

# IWORLD 7: Industrial Applications

Applications and new  
Developments in X-ray Materials  
Analysis

PANalytical

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P. Kidd, P. Fewster /  
PAN Sussex Research

## Outline

- Background
- Medipix Tech-Transfer
- First results on detector properties
- Expectations for XRD applications
- First results of Medipix in XRD
- New EUREKA project “RELAXD”
- Conclusions

## Main Activities

# X-ray Diffraction and Fluorescence for Scientific and Industrial Research

Winning by Sharing  
know-how and experience  
in X-ray diffraction and fluorescence

## Main Activities

- XRF (Industry and Research)
  - Elemental Analysis (qualitative and quantitative)
  - Applications: Cement, Petrochemical, Plastics, Steel, Aluminium, Environmental, Geology
  - Automation
- XRD (Research and Industry)
  - Phase Analysis (qualitative and quantitative)
  - Applications: Pharmaceuticals, Cement, Minerals
  - Other: Thin Films and Semiconductors, Nanotech,
  - Automation

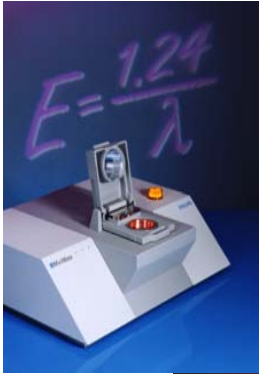


PANalytical is located in Almelo for over 35 years



Proportional (Elemental Analysis)

**MiniMate**



**MiniPal 2**

**Venus**



**CubiX**



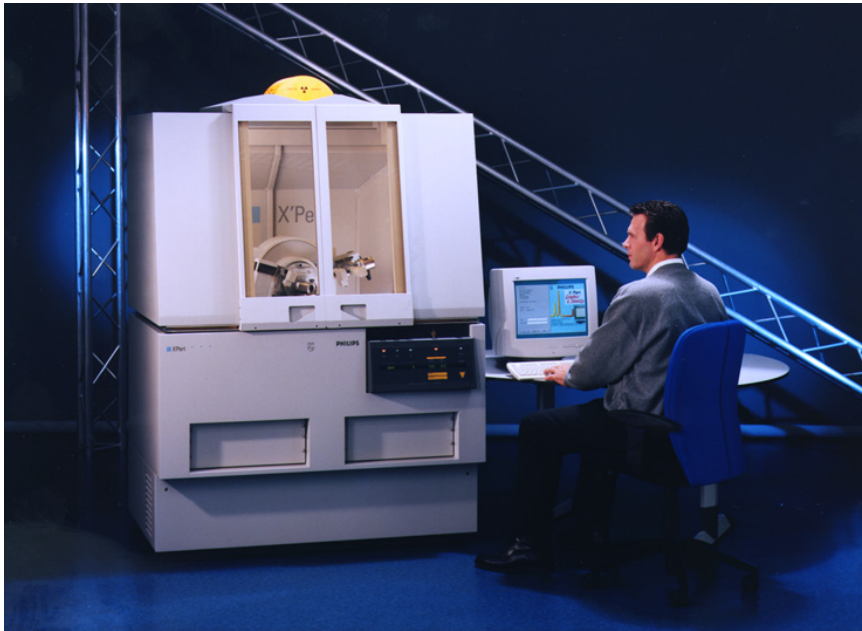
**Axios**



**MagiX FAST**

Product range (Phase analysis)

## X'Pert PRO



## CubiX PRO



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## X'Pert PRO MRD

- Advanced X-ray analysis for new materials research and development
- For thin films, semiconductors and microstructures



## X'Pert PRO MRD XL

- X-ray analysis for research and process development of advanced materials
- Analysis of wafers up to 300 mm diameter
- Automatic wafer loading



## X'Pert PRO MRD XL

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- Analysis of wafers up to 300 mm diameter
- Automatic wafer loading



## X'Celerator

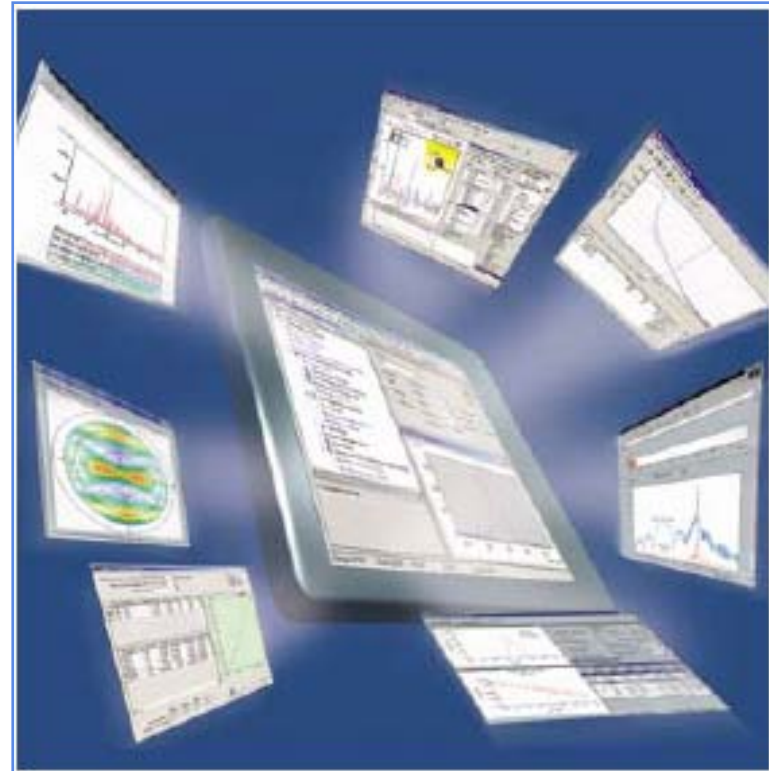
- The standard in X-ray powder diffraction
- Speed and resolution
- Rapid data collection of complete powder diffractograms





## X'Pert Software

- A complete range of software packages for X-ray diffraction
- Based on the universal standard for data sharing and file transfer and XML



## CSI MIAMI

- FORENSIC SCIENCE





**Acrobat Document**

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# Medipix Collaboration at work

**CAPRI**



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# Acknowledgement

## CERN team

Michael Campbell

Xavier Llopart

Erik Heijne

## CERN ETT

Marilena Streit-Bianchi

Beatrice Bressan

## NIKHEF team

Jan Visschers

## Medipix Collaboration

Paolo Russo, Hans Erik Nielsson

## Chip design

Tech-transfer Office

+combined exhibitions

IEEE/Rome, Salon Paris

R/o electronics,

partnership new project

Origin Medipix activities



# PARIS: Salon de la Recherche et l'Innovation

**MultiPIX2@PANalytical**  
Un produit pour l'analyse par rayons X



### Le concept

La collaboration Medipix2 conjugue au CERN le savoir-faire PANalytical qui domine au monde mondial la fabrication de composants de fluorescence X. Les appareils à rayons X de la recherche sur les matériaux existant et sur les nouveaux matériaux du monde académique et industriel ont besoin de détecteurs de photons individuels MultiPIX2. Le développement des puces est effectué au CERN pour la réalisation d'une nouvelle génération de machines, et renforce sa position sur le marché d'acquisition des données au NEHEF à l'Université de Naples (Italie).

