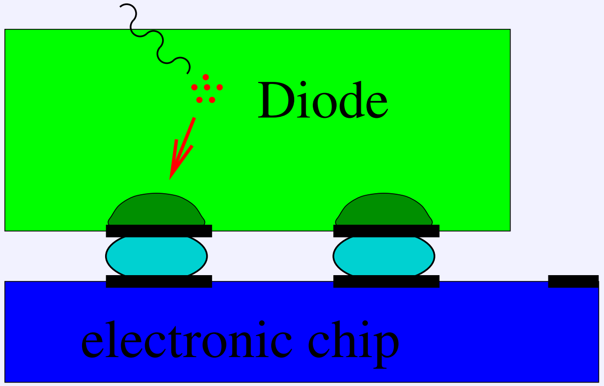
Data from M. de Boissieu, see Phil. Mag. Let. (2001) 81, 273-283 and (2003) 83, 1-29

D2AM-CRG/ESRF detector requirement

dynamic range	$> 10^9 count/pixel$	$\Rightarrow$ 32 bits architecture
saturation rate	$> 10^7 \nu/s/pixel$	$\Rightarrow$ noise $< 0.1\, \nu/s/pixel$
energy range	$5 \rightarrow 25\, keV$	from dynamic range
pixel size	$250 \times 400\, \mu m^2$	mean spot size in 1995
exposure time	$1 ms \rightarrow 1000\, s$	kinetics potentiality

High energy physics experiments lead to built detector like Delphi at CERN which uses the potentiabilities offered by microelectronics and direct photon conversion in silicon.





The silicium thickness $300 \mu m$ and the pixel sizes $330 \times 330 \mu m^2$ were convenient to our beamline requirements leading to the project of buildind a new X-ray detector starting from the knowledge of the Delphi detector peoples.

The XPAD project

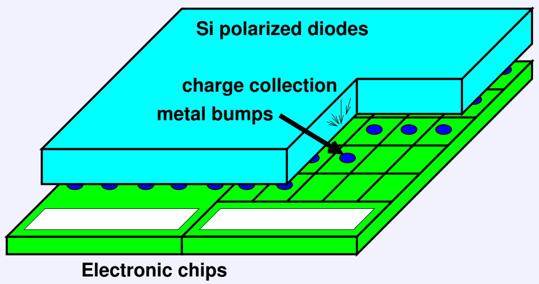
- Absorbed photons
- → electron clouds
- → charge migration
- → electron bunches
- → pixel threshold
- → pixel counters
- → on-board memories
- → ethernet data

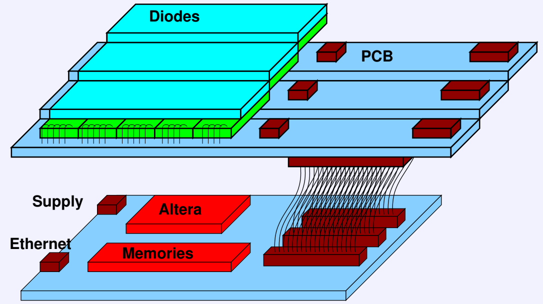
Diodes :

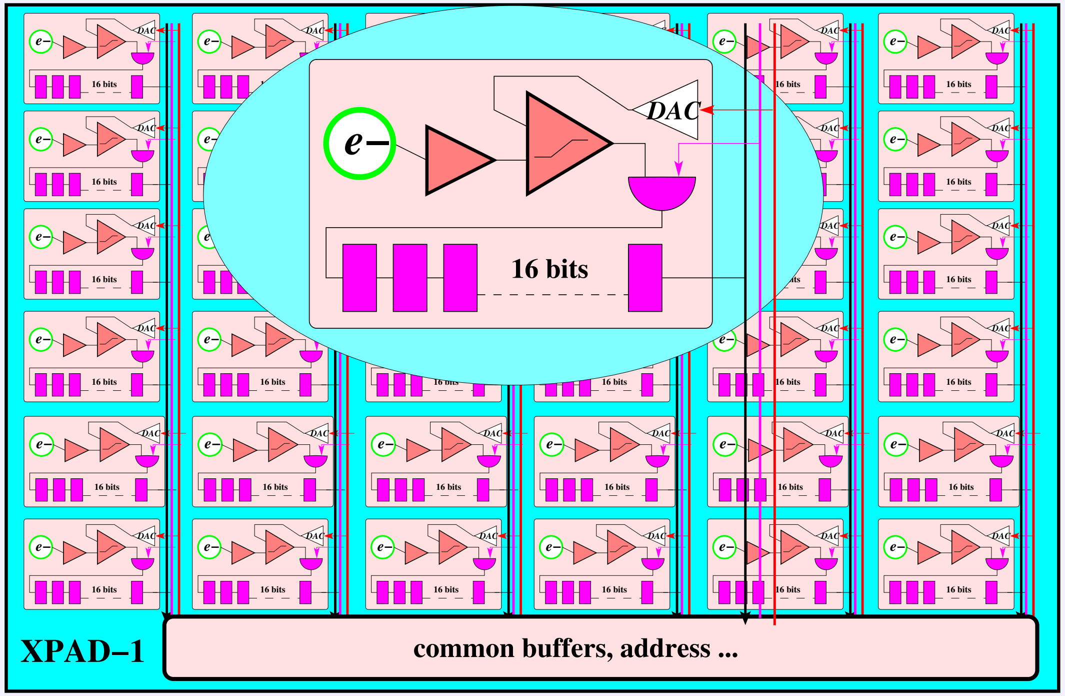
- high resistivity Si

Chips :

- AMS CMOS 0.8 μm
- 24 $\times$ 25 pixel/chip



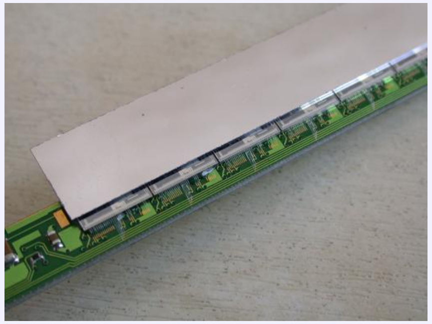




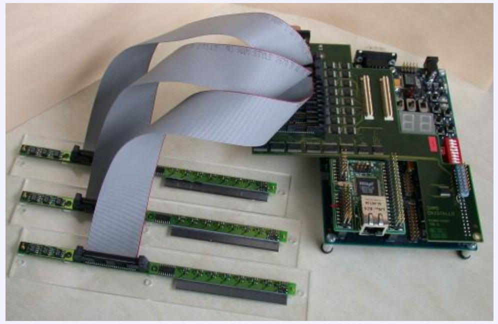
Boudet *et all.*, NIM A510 (2003) 41-44, Berar *et all.*, J. Appl. Cryst. 35 (2002) 471-476

