XPAD : hybrid pixel detectors for material sciences studies using X-ray synchrotron radiation.

J.-F. Bérar, S. Arnaud, S. Basolo, N. Boudet, P. Breugnon, B. Caillot, J.-C. Clemens, P. Delpierre, B. Dinkespiler, S. Hustache, I. Koudobine, M. Menouni, P. Pangaud, R. Potheau, E. Vigeolas

D2AM/CRG-ESRF Grenoble | Inst. NEEL | CPPM-IN2P3 Marseille | Sync. SOLEIL St Aubin

CENTRE NATIONAL DE LA RECHERCHE SCIENTIFIQUE

Jan. 18th, 2007, Trieste

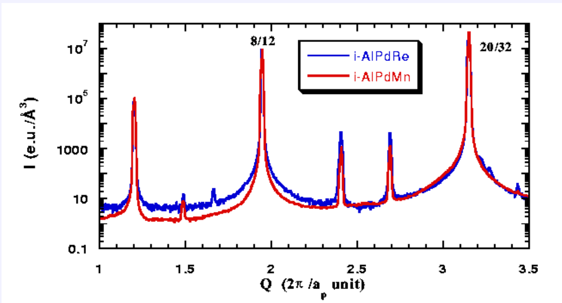
Summary.

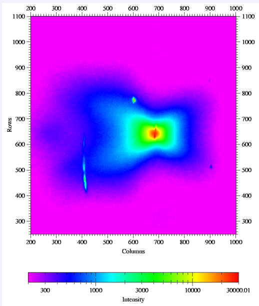
Detectors & material sciences scattering

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 → 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 $0.5\,m \approx 0.01^{\,o}$

slits and photomultiplier :
7 orders of magnitude required
Dynamic extended by **attenuators**, time consuming mapping



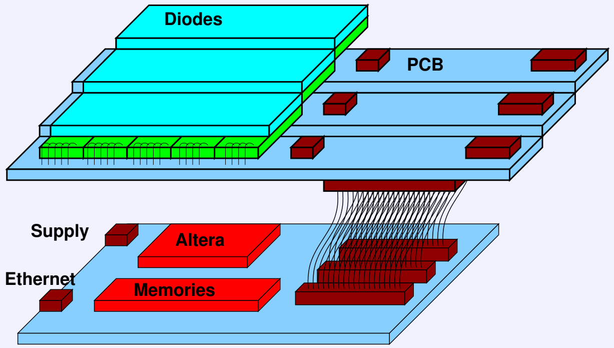


Complex shape of the diffusion around Bragg peak obtained by adding 10 (1000) frames.
Out of peak to avoid blooming effects within CCD.

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

The XPAD project (XPAD1).

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 beamline energy range
pixel size	$330 \times 330 \mu m^2$	mean spot size (1995) : $250 \times 400\, \mu m^2$
exposure time	$1 ms \rightarrow 1000\, s$	kinetics potentiality

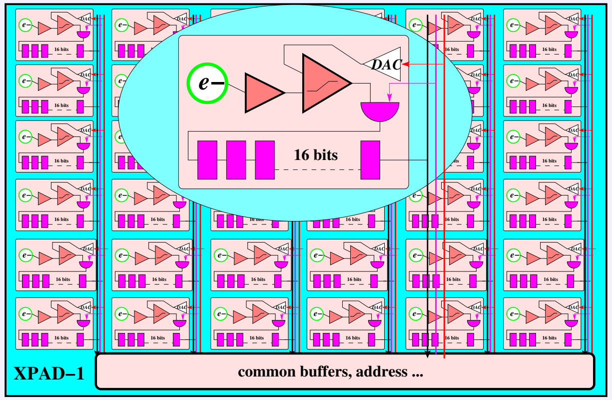


Diodes

- high resistivity Si
- $300 \mu m$ thick

Chips :

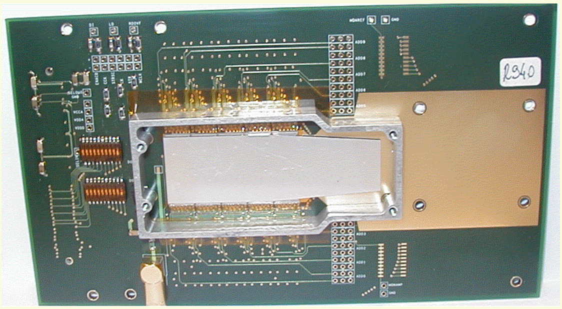
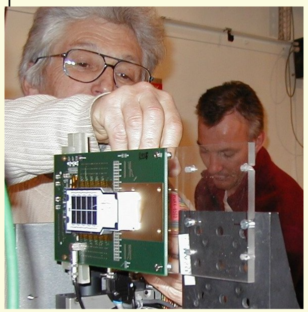
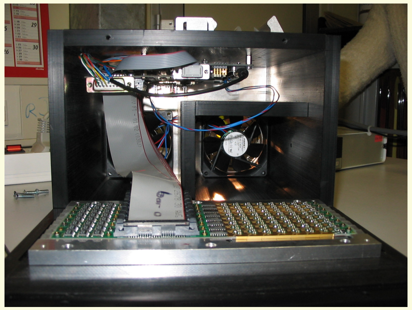
- AMS CMOS $0.8\, \mu m$
- 24×25 pixel/chip



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

XPAD detectors.

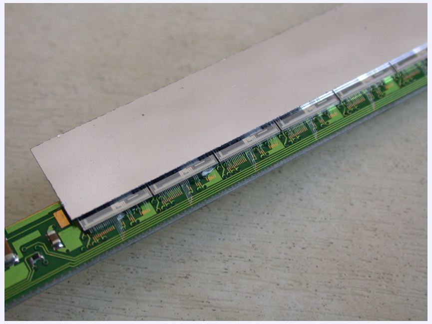
	XPAD1 2001	XPAD2 2003	XPAD3S/C 2006
pixel size	330 x 330 μm		130 x 130μm
foundry	AMS 0.8 μm CMOS		IBM 0.25μm
pixel / chips	24 x 25 pixels		80 x 120 pixels
internal counters	16 bits		12 bits
ext. ovf counters	16 bits		16 bits
energy range	15 to 25 keV		7 to 25 keV
sensor	Si 300 μm	Si 500 μm	
photons ($/pixel$)	$1.10^6 ph/s$	$2.10^6 ph/s$	$2.10^5 ph/s$
time constant (incl. det.)	500 ns	208 ns	*to be measured*
modules	5 $\times$ 2 chips	8 x 1 chips	7 x 1 chips
detector	1 module	up to 8 modules	8 modules or more
electronic	limited	back plugged	
connection	parallel wires	ethernet 100MB	ethernet 1GB?