### Les applications

De nouveaux détecteurs à rayons X, conçus par la collaboration Medipix2 au CERN, vont beaucoup accélérer et améliorer la qualité de nombreuses applications, et permettre des applications tout à fait nouvelles. Voici des exemples d'applications actuelles :

- Recherche en nanotechnologie
- Industrie du ciment
- Alimentation
- Aluminium, acier et métaux
- Cosmétique
- Plastiques et polymères

### Développements

Un nouveau projet de recherche et développement a été créé, basé sur la puce Medipix2. Un consortium de partenaires se crée, comprenant L'Université de Liège, l'Université de Liège, PANalytical et Le but est de miser sur une technologie à un détecteur à grande surface et à une résolution très rapide. Ce projet de collaboration entre recherche et industrie se situe au stade de la demande de fonds auprès des détecteurs à grande surface sont très demandés, dans de nombreuses applications industrielles, telles que la recherche structurelle et fonctionnelle des molécules biologiques (protéomique). Ils peuvent ainsi contribuer à raccourcir les processus de développement pour de nouveaux traitements médicamenteux dans les industries pharmaceutiques et à développer des recherches nouvelles en imagerie médicale.




Exemple de XRD utilisé en recherche



Détail d'un composant du XRD



Le nouveau détecteur « RELAXD » - high Resolution Large Area X-ray Detector - développé par PANalytical au cours d'un projet de R&D.

**IN2P3**  
Institut National de Physique Nucléaire et de Physique des Particules

**dapnia**  
**cea**  
**saclay**



- Univ + INFN Cagliari
- CEA-LIST Saclay
- CERN Genève
- Univ d'Auvergne
- Univ Erlangen
- ESRF Grenoble
- Univ Freiburg
- Univ Glasgow
- IFAE Barcelona
- Mitthoegskolan Sundsvall
- MRC-LMB Cambridge
- Univ + INFN Napoli
- **NIKHEF Amsterdam**
- Univ + INFN Pisa
- FZU CAS Prague
- IEAP CTU Prague
- SSL Berkeley



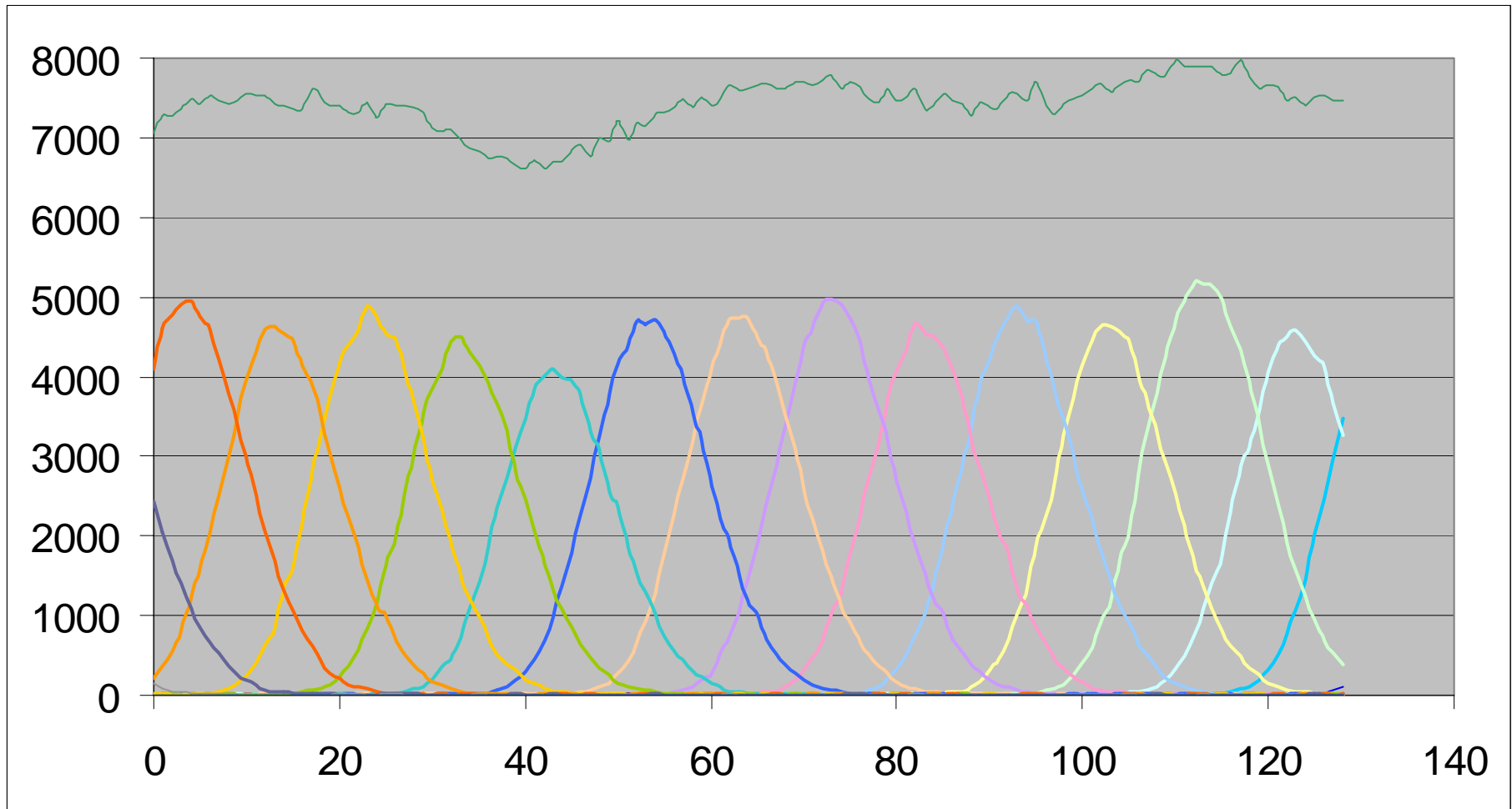
**Spokespersons:**  
**Michael CAMPBELL** CERN  
**Jan VISSCHERS** NIKHEF



