Détecteur 2D de nouvelle génération et application à la diffraction de poudres.

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

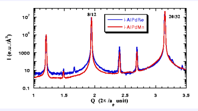
D2AM/CRG-ESRF Grenoble
LdC-CNRS
SOLEIL St Aubin
CPPM-IN2P3-CNRS Marseille

2 mars 2006 **Synchrotron Soleil, Saint-Aubin**

Summary.

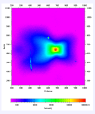
On D2AM-CRG/ESRF beamline (BM2).

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 **attenuators**, time consuming mapping

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



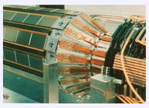
Complex shape of the diffusion around Bragg peak obtained by adding 10 (1000) frames. Out of peak to avoid blooming effects

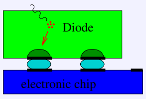
Data from M. de Boissieu, see Phil. Mag. Let. (2001) 81, 273-283 and (2003) 83, 1-29

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 beamline
pixel size	$250 \times 400\,\mu m^2$	mean spot size in 1995
exposure time	$1ms \rightarrow 1000\,s$	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 taking benefit of the Delphi detector peoples knowledge.

The XPAD project (XPAD1).

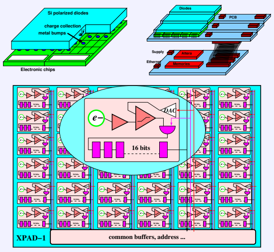
- Absorbed photons
- → electron clouds
- → charge migration
- → electron bunches
- → pixel threshold
- → pixel counters
- → on-board memories
- → ethernet data

Diodes :

- high resistivity Si

Chips :

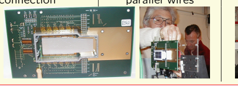
- AMS CMOS $0.8\,\mu m$
- 24×25 pixel/chip

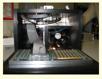


Boudet *et all.*, NIM A510 (2003) 41-44, Berar *et all.*, J. Appl. Cryst. 35 (2002) 471-476

XPAD detectors.

	XPAD1 2001	XPAD2 2003	XPAD3 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		14 bits
overflow counters	16 bits		16 bits
energy range	15 to 25 keV		7 to 25 keV
sensor	Si 300 μm (Delphi)	Si 500 μm	
counting rate	1.10$^6 ph/s$	2.10$^6 ph/s$	2.10$^5 ph/s$
time constant	500 ns with detector	208 ns with detector	*to be measured*
modules	5 × 2 chips	8 x 1 or 8 x 8 chips	8 x 7 chips
detector	1 module	up to 8 modules	
electronic	reduced	back plugged	
connection	parallel wires	ethernet 100MB	

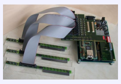


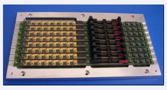


XPAD2 detector : 8 modules × 8chips

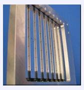
New diodes of 500μm Si thick → efficiency 78 % @15keV, 21% @25keV

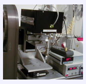
Diode ⥫ 8 chips of 24×25 pixels
PCB card : drivers and regulators.
Modules ⥫ acquisition card
Alterra Nios kit + ethernet



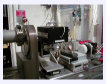


Tiled as close as possible
→ reduce shading, dead zones.
Metallic holder → few μm.
Size : 200×192 pixels
Surface $\approx 68 \times 68 mm^2$.



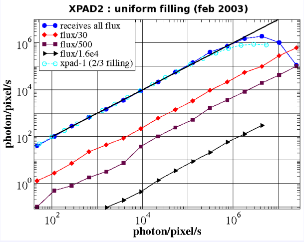


Interface software
developed using LabWindows/CVI application software moves to Linux.
XPAD prototype at SAXS station.



Dynamical range

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



No blooming effect

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

Kinetics : low resolution powders

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 ($\approx 10\,s$).

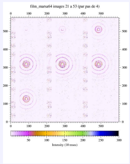
To study such phases diagram, various setups can be considered.

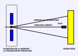
- Moving films (IP) are often too slow.
- Gas detectors with dedicated acquisition electronics (256 frames of 10 ms, no read out time) : limited detector counting rate (JF Javel, Thesis, Nancy 1998)
- X-Ray Intensifier and Frelon CCD camera (25ms frames but readout time 110ms/frame) : (C. Curfs, Thesis 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.
- . . .