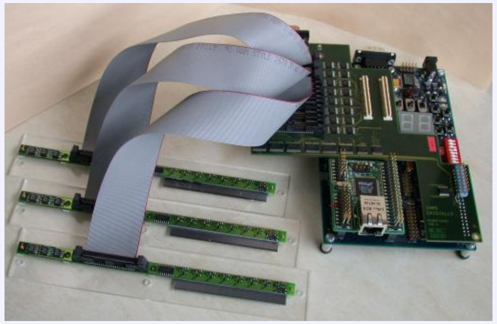

preliminary tests card

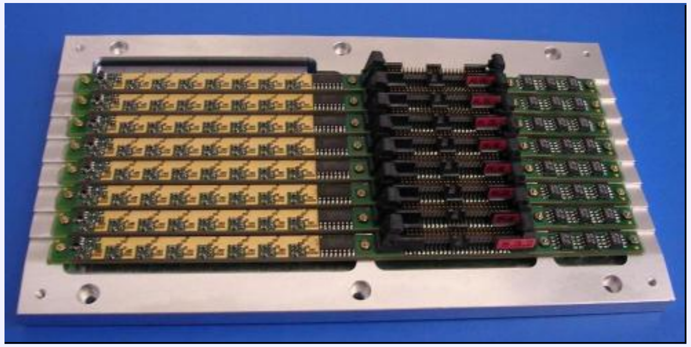
XPAD2 detector : 8 modules $\times$ 8chips

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

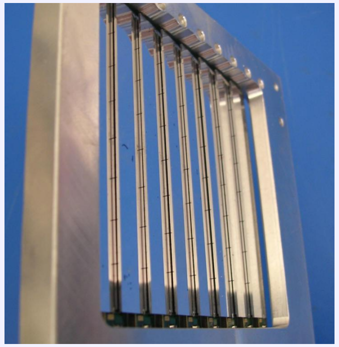


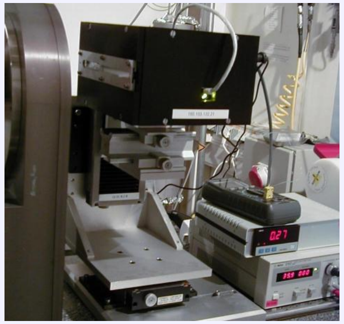
Diode $\rightleftharpoons$ 8 chips of 24 $\times$ 25 pixels
PCB card : drivers and regulators.
Modules $\rightleftharpoons$ acquisition card
Alterra Nios kit + ethernet



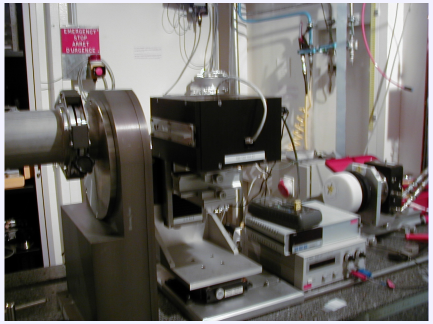


Tiled as close as possible
$\rightarrow$ reduce shading, dead zones.
Metallic holder $\rightarrow$ few μm.
Size : 200 $\times$ 192 pixels
Surface $\approx$ 68 $\times$ 68mm^2.



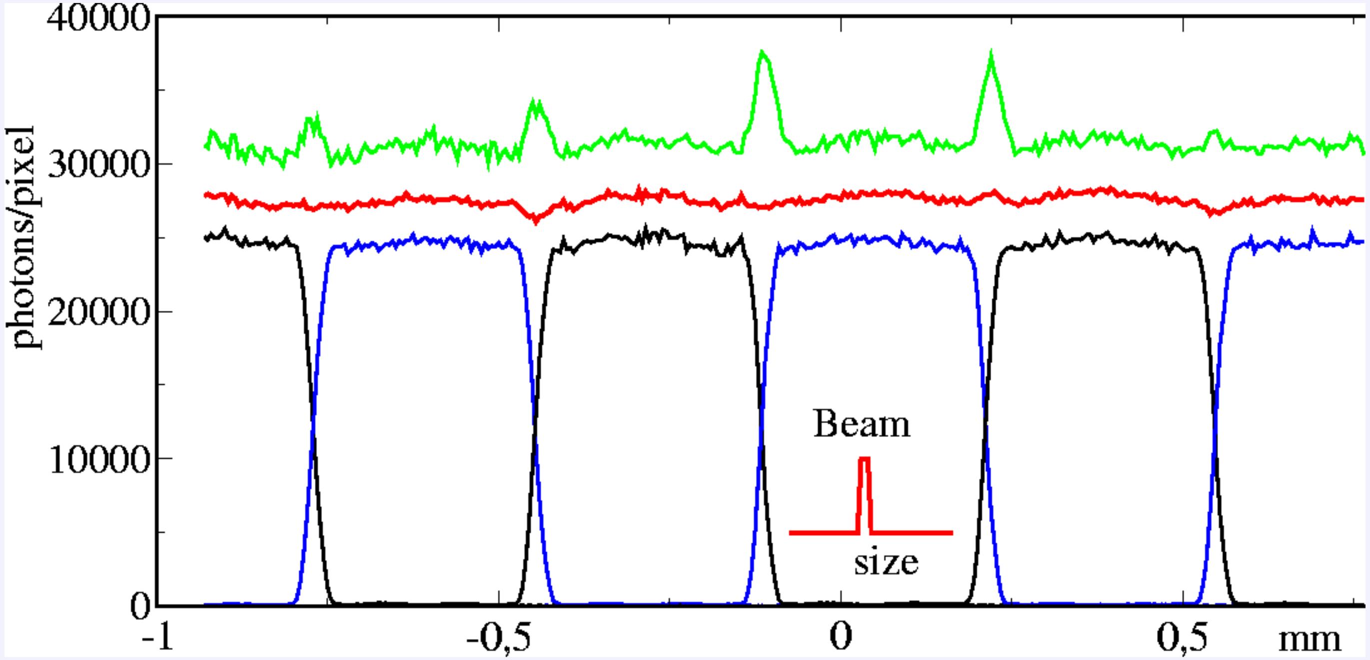


Interface software
developed using LabWindows/CVI
application software moves to Linux.
XPAD prototype at SAXS station.



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 μm.