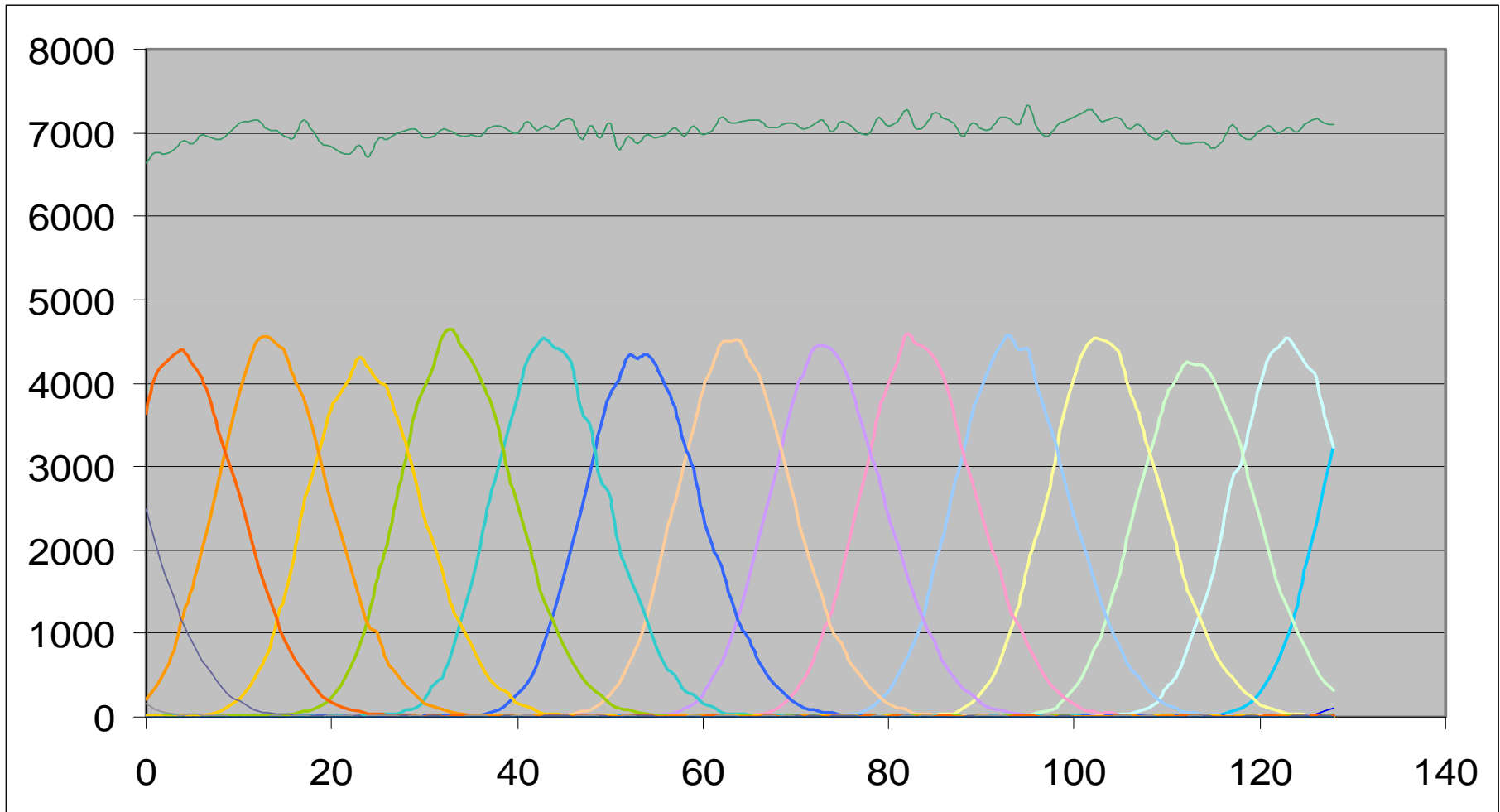
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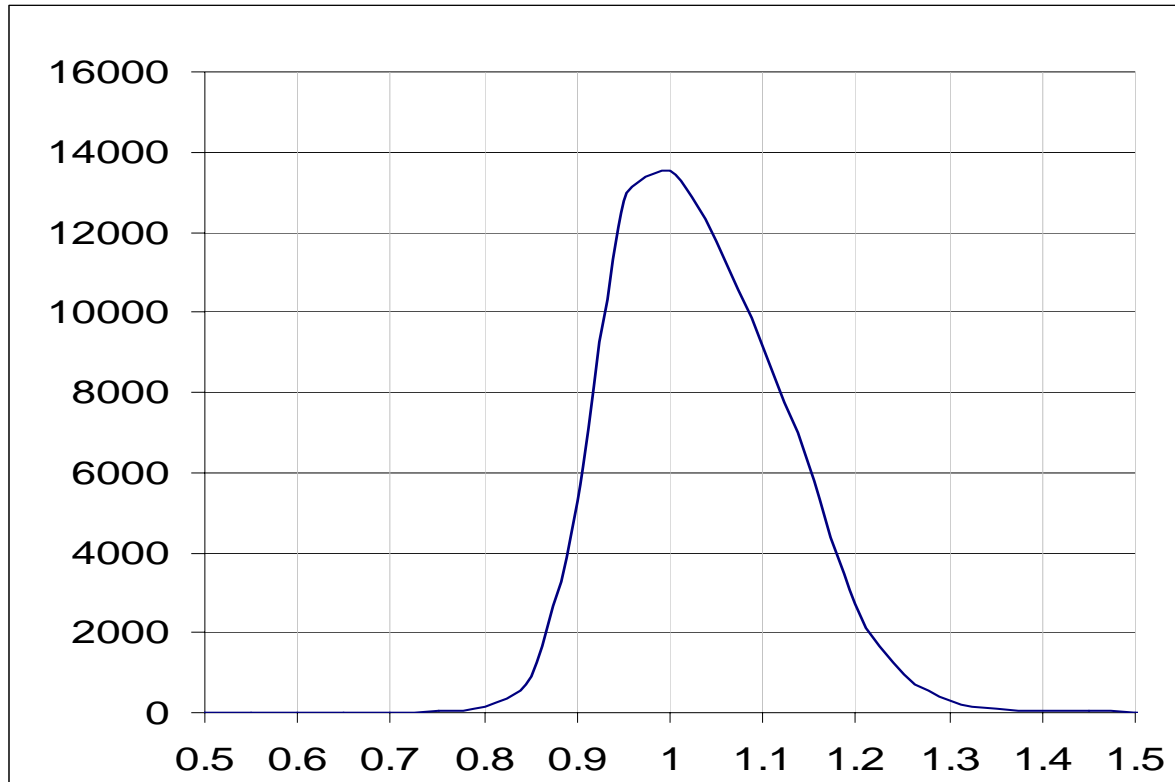
## Pixel response (before flatfield correction)



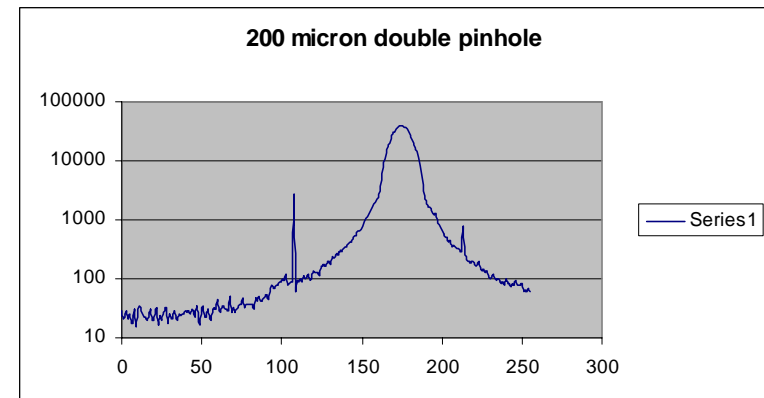
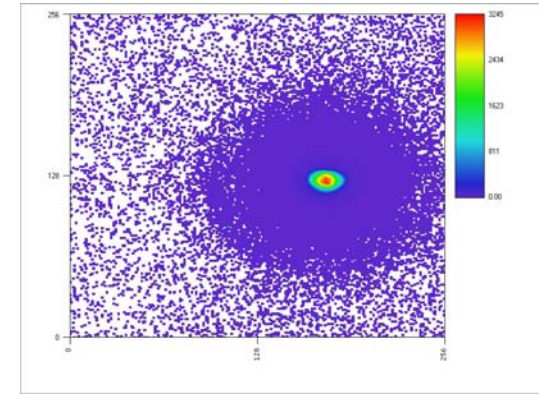
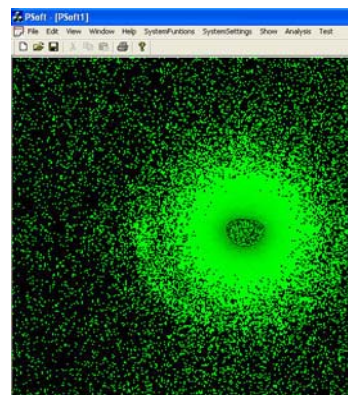
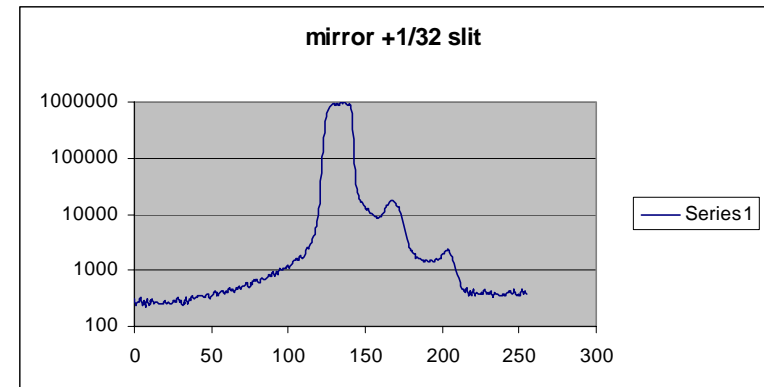
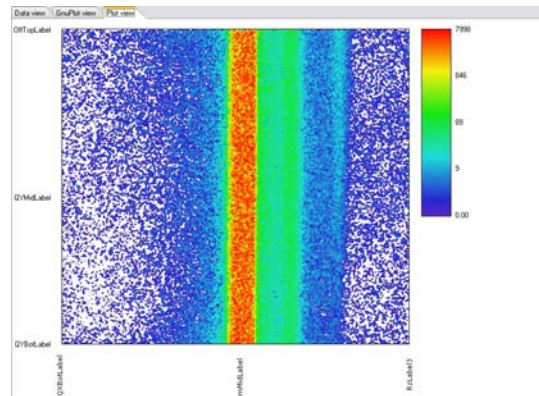
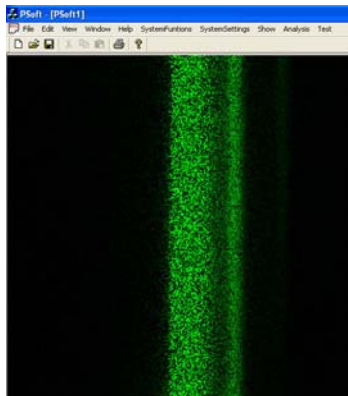
## Pixel response (after flatfield correction)