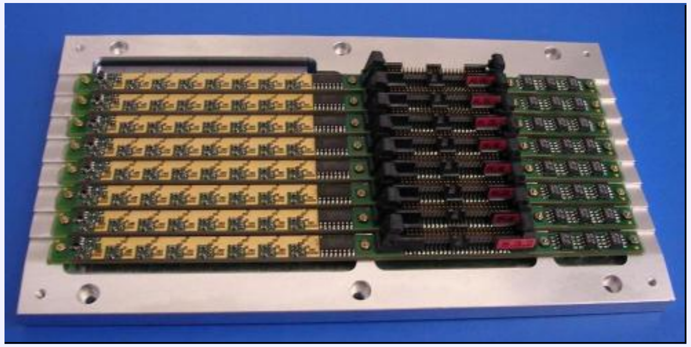
XPAD2 detector : 8 modules $\times$ 8chips

New diodes of 500μm Si thick $\rightarrow$ efficiency 78 % @15keV, 21% @25keV

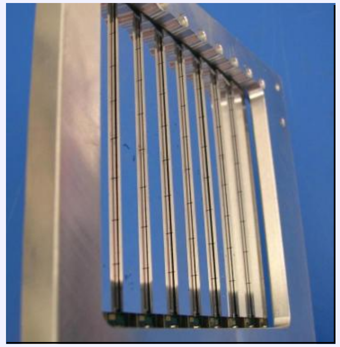


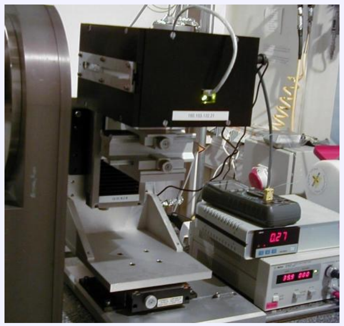
Diode $\rightleftharpoons$ 8 chips of 24 $\times$ 25 pixels
PCB card : drivers and regulators.
Modules $\rightleftharpoons$ acquisition card
Alterra Nios kit + ethernet



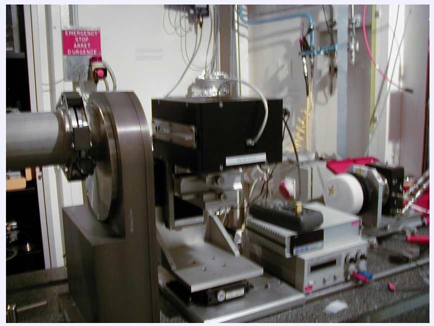


Tiled as close as possible
$\rightarrow$ reduce shading, dead zones.
Metallic holder $\rightarrow$ few μm.
Size : 200 $\times$ 192 pixels
Surface $\approx$ 68 $\times$ 68mm^2.



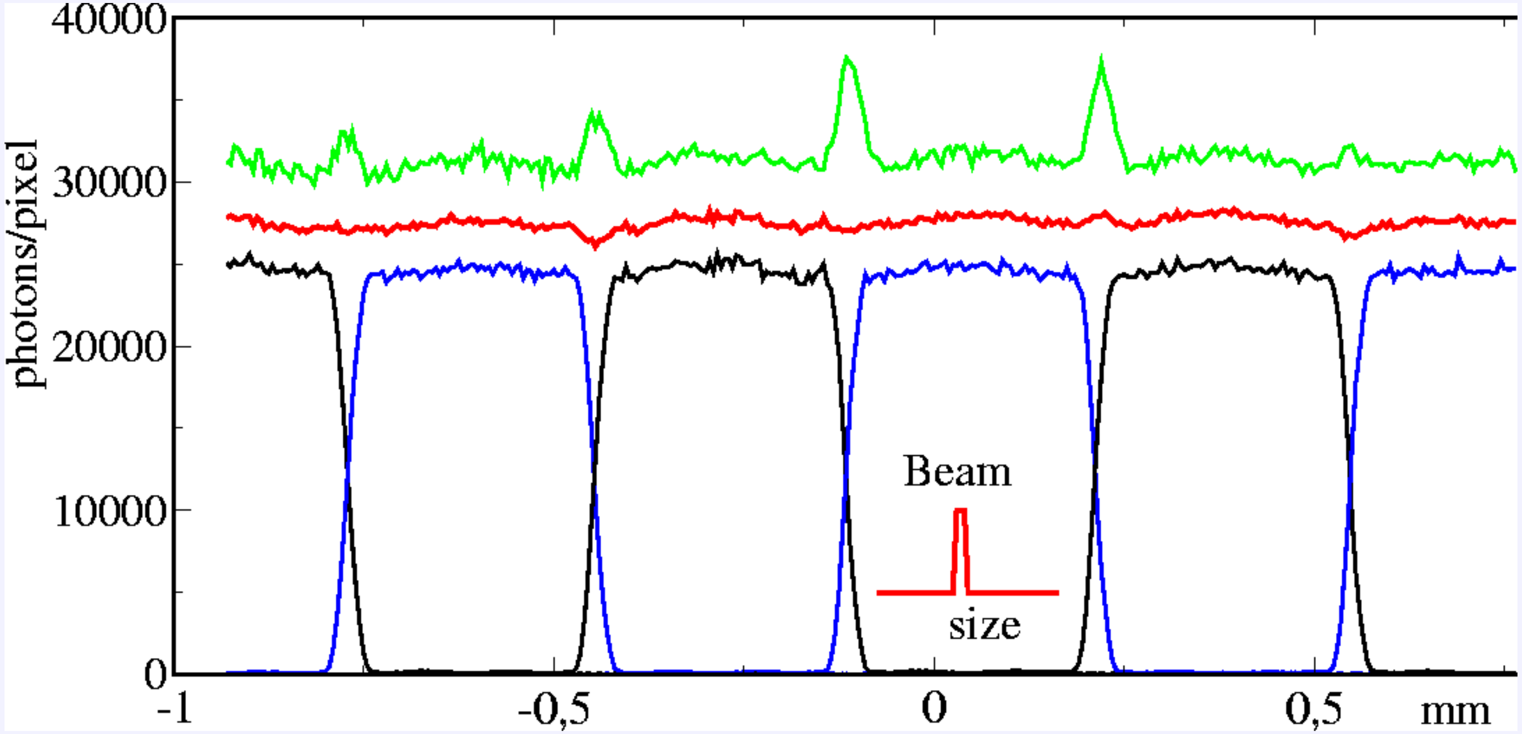


Interface software
developed using LabWindows/CVI
application software moves to Linux.
XPAD prototype at SAXS station.