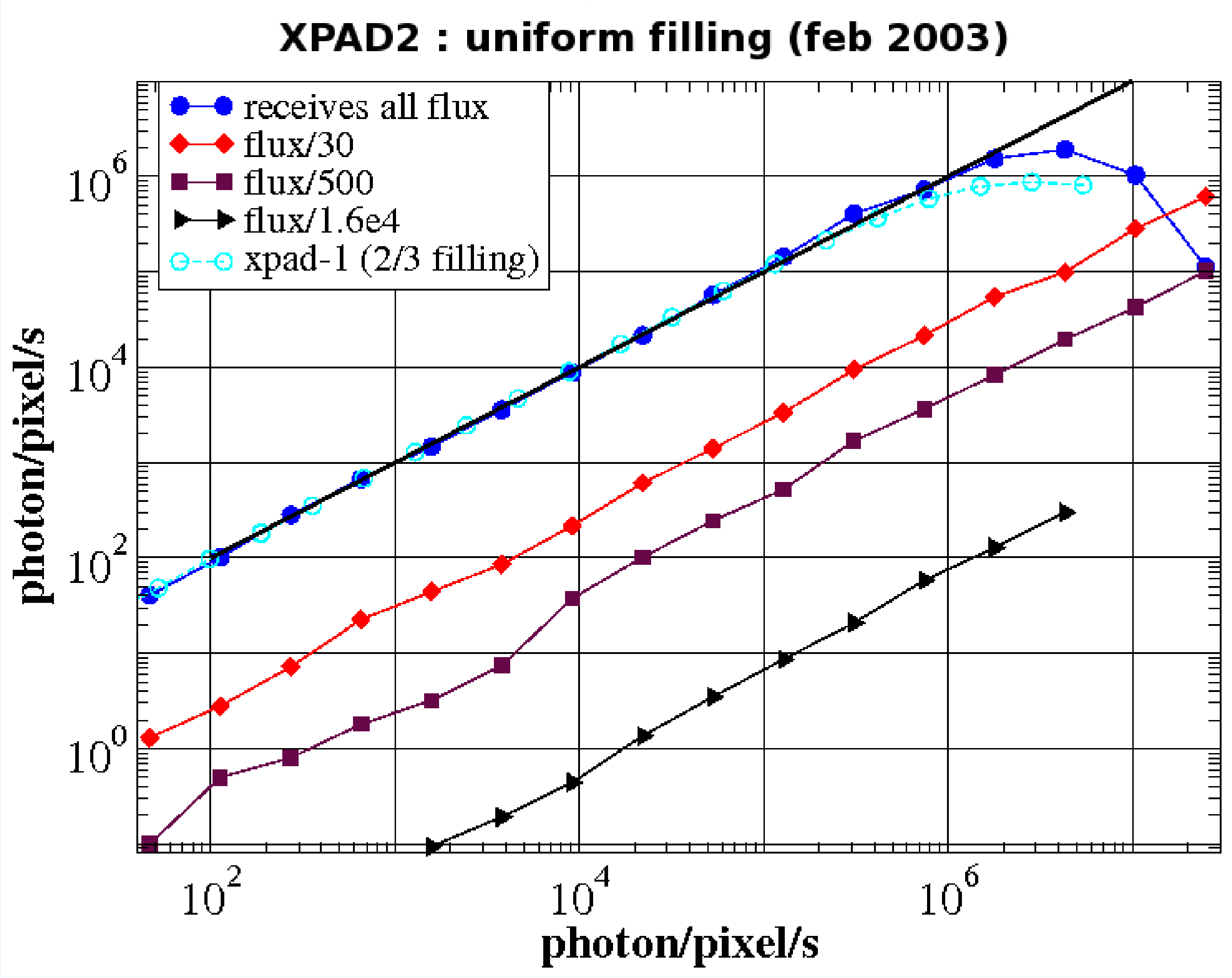
Too low edges
⇒ overcounting on pixel borders

Flat field
⇒ energy edges perfectly adjusted at $E_0/2$

- no dependance found from diode bias polarisation
- need to be checked with XPAD3 smaller pixels

Dynamical range

Counts in adjacent pixels as a function of the incoming flux.

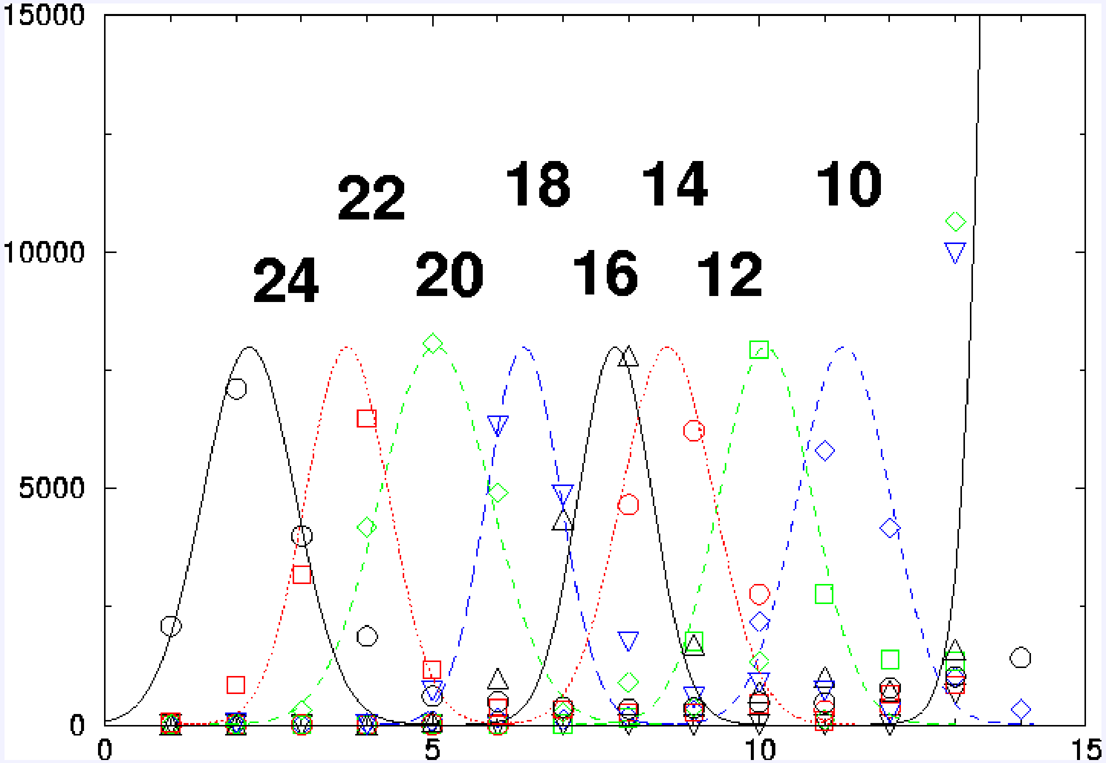


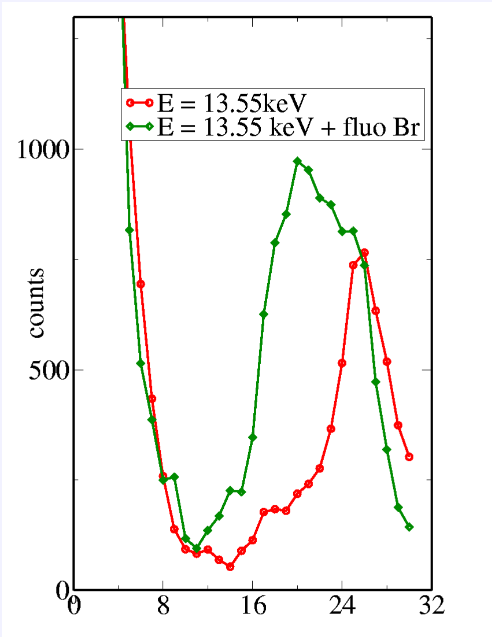
No blooming effect

- Overall dead time $\approx 0.21\mu s$
- Xpad chip dead time $< 0.10\mu s$

Energy resolution

The conversion of incoming photons in silicon leads to a charge proportional to the incoming energy. The XPAD2 chip energy resolution is near $1\, keV$.