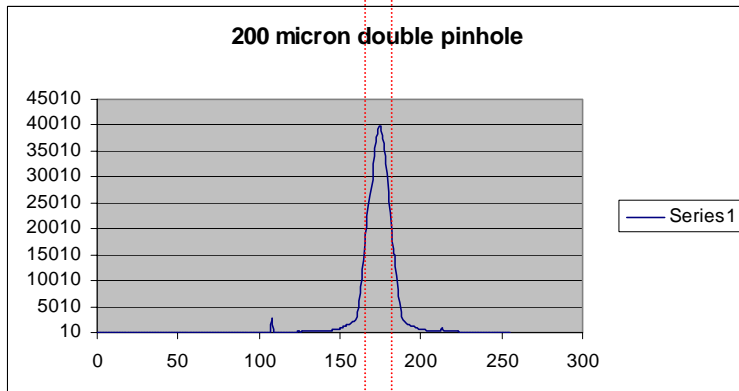
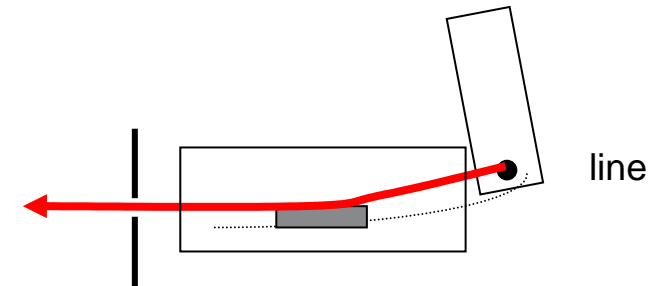
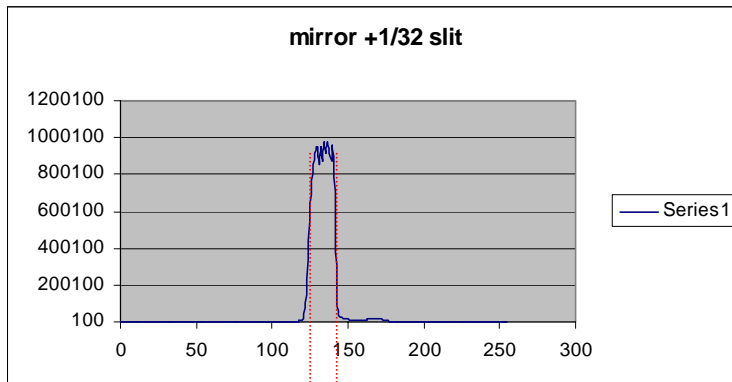
## Flatfield correction factors (all pixels)