Spatial resolution

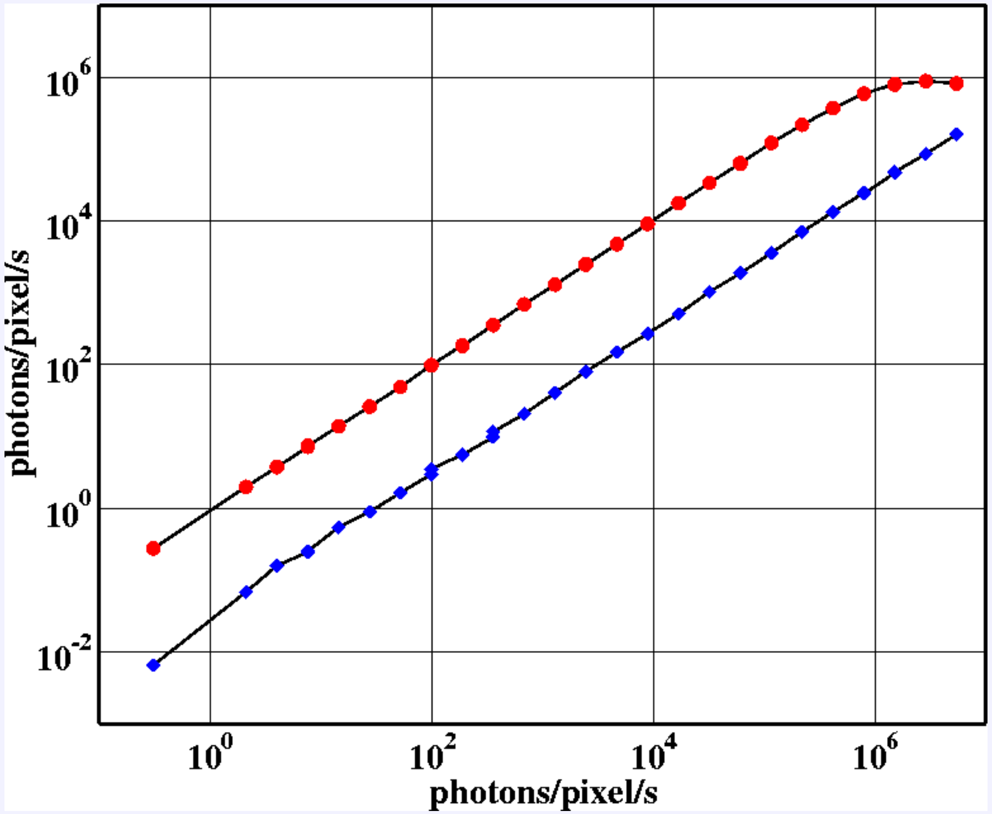
As the diode is common to pixels belonging to the same chip, some charge sharing may occurs between adjacent pixels. Measurements show that the charge sharing occurs on $\approx 60\,\mu m$.

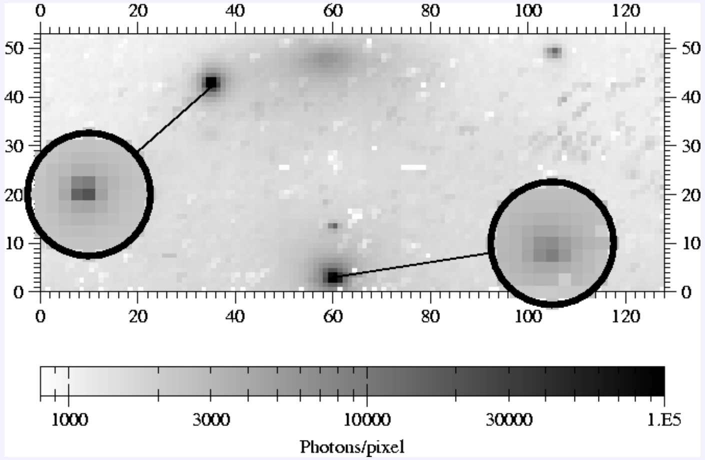


A flat field can be obtained when energies edge is perfectly adjusted in each pixel (red). In case of too low edges, this share sharing create some over-counting at pixel borders (green).

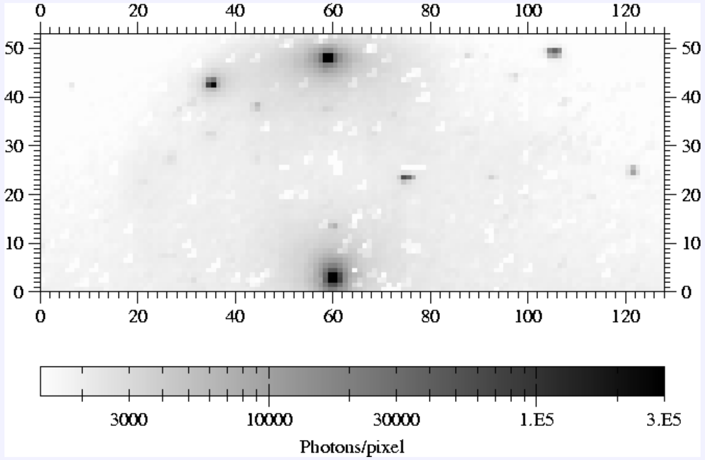
Dynamical range (XPAD1)

The curves show the counts in two adjacent pixels as a function of the incoming flux on the more exposed pixel using XPAD chips.