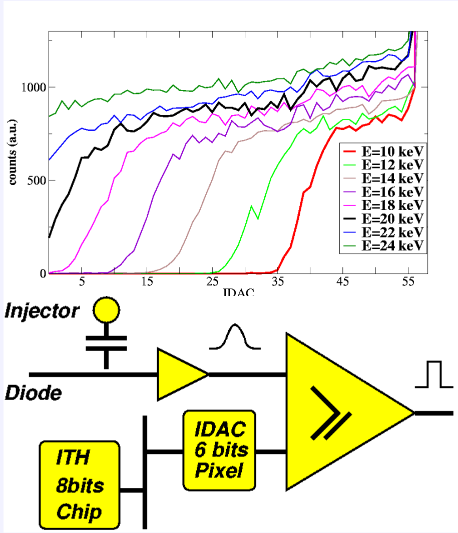




Measured counts as a function of the threshold for the diffusion of a Br solution on both sides of Br absoption edge.

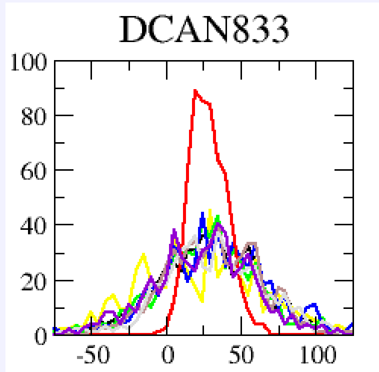
Pixel threshold register : 4 bits (XPAD1) → 6 bits (XPAD2)

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 : $C, \alpha, \beta, E_{inj}(noise)$
  $E_x = CE_{inj} = \alpha(I_{th}) + \beta(I_{dac})$
  $E_x(noise) = CE_{inj}(noise)$
- $\approx 4\,10^4$ pixels $\Rightarrow$ automatic configuration/calibration procedure.
  $\Rightarrow$ chip common threshold I_{th} and pixels I_{dac}.

- 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 $< 15\%$

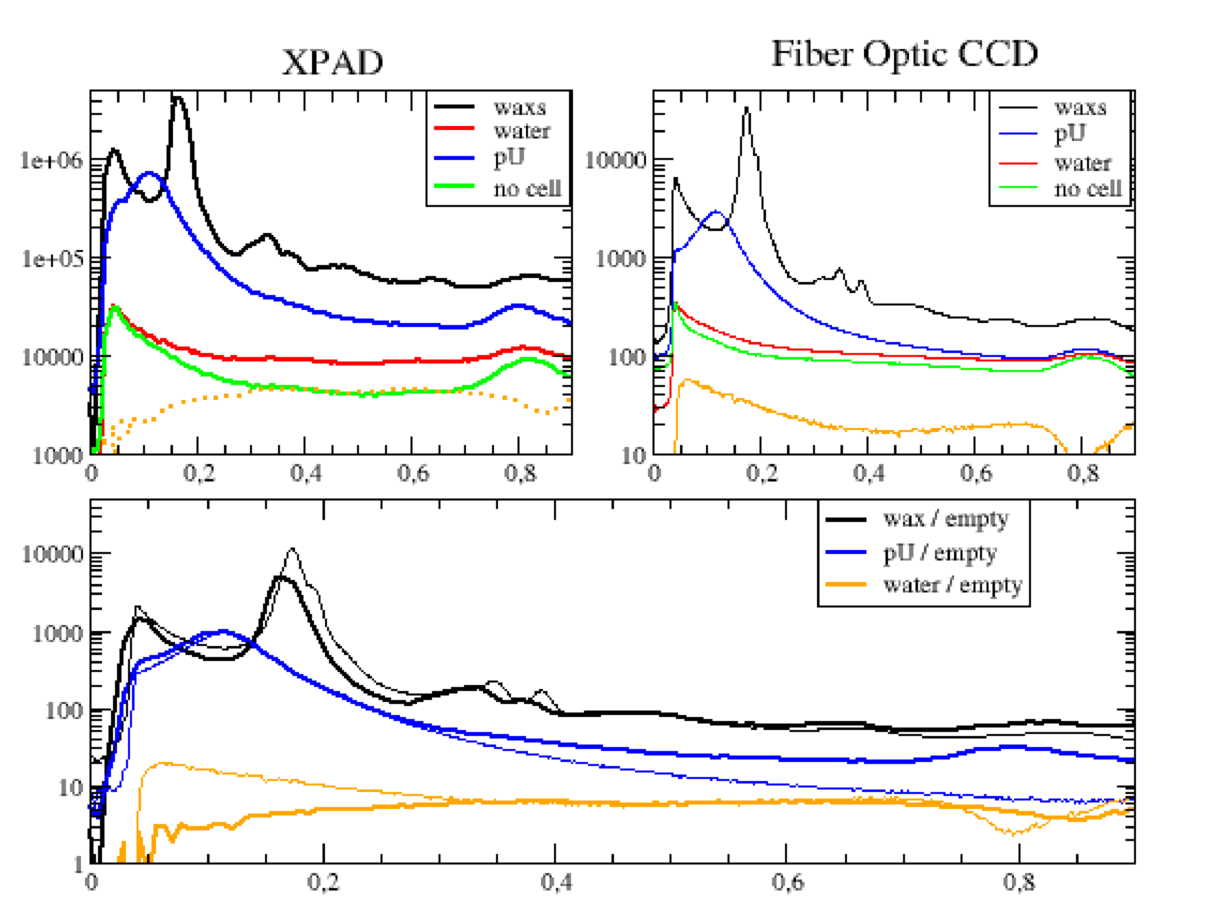


However, even if all the pixels are not perfectly set, the XPAD2 detector appears as a usefull tool for recording new data in SAXS and diffraction on a synchrotron beamline in the range 15 - 25 keV.

SAXS application

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 ...)