# Medipix at PARC: Images of the direct beam



# Medipix at PARC: Images of the direct beam

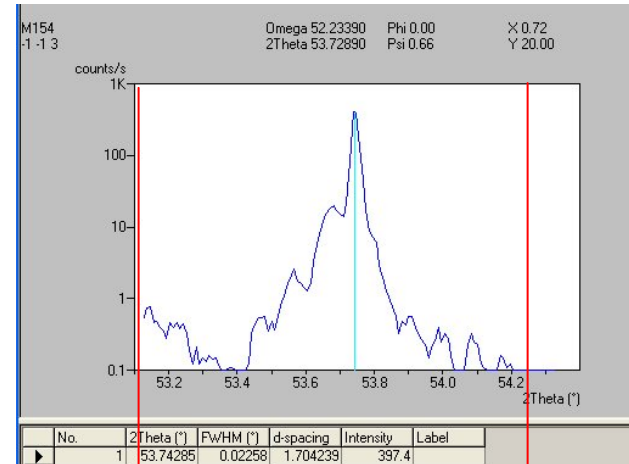


Long fine focus

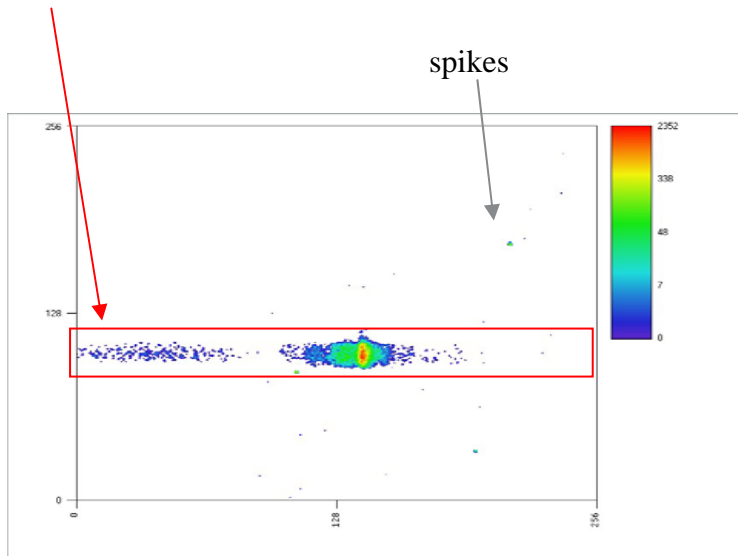
# Medipix Installation at PARC

## Early comparison with X'celerator Static

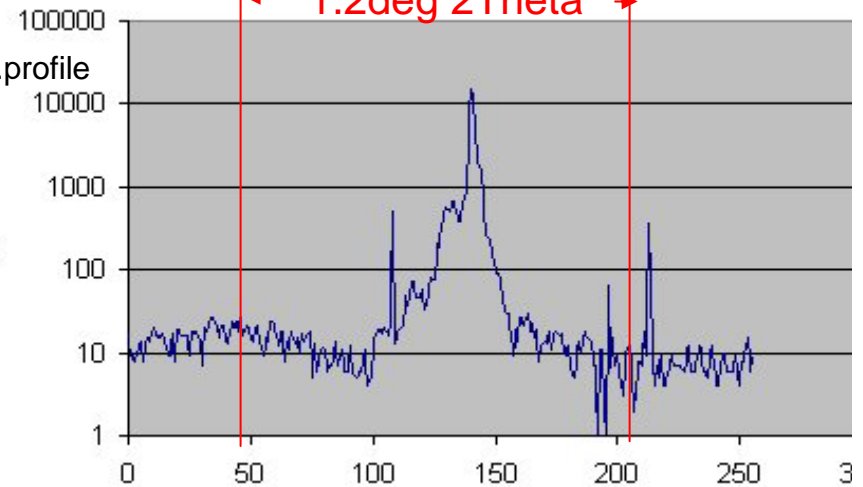
X'celerator 100s  
KX0427B



Improve data by isolating useful pixels

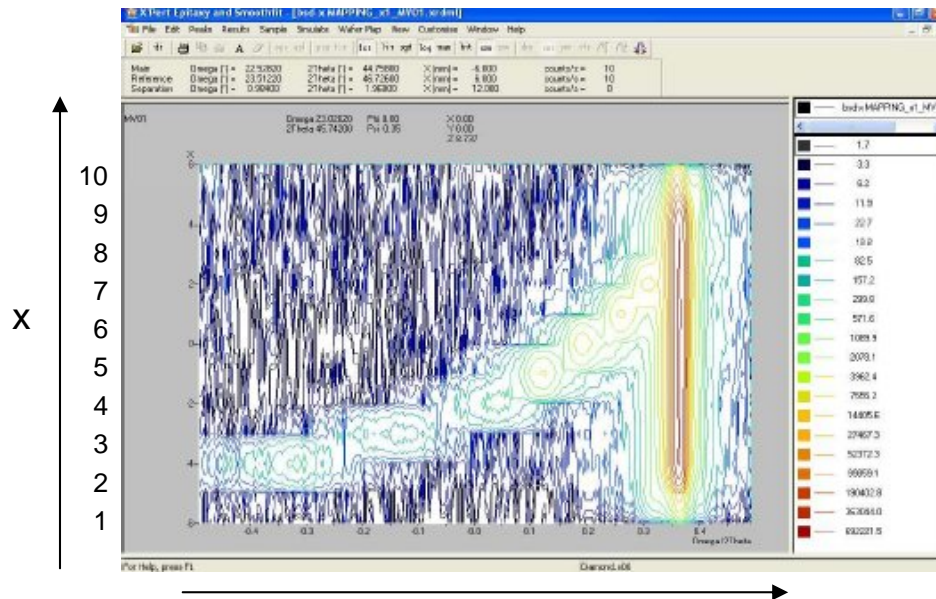


Medipix 100s  
KM0427C.dat.profile



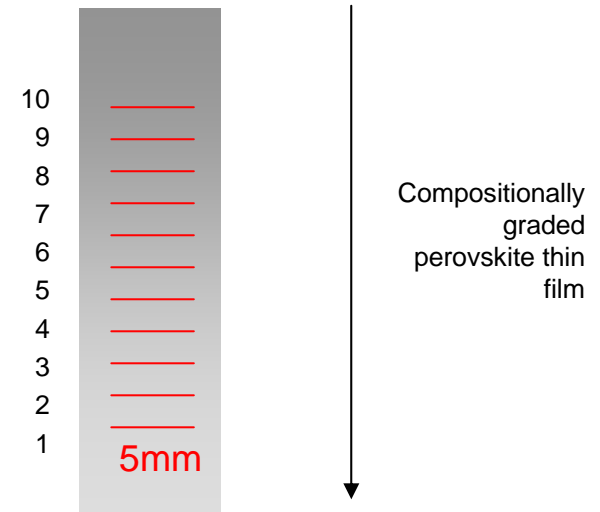
## High Resolution XRD rapid screening

- Rapid feedback on samples with compositional grade: 12 measurements in 12 minutes!