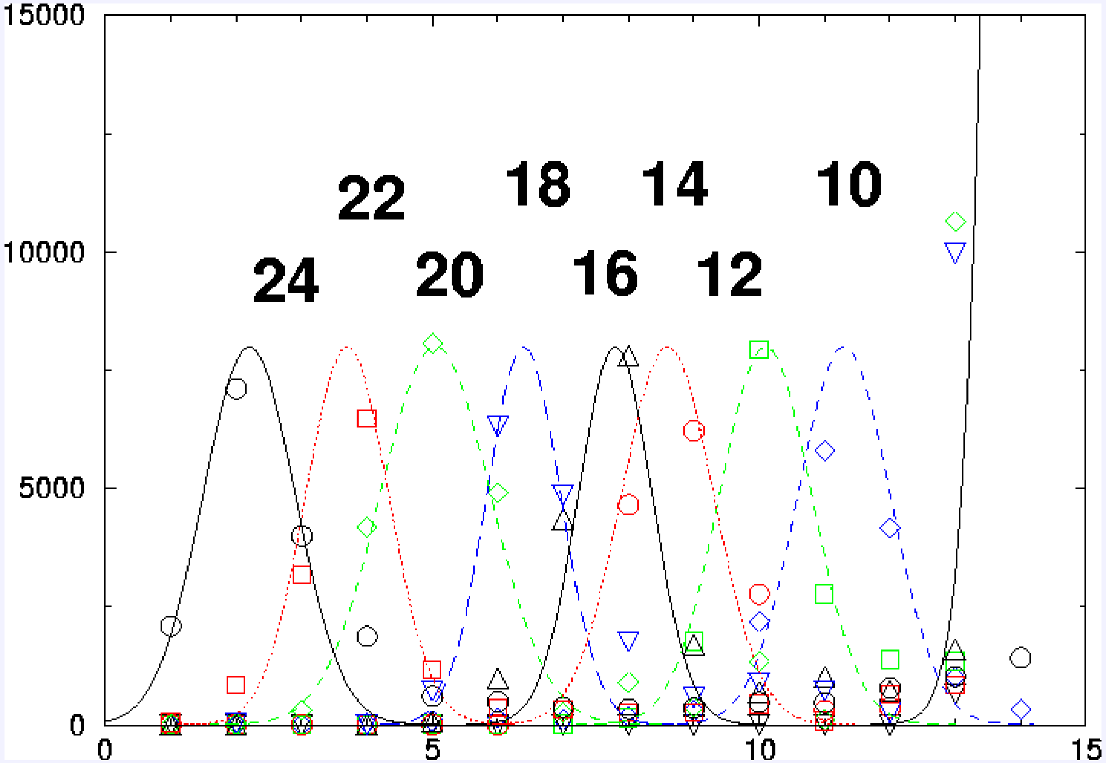


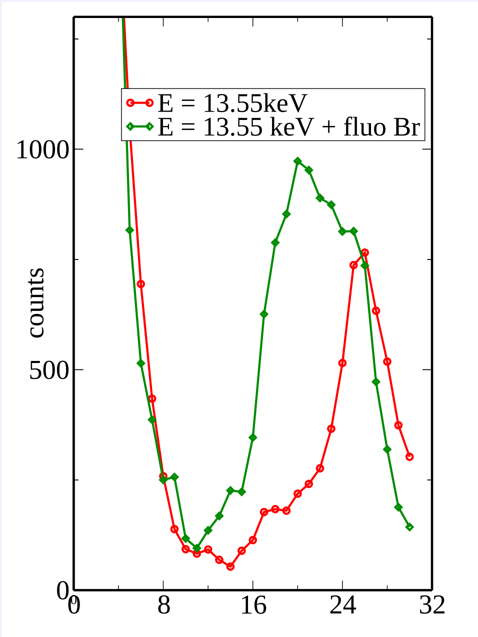
Diffusion (above) of a CdYb icosahedral quasicrystal and associated rotation image (below).



Energy resolution

The conversion of incoming photons in silicon leads to a charge proportional to the incoming energy. The XPAD chip energy resolution is near $1\,keV$.

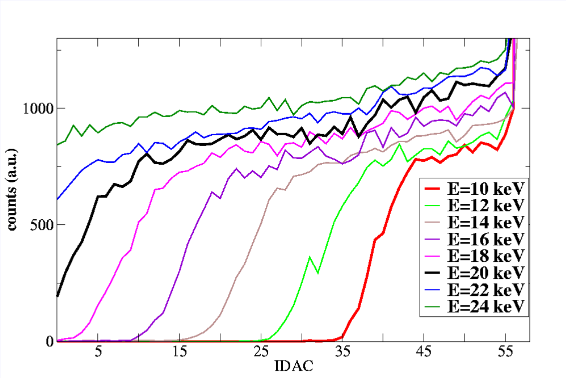




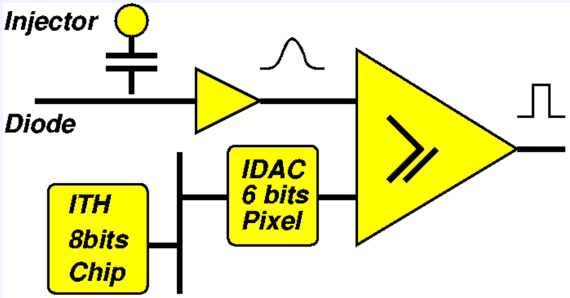
Measured counts as a function of the threshold for the diffusion in of a Br solution on both sides of Br absoption edge.

Pixel threshold register : 4 bits (XPAD1) $\rightarrow$ 6 bits (XPAD2)

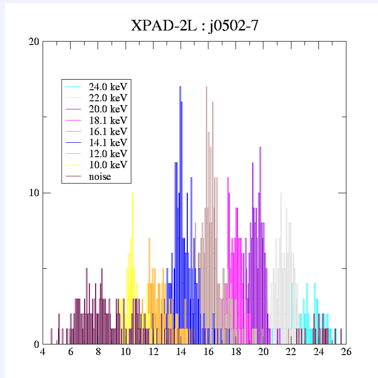
XPAD2 calibration and dispersion (1)



- *beam* E_x : monochromatic flat scattering (amorphous), noisy, time expensive
- *injection* E_{inj} : simulate the beam, quich and easy but need calibration
- Each pixel is described by : $C, \alpha, \beta, E_{inj}(noise)$
  $E_x = CE_{inj} = \alpha(I_{th}) + \beta(I_{dac})$
  $E_x(noise) = CE_{inj}(noise)$
- $\approx$ $4\,10^4$ pixels $\Rightarrow$ automatic configuration/calibration procedure.

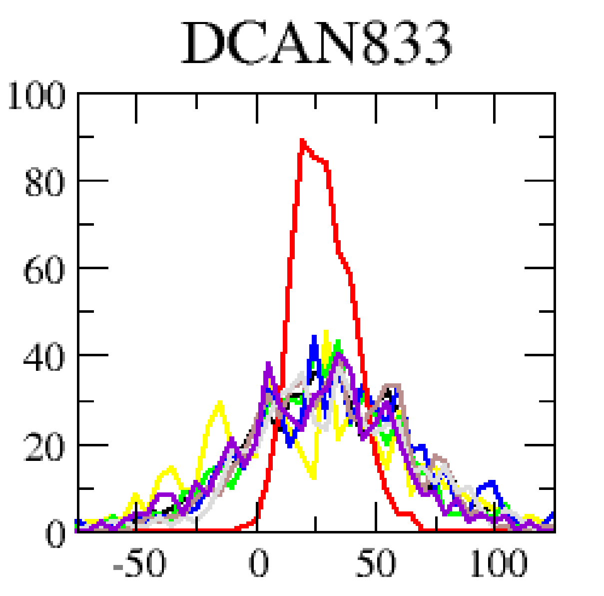


Knowing then these characteristics :
the setup of each chip at a given energy E can be defined as the value of the chip common threshold level I_{th} for which most of the pixels can be fine tune, $I_{dac} \in [0, 63]$.