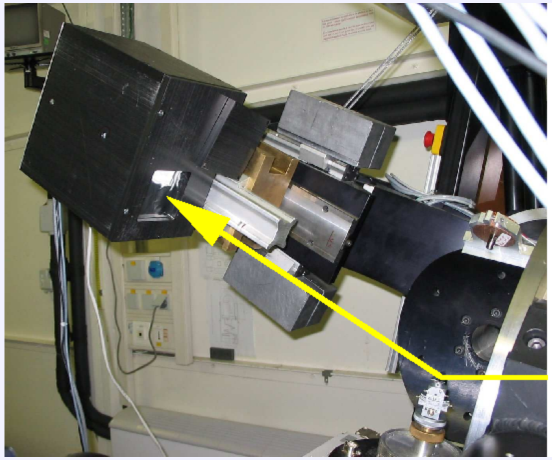


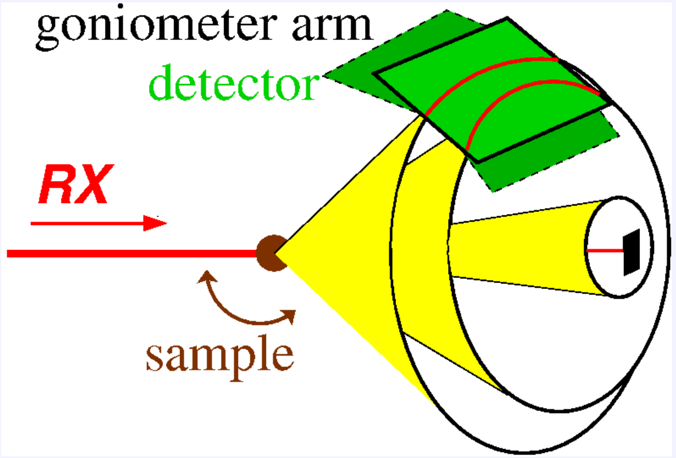
* PI-SCX-1300, Roper Scientific (EEG 1340x1300, 50μm pixel size, dark corrected)

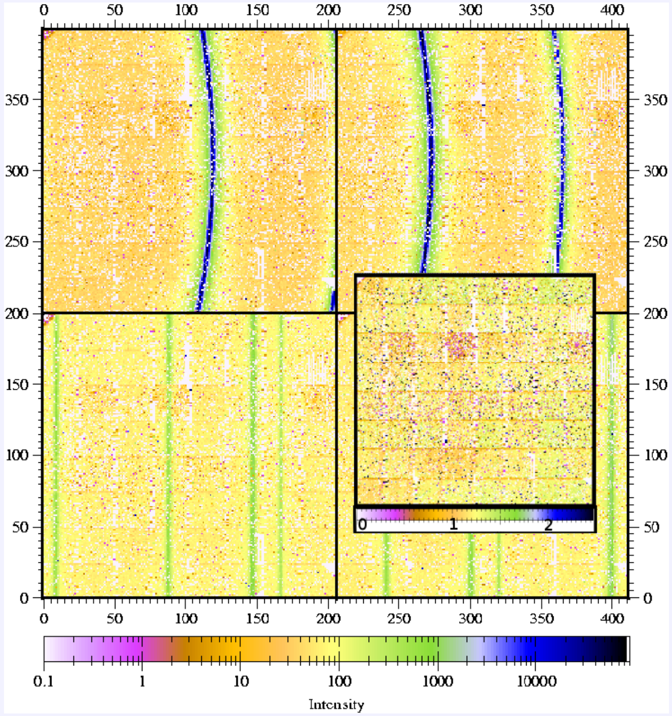
Powder diffraction application

Scintillator and slits → 2d-detector.

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







With 0-D detector pipes and slits remove diffuse scattering, background level partly removed with conic pipes on 2d-detector.

Powder diffraction application (2)

Resulting counts Y on pixel p :

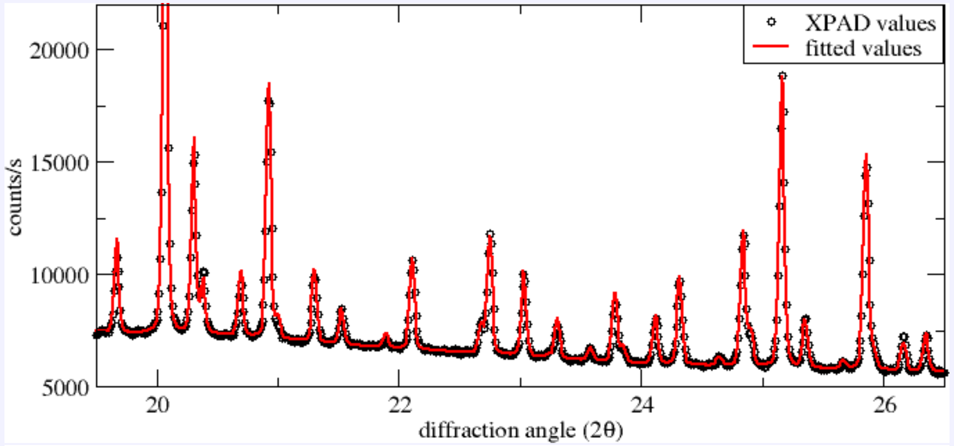
$$Y_p = N_p^{-1} \Sigma f_q \, y_{q,i}$$

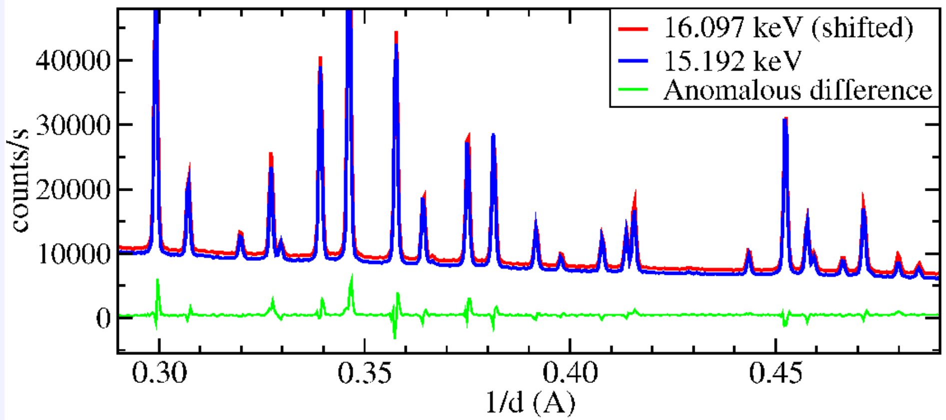
$y_{q,i}$ counts on image i of pixel q,
f_q flatfield of pixel q :

$q \in Image_i \;\rightarrow\; p \in Image_{merged} \;:\; q = q(p,i)$
Minimisation: $\sum_p (Y_p - N_p^{-1} \sum_i f_q \, y_{q,i})^2$

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

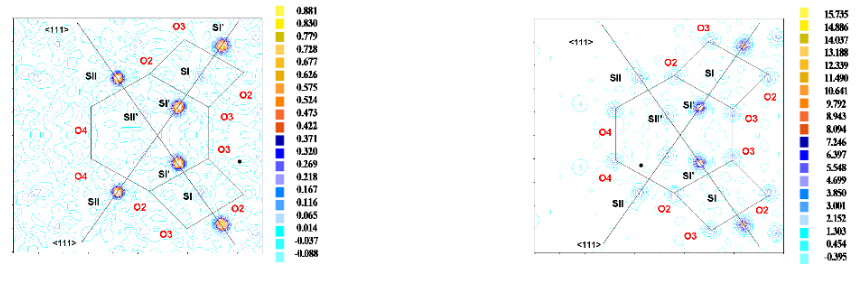
$$\sum_{Ring} (Y_{Ring} - N_{Ring}^{-1} \sum_{p \in Ring} \sum_i f_q \, y_{q,i})^2$$





location a few Rb cation (≈2%) in complex Rb X-zeolite

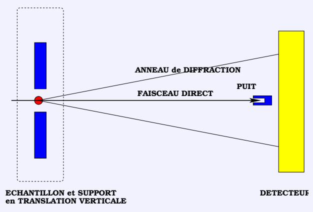
Basolo *et all.*, J.Synch. Rad.14 (2007) 151-157,