$1^\circ$  range 2Theta/Omega

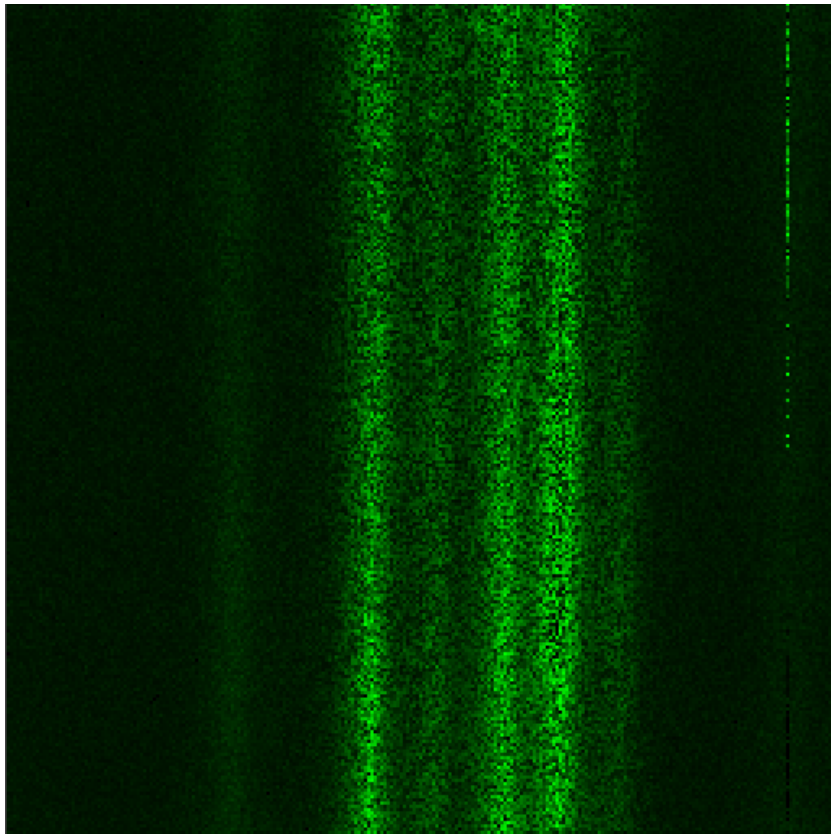
Plan View



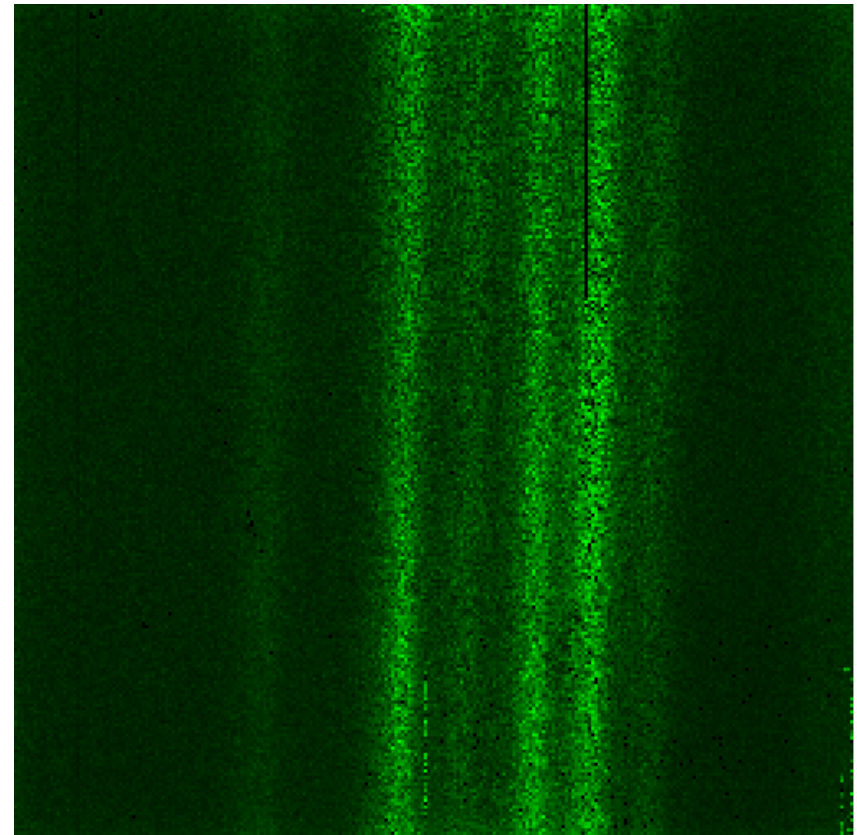
rapid 2Theta/Omega scans



**“Five fingers of quartz”, 300 vs 700 micron sensor**

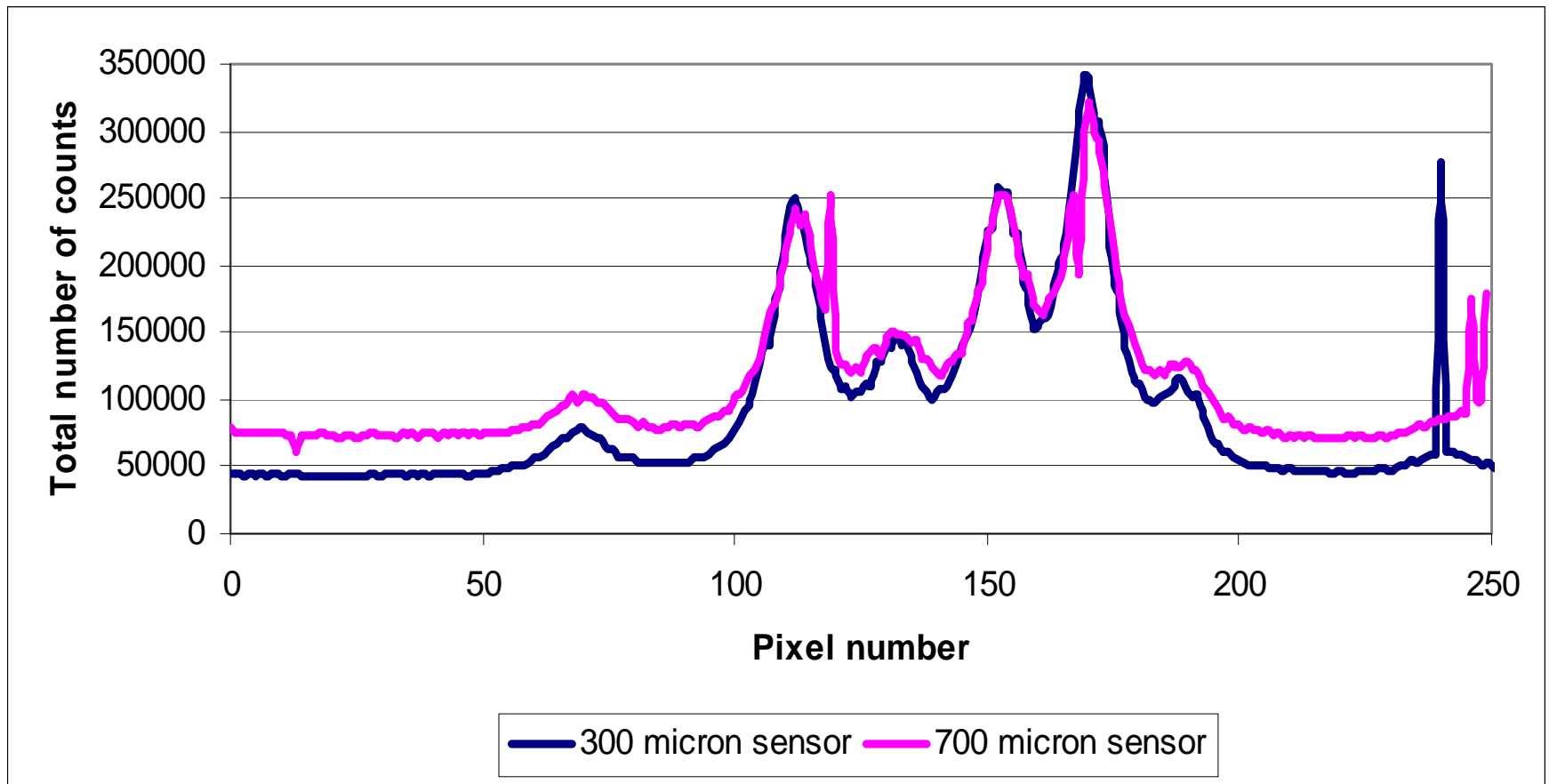


**300 micron sensor**



**700 micron sensor**

## “Five fingers of quartz”, 300 vs 700 micron sensor

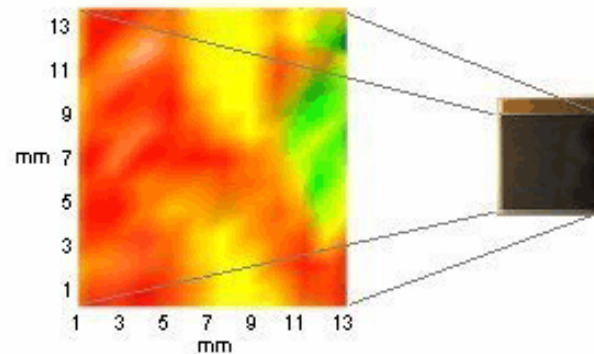