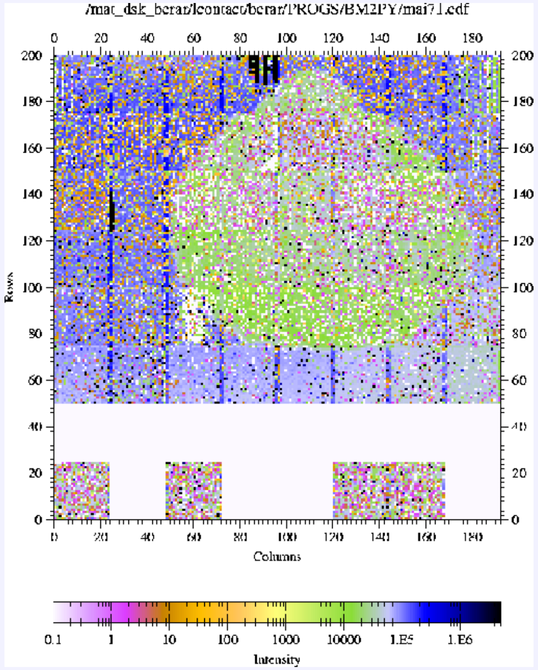
XPAD2 calibration and dispersion (2)

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

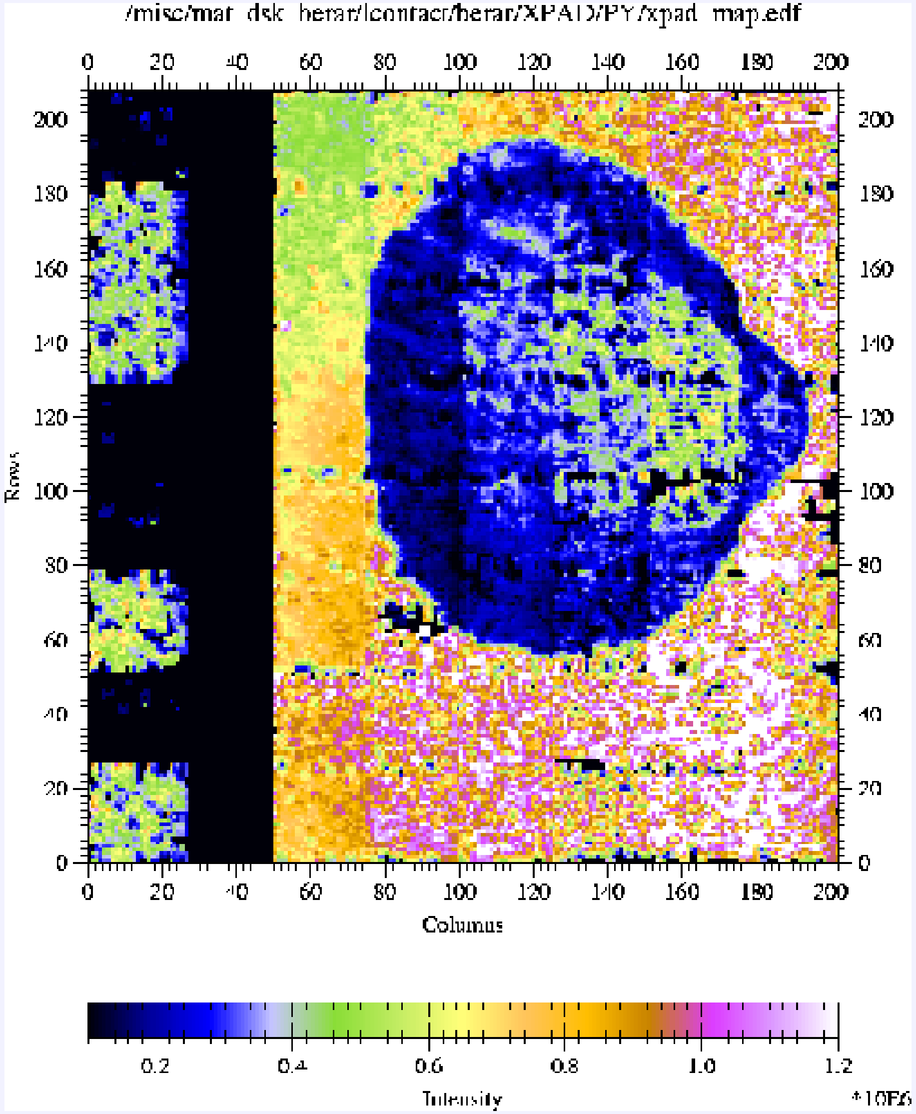


However, even if all the pixels are not perfectly set, the XPAD2 detector appears as a usefull tool for recording new data in SAXS and diffraction on a synchrotron beamline in the range 15 - 25 keV.

XPAD2 calibration and dispersion (3)



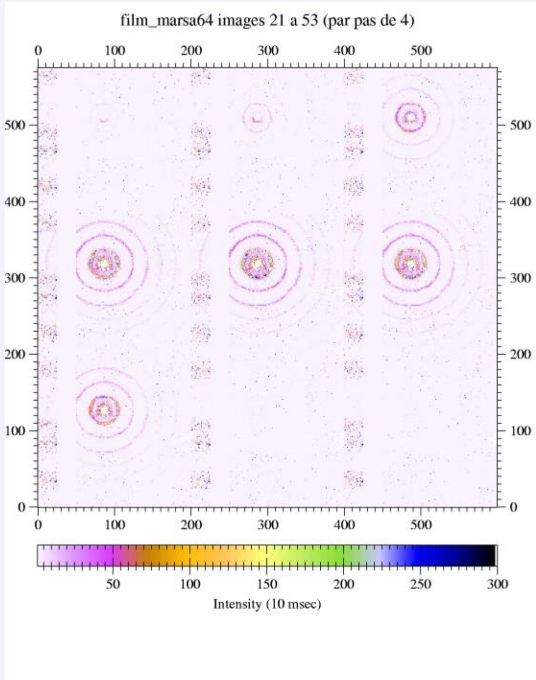
Correcting raw image from surface,... and merging 2/4 images lead to reasonable one.

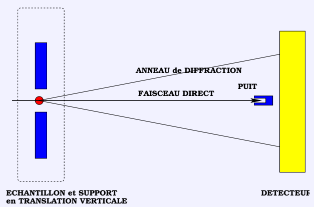


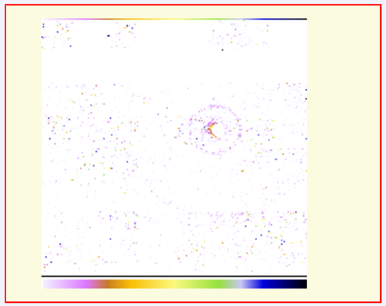
Kinetics potentiality of XPAD2

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 $>= 10ms$



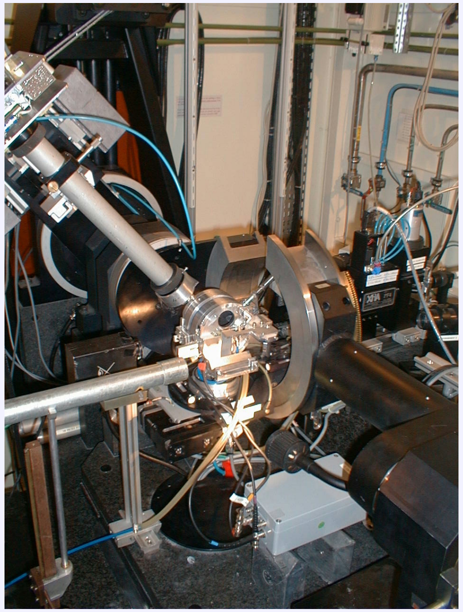


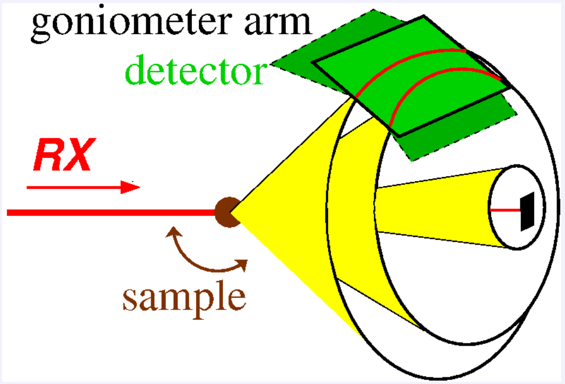


Images of 10 ms each taken off a 2s movies showing difraction while the sample crosses the beam at D2AM SAXS camera.