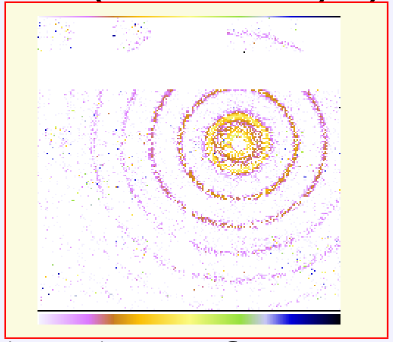


Kinetics potentiality of XPAD2

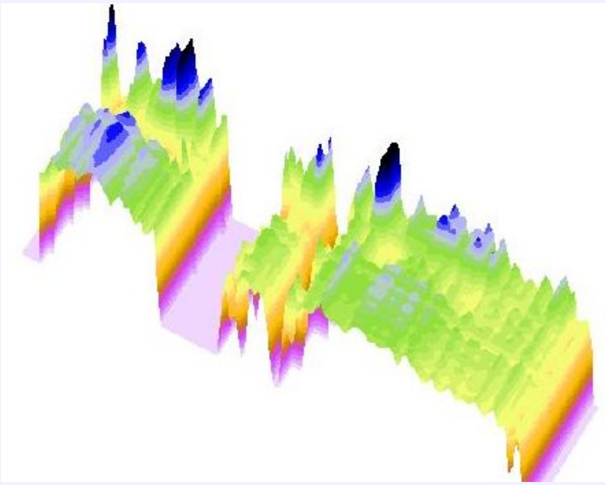
Whole electronic designed to allow kinetics studies (ms range)

- 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 of a 2s movies showing diffraction while the sample crosses the beam at D2AM SAXS camera.

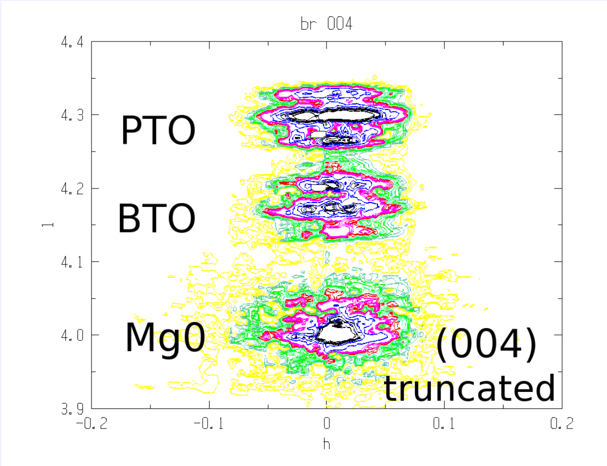


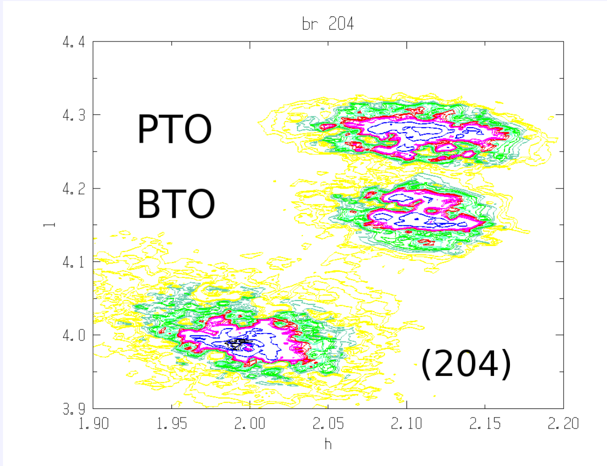
The quench of $Al_{2x}Ca_yO_{3x+y}$ ceramics can lead to vitrous or crystalline oxides. The transition between the liquid state and the cristalline one occurs in less than 20ms and may exhibit some transient phases. Data collection is limited by the cell aperture, which has been designed for linear detector, diagram reconstructed from a few frames of 20ms around crystallisation.

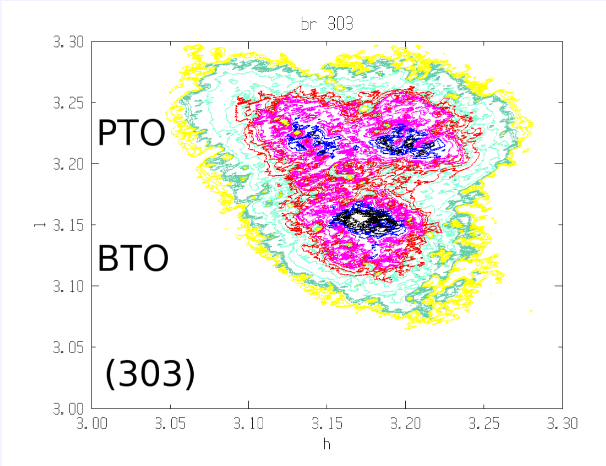
Multilayers : Ferroelectric superlattice

27 (17 PbTiO3,17 BaTiO3) superlattice / MgO :
large lattice mismatch → in-plane polarization → tetragonal distortion.

Physical behaviour of such compounds is primarily dependent on their epitaxial crystalline quality, their composition and their structural perfection.







Out of plane : strain / chemical — In plane : 2 PTO domains tetragonal distortion

The reciprocal maps are recorded scanning the XPAD detector and rebuilt from the collected reciprocal slices. Compared to standard data collection the time can be reduced by 100. Intensity on substrate peak can reach $10^9 \nu/s$!

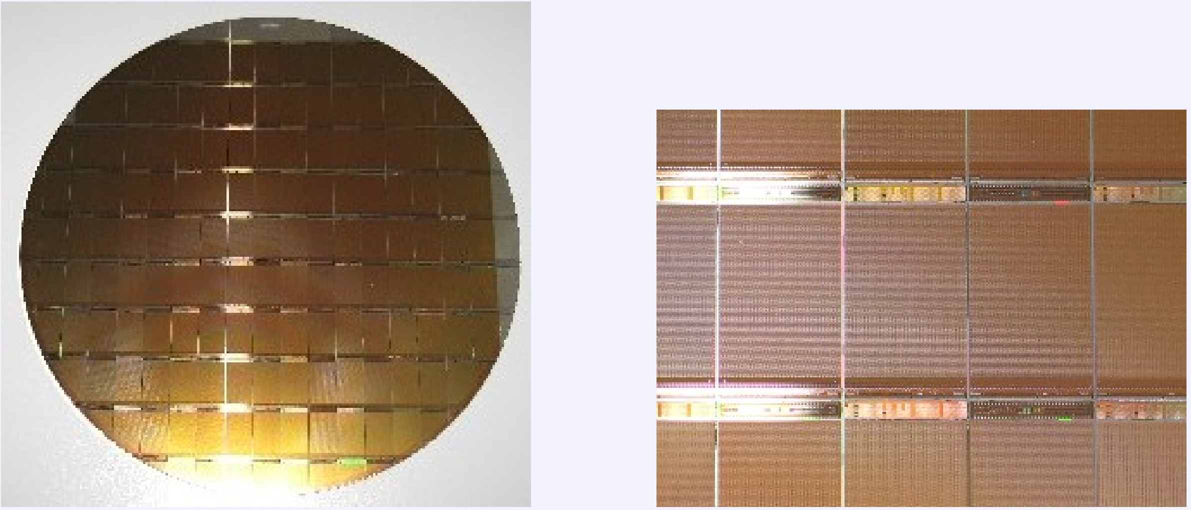
At the time, one major limitation is related to the software needed to map data collected on images to reciprocal lattice with the needed accuracy.