# XRD as tool for detector development (www. Dannelab.com)

## XRD Use for Production Quality Assurance

### Novel Devices from Particle Physics

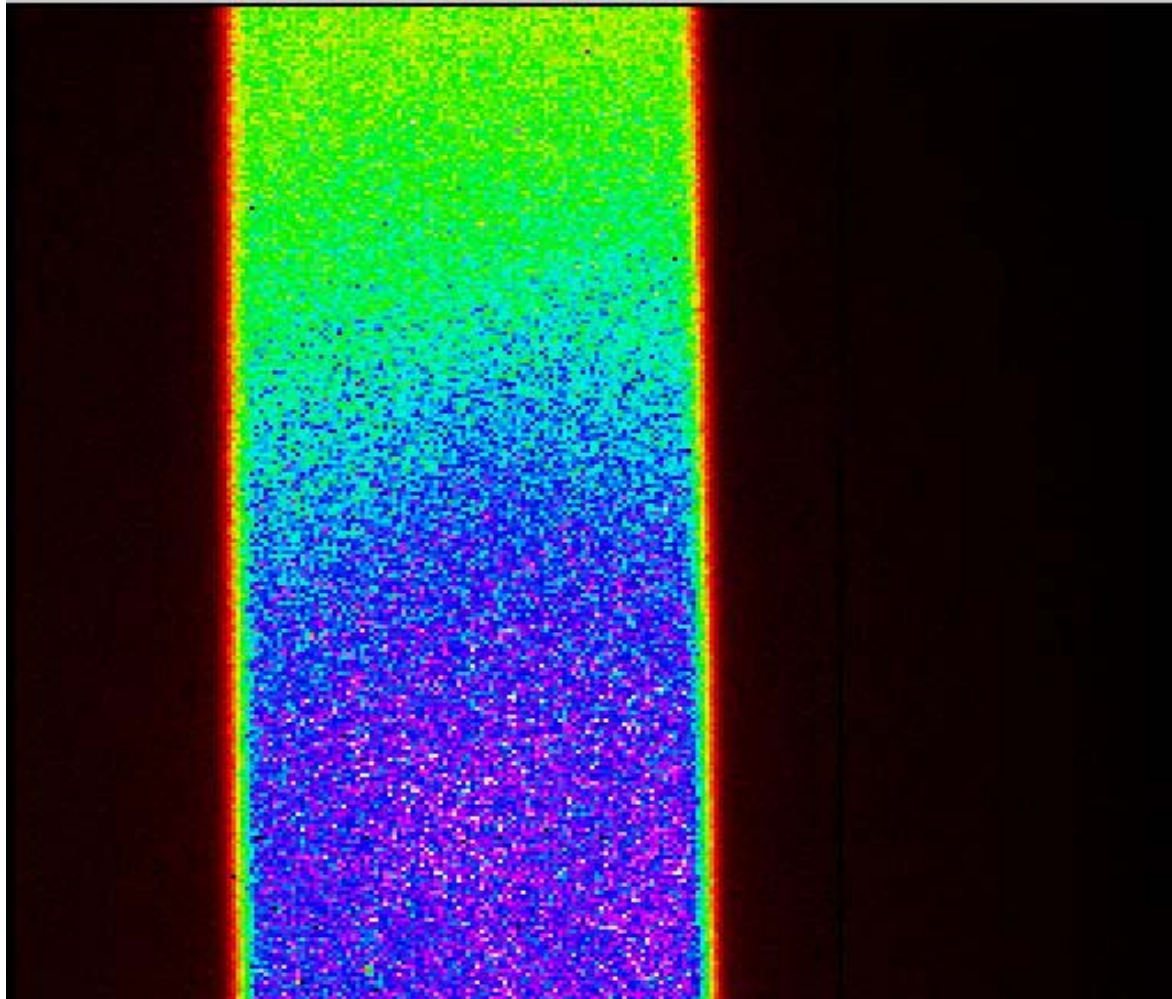
Mapping the surface of Si sensor bump-bonded onto the large IC chip helps to identify the areas of stress concentration and optimize the production process.



Mapping performed by measuring rocking curves at the different points of the sensor. Red color represents non-stressed Si and green indicates the presence of stress.