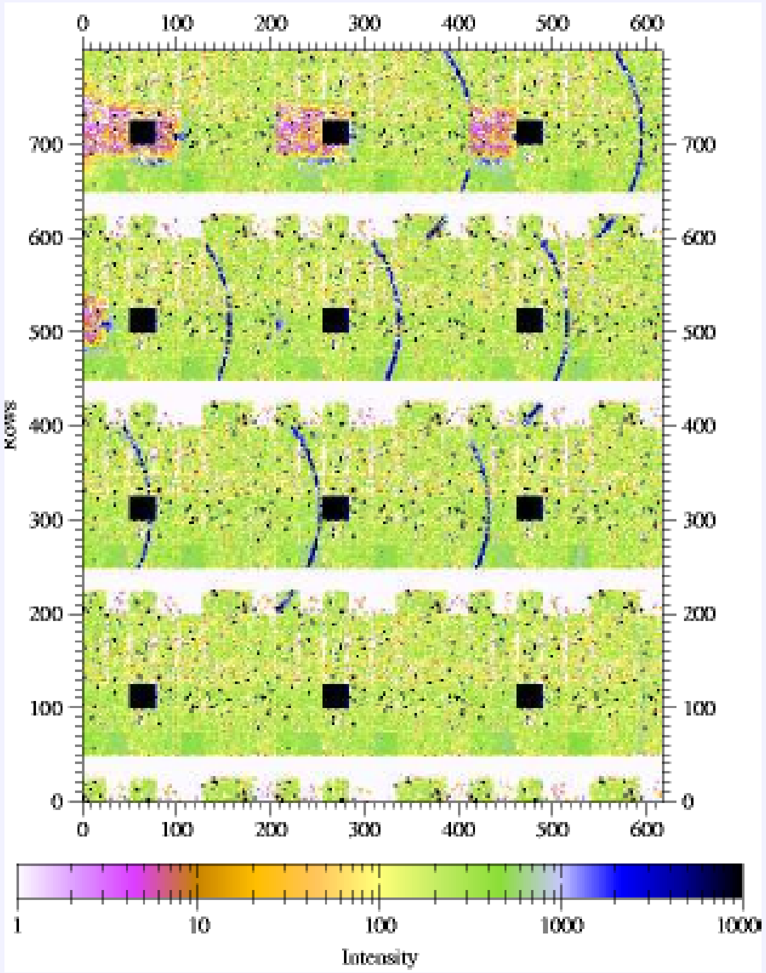
Powder diffraction application (1)

- Diffraction along cones
- Data redondancy with 2D detector
- 60^o collected at high resolution
- angular apperture 4^o at $1m$





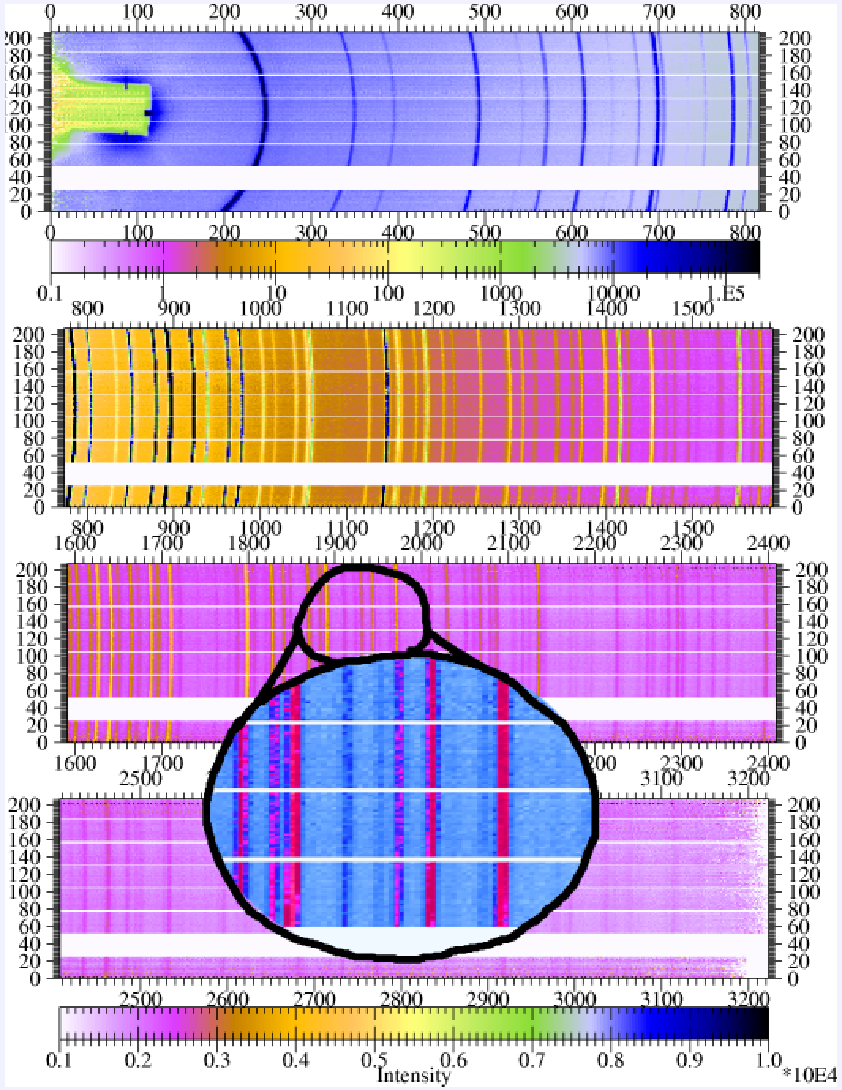
Pipes and slits → 2d-detector.



First raw images with beam stop and one Bragg line.