XPAD3 design

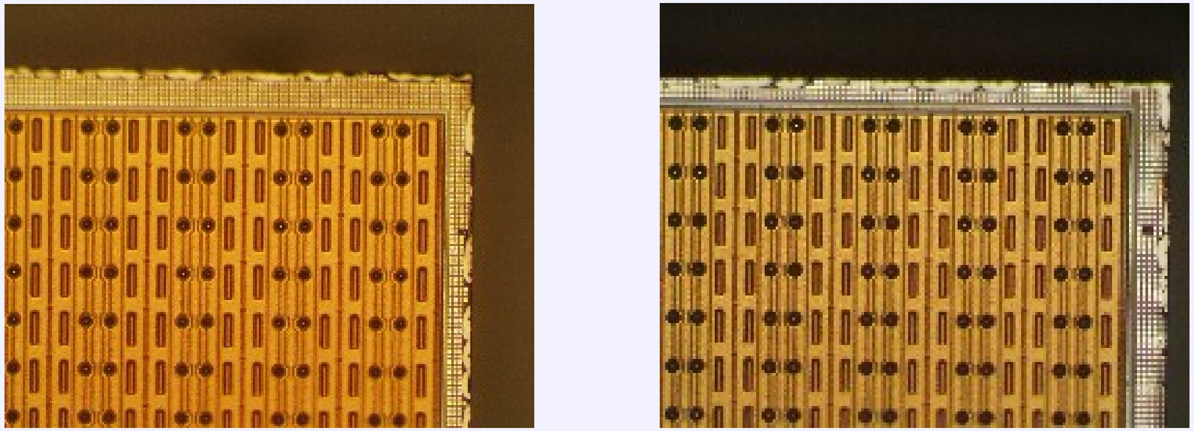
- Obsolescence of the AMS-CMOS 0.8 μm technology used for XPAD2
- A new XPAD3 using 0.25 μm technology with 25 μm bumps
- New analog chain

	XPAD2	XPAD3S	XPAD3C
polarization	both	e^+ (Si)	e^- (CdTe)
pixel size	$330 \times 330\,\mu m^2$	$130 \times 130\,\mu m^2$	
chip size	$8 \times 10\,mm^2$	$10 \times 15\,mm^2$	
counting rate	$2.10^7 ph/s/pixel$	$2.10^6 ph/s/pixel$ ($\equiv$ count/surface)	
photons rate	$2.10^6 ph/s/pixel$	$2.10^5 ph/s/pixel$ ($\equiv$ count/surface)	
counters (bits)	$16 + 16\,ext$	$12 + 16\,ext$ ($\equiv$ count/surface)	
energy range	(5) $15 \rightarrow 25 keV$	$7 \rightarrow 32 keV$	$7 \rightarrow 60 keV$
energy edges	low level	low level	low and up levels
pixels/chip	$24 \times 25 = 600$	$80 \times 120 \approx 1.10^4$	
pixels/module	$8 \times 600 \approx 5.10^3$	$\approx 7.10^4$	
pixels/detector	$\approx 4.10^4$	$\approx 5.10^5$	
geometries	8×8 or 2×5	7×8 and ...	

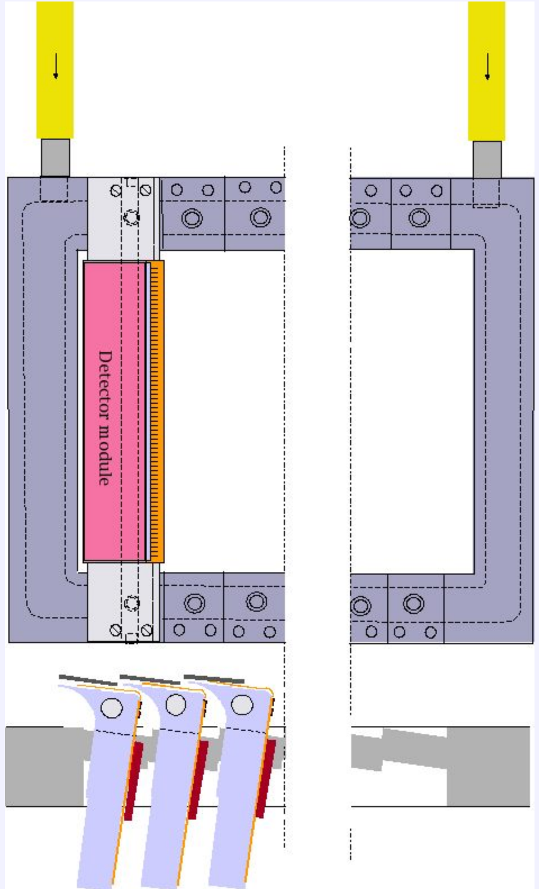
XPAD3 chips



wafer and its cell with the XPAD3S and XPAD3C chips

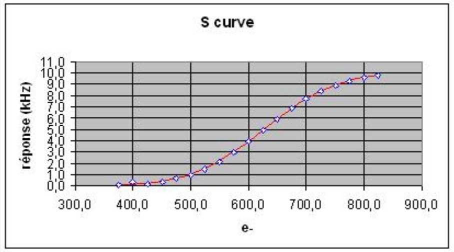


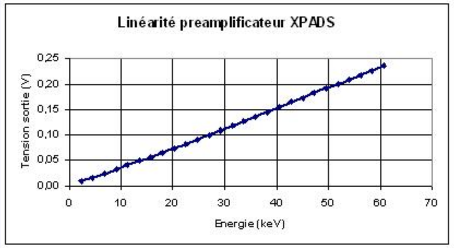
details of XPAD3S with bumps according manufacturer