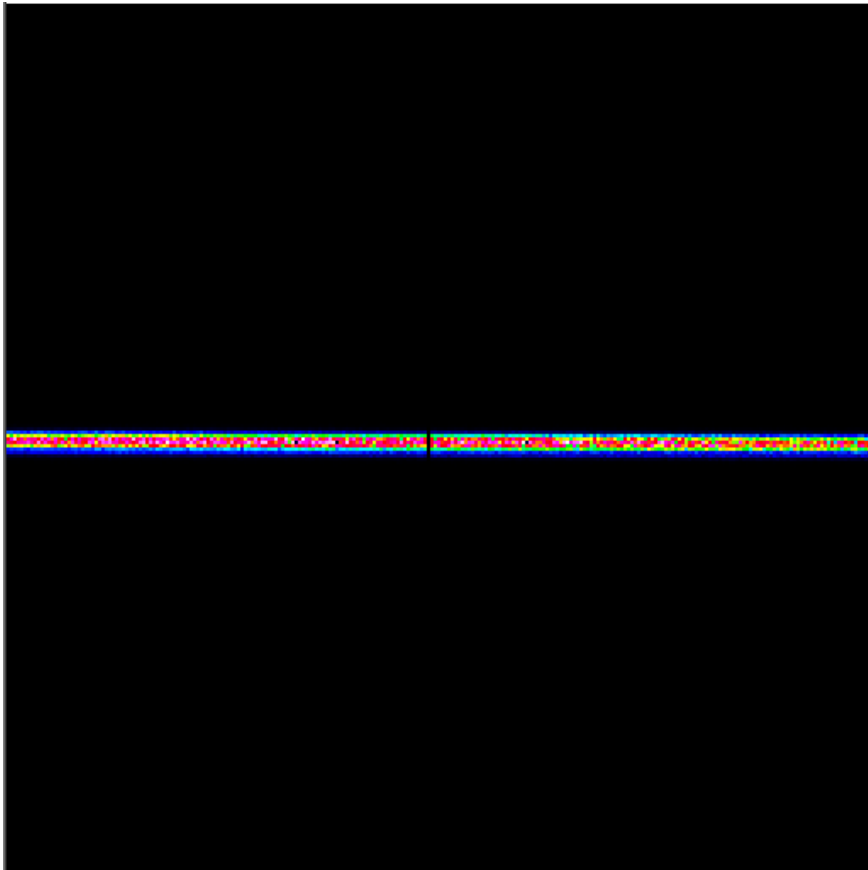
*Project of CERN*

First shot: direct beam

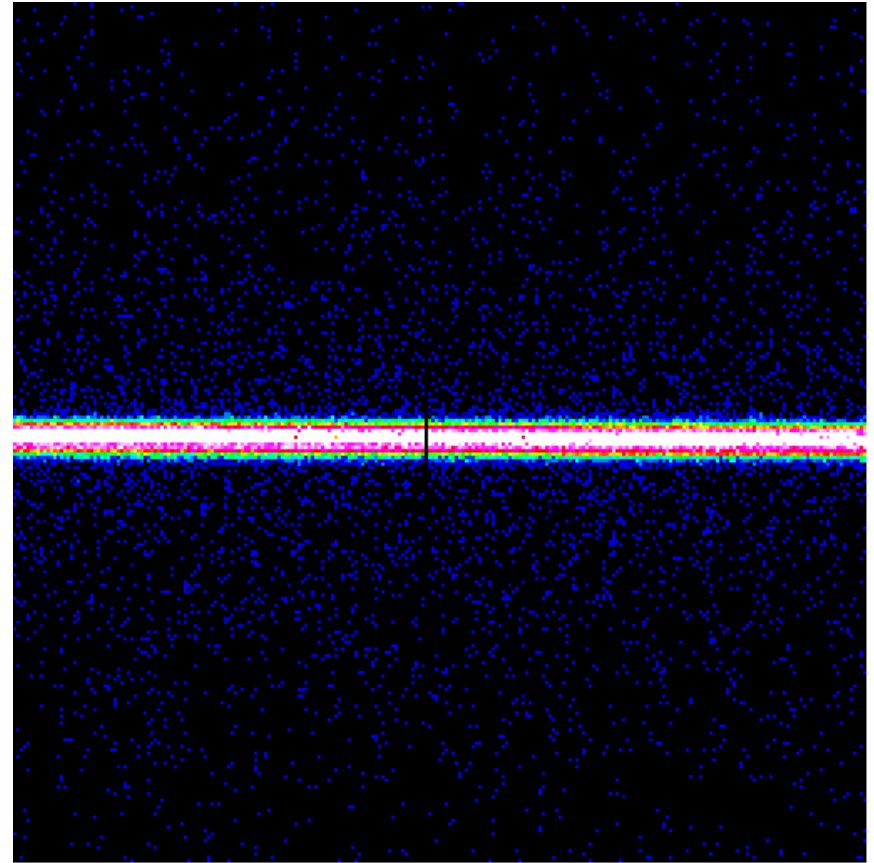


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## Direct beam



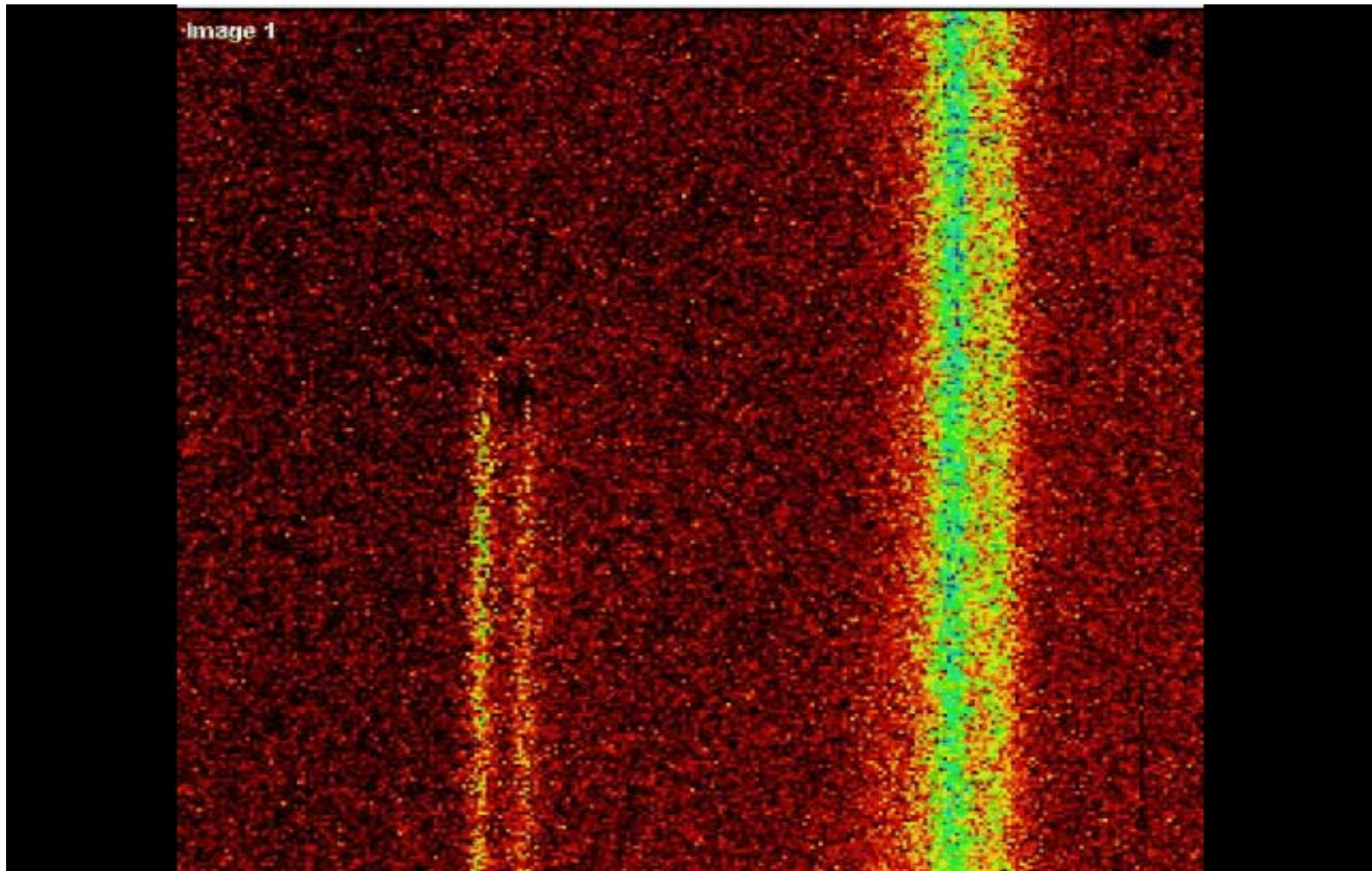
**Linear scale**



**Logarithmic scale**



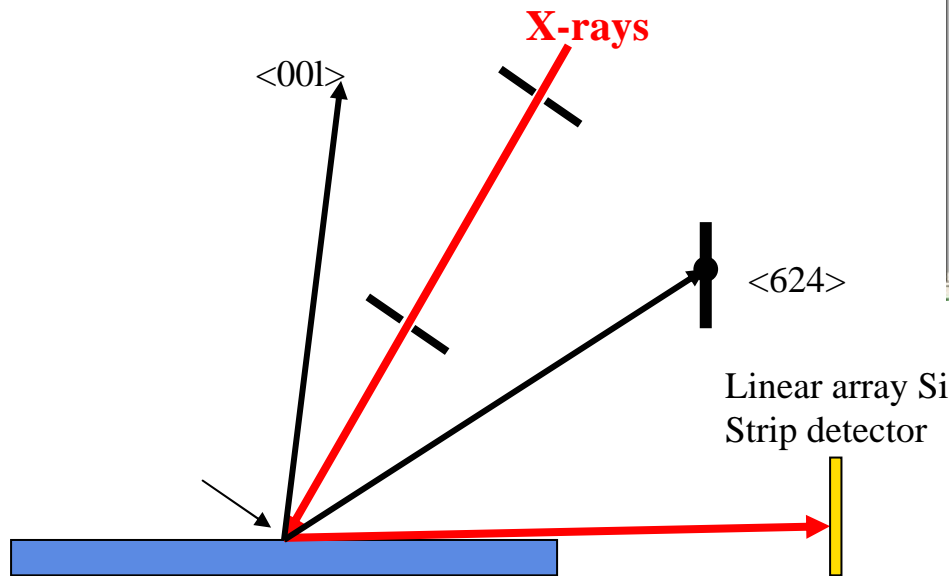
R&D: Medipix collaboration - PANalytical



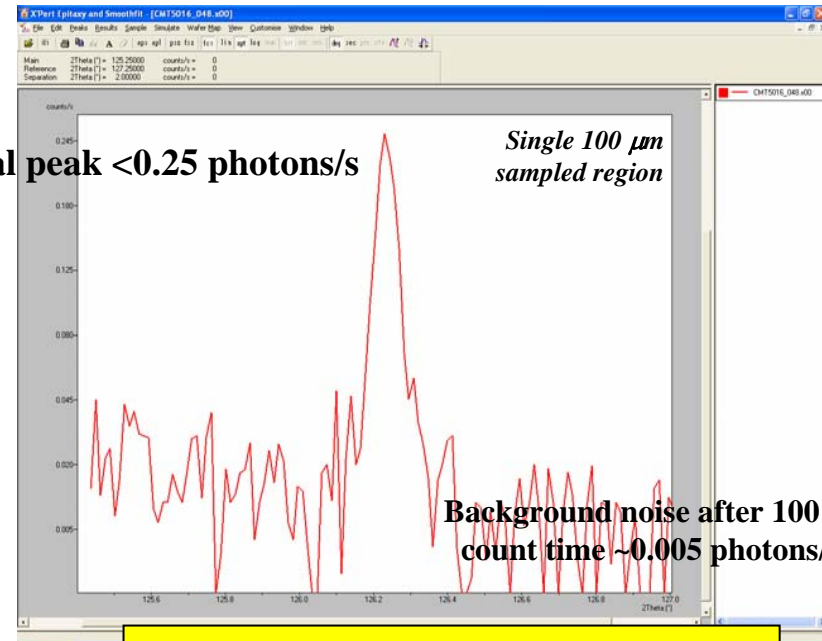
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# Micro-high-resolution wafer mapping

“Static” measurement geometry for wafer screening of CdHgTe diode arrays for thermal imaging cameras