Powder diffraction application (2)

- Complex pattern of a Zeolite
- Reconstructed Debye-Scherrer film →



Resulting Y counts on pixel p :

$$Y_p = N_p^{-1} \Sigma f_q \, y_{q,i}$$

$y_{q,i}$ counts on image i of pixel q

f_q flatfield of pixel q :

Pixel : q in image i $\rightarrow$ p in merged image

$$q = q(p,i)$$

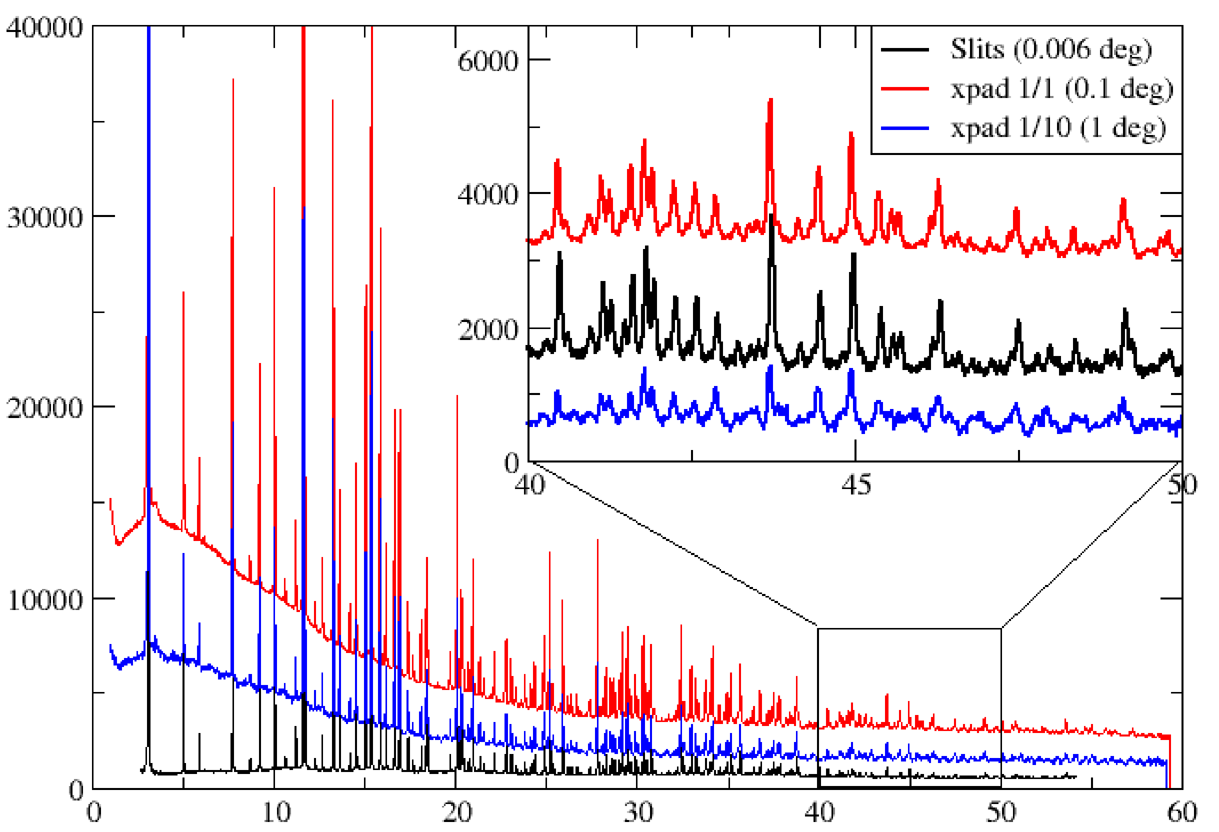
Minimisation of :

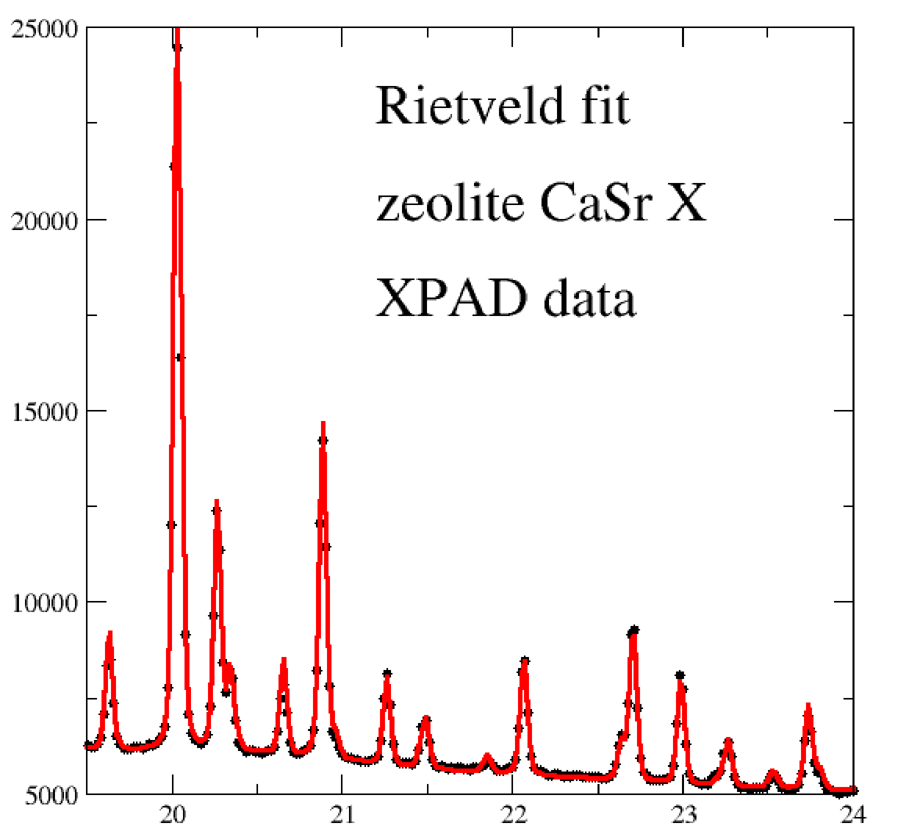
$$\sum_p (Y_p - N_p^{-1} \sum_i f_q \, y_{q,i})^2$$

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

$$\sum_{Ring} (Y_{Ring} - N_{Ring}^{-1} \sum_{p \in Ring} \sum_i f_q \, y_{q,i})^2$$

Powder diffraction application (3)