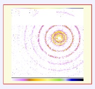
Kinetics potentiality of XPAD2

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 $>= 10ms$

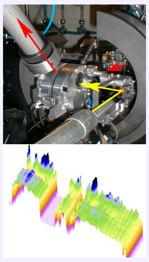




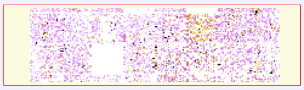


Images of 10 ms each taken off a 2s movies showing difraction while the sample crosses the beam at D2AM SAXS camera.

Kinetics of quench studied by diffraction



Data collection is limited by the cell apperture, which has been designed for linear detector, a few frames of 20ms around crystallisation shown at 10 frames/s during $CaAl_4O_7$ quench.

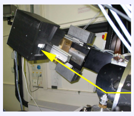


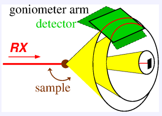
The quench of $Al_{2x}Ca_yO_{3x+y}$ ceramics can lead to vitrous or crystalline oxides. The transition between the liquid state and the cristalline one occurs in less than 20ms and may exhibit some transient phases.

Powder diffraction application (1)

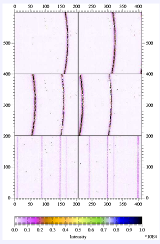
Scintillator and slits → 2d-detector.

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





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



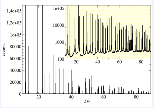
Raw images with Bragg lines, low and high angles.

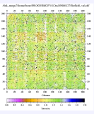
Powder diffraction application (2)

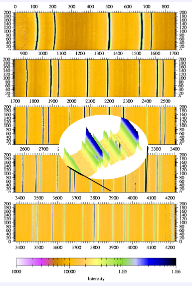
Resulting counts Y on pixel p : $Y_p = N_p^{-1} \Sigma f_q\, y_{q,i}$
$y_{q,i}$ counts on image i of pixel q,
f_q flatfield of pixel q :
Pixel : $q \in Image_i \;\rightarrow\; p \in Image_{merged}$: $q = q(p,i)$
Minimisation: $\sum_p (Y_p - N_p^{-1} \sum_i f_q\, y_{q,i})^2$
Powder lines : $Y_{p \in Ring} \rightarrow Y_{Ring}$

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

- Reconstructed Debye-Scherrer film →
- Powder diagram + flatfield extraction

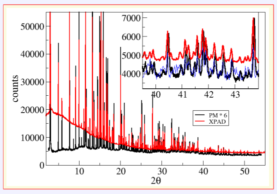


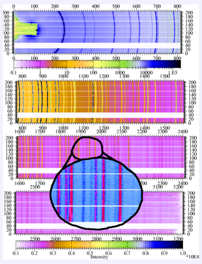




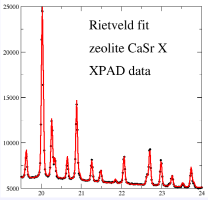
Powder diffraction application (3)

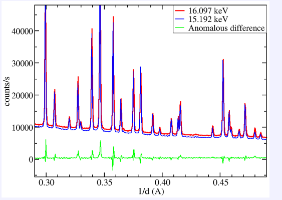
- Can be applied to complex materials : Zeolite
- High quality powder pattern can be extracted with time reduced by 20.





Powder diffraction application (4)





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.

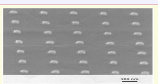
Data quality allow the use of anomalous difference.
Data recorded in 1/200 time will lead to similar results.

Multilayers

Challenging applications on material science beamlines consist in :

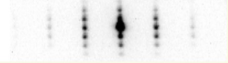
→ **anomalous mapping** of layered materials

→ **local structure** of self organized layers (Qdots...)

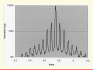


Ferroelectric $PbZrTiO_3$ dots
size $= 80 - 100\ nm$
spacing $= 300\ nm$
coverage area $= 0.4 \times 0.4\ mm^2$.

Diffraction imaging is a non destructive study.



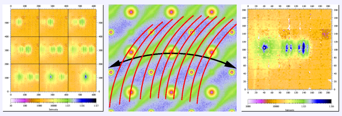
CCD / Counter



Pixel detector is required to reach the needed data quality.

Multilayers measurements

Epitaxly grown multilayer are now common sample to characterize : they need mapping of the reciprocal space which is time consuming. At the time such maps are recorded with slits and fixed (h,k,l) point of the reciprocal lattice, attenuator are often required near the substrate. 2-D detection allows an important improvement in these acquisition but it need to be able to manage high dynamics and to transform your reciprocal slices into reciprocal maps.



A PBT superlattice / MgO has been scanned allong the $(00l)$ direction. Images of 200s have been recorded from 5.95 to 4.05 with a l step of 0.05 , this was prefered to direct integration of this space as it allows to measure more accuratly the shape of the lower peaks.