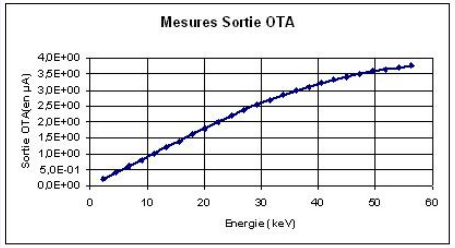


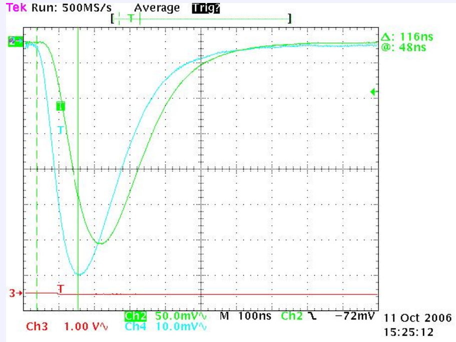
modules assembly in the detector

XPAD3 preliminary results

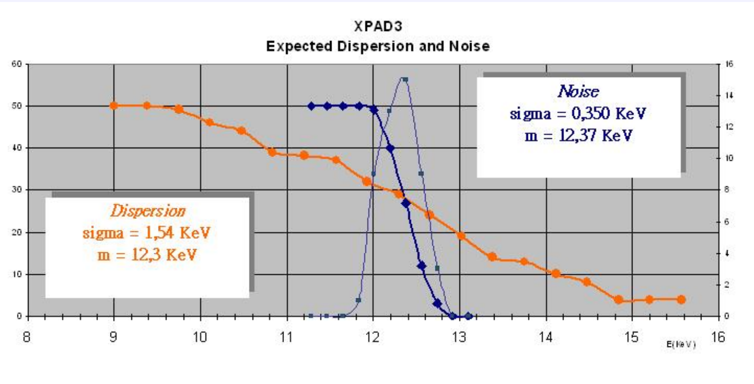


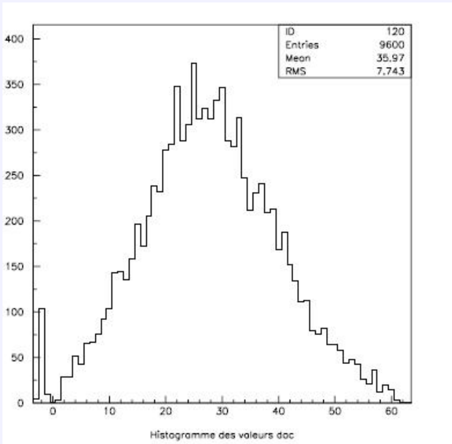


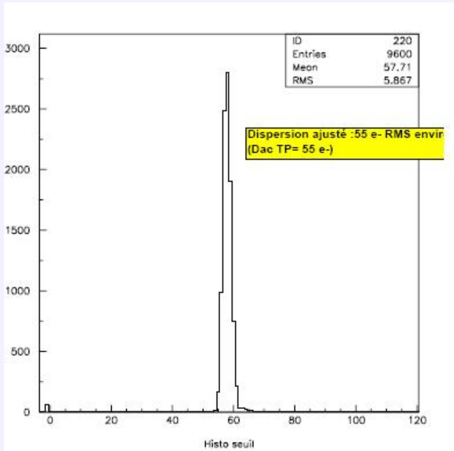




Test of the low edge and of amplifier linearity and response



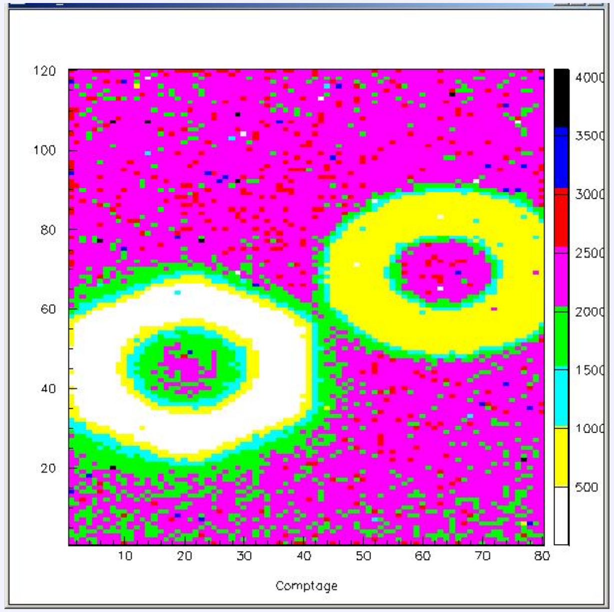


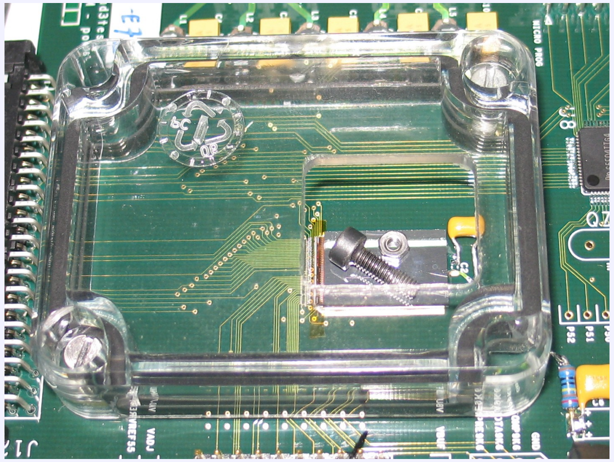


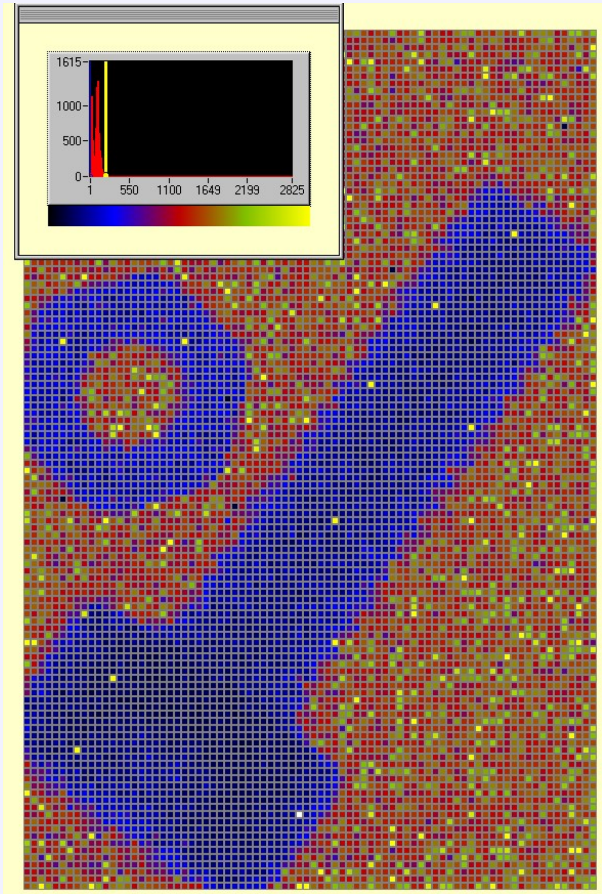
First setting of the pixels using the internal calibration mode.

The dispersion remains very low as expected from theoretical modelisation.

XPAD3 first images using Am source







Calibration test are scheluded on Jan. 23th at D2AM

Optical test will follow on next BLC days.