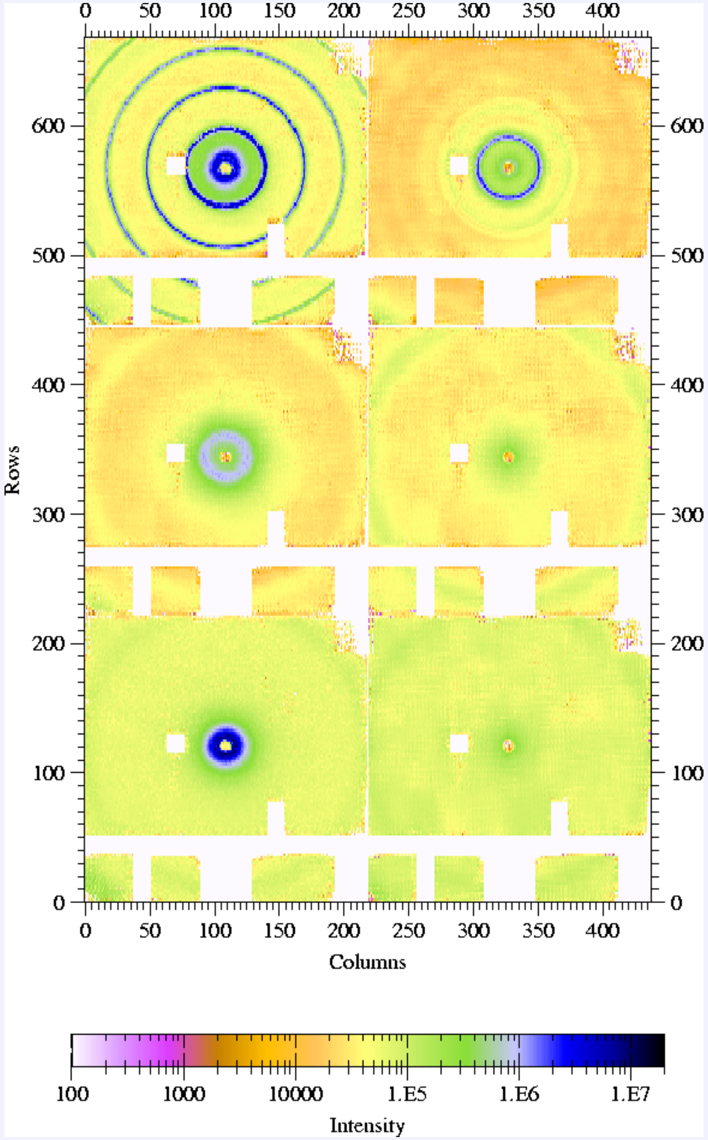
Rietveld method : R_{wp}=8.8%
R_{exp}=4.1% and R_{bragg}=4.4%
Atomic parameters same as conventional
Whole experiment time → 1/20.

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

SAXS application (1)

Scattering of some samples recorded at BM2-SAXS camera using XPAD detector at 20 keV.

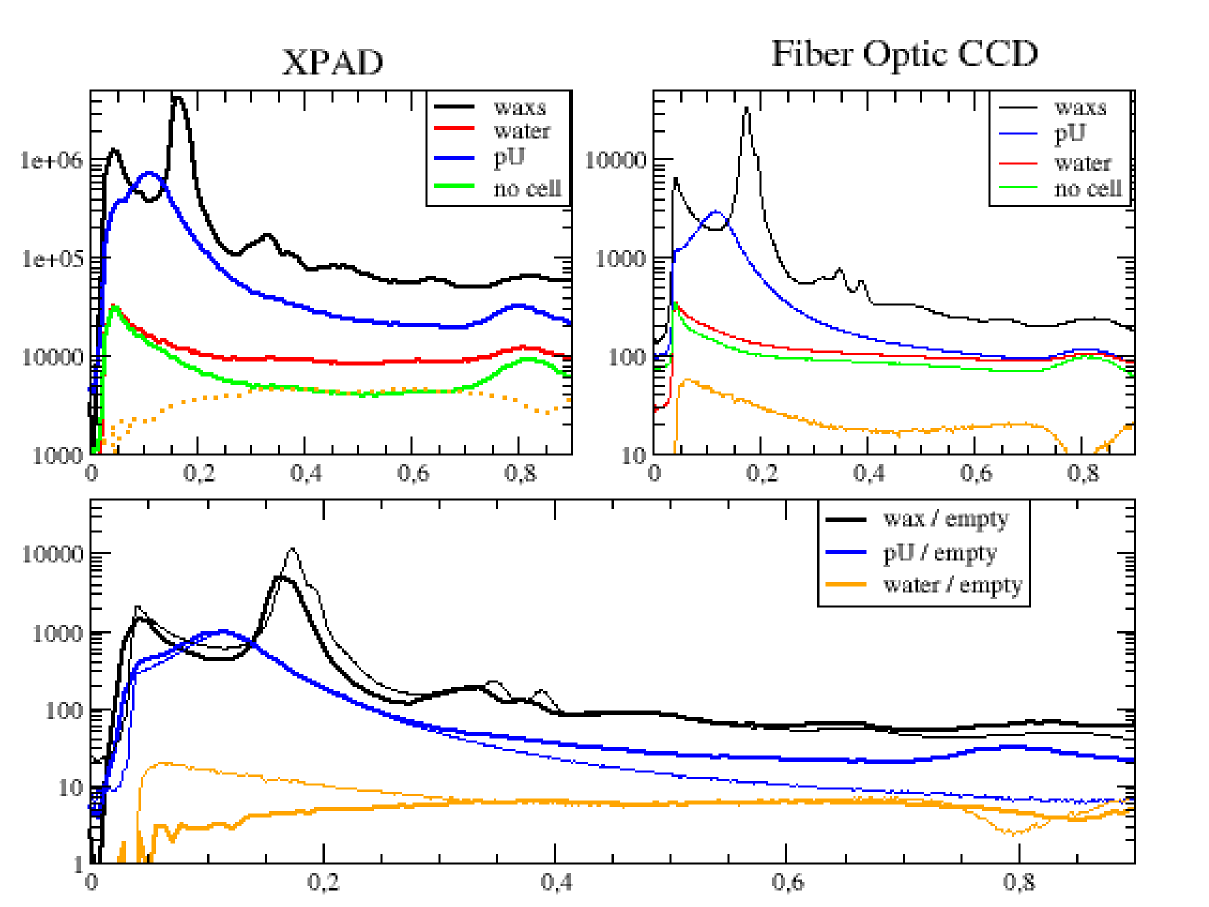
- Ag Behenate
- Bee waxs
- Polyurethan
- Empty cell
- Teflon
- Water (5mm)



SAXS application (2)

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, 50μm pixel size, dark corrected)

from XPAD2 to XPAD3

- Impoving XPAD2 chips in AMS-CMOS 0.8 μm technology ?
- Or design a new XPAD3 using 0.25 μm technology ?

	XPAD2	XPAD3	comments
polarization	both	e^+	dedicated to Si diodes
pixel size	330 μm	125 μm	
chip size	$8 \times 10\, mm^2$	$10 \times 15\, mm^2$	$\rightarrow$ reduce tiling
counting rate	$2.10^6 ph/s$	$2.10^5 ph/s$	similar by surface unit
energy range	(5) $15 \rightarrow 25 keV$	$7 \rightarrow 25 keV$	analog chain modified
pixels/chip	$24 \times 25 = 600$	$80 \times 120 \approx 1.10^4$	
pixels/module	$8 \times 600 \approx 5.10^3$	$\approx 1.10^5$	
pixels/detector	$\approx 4.10^4$	$\approx 5.10^5$	
geometries	8×8 or 2×5	8×8 and ?	

Prototype expected for spring 2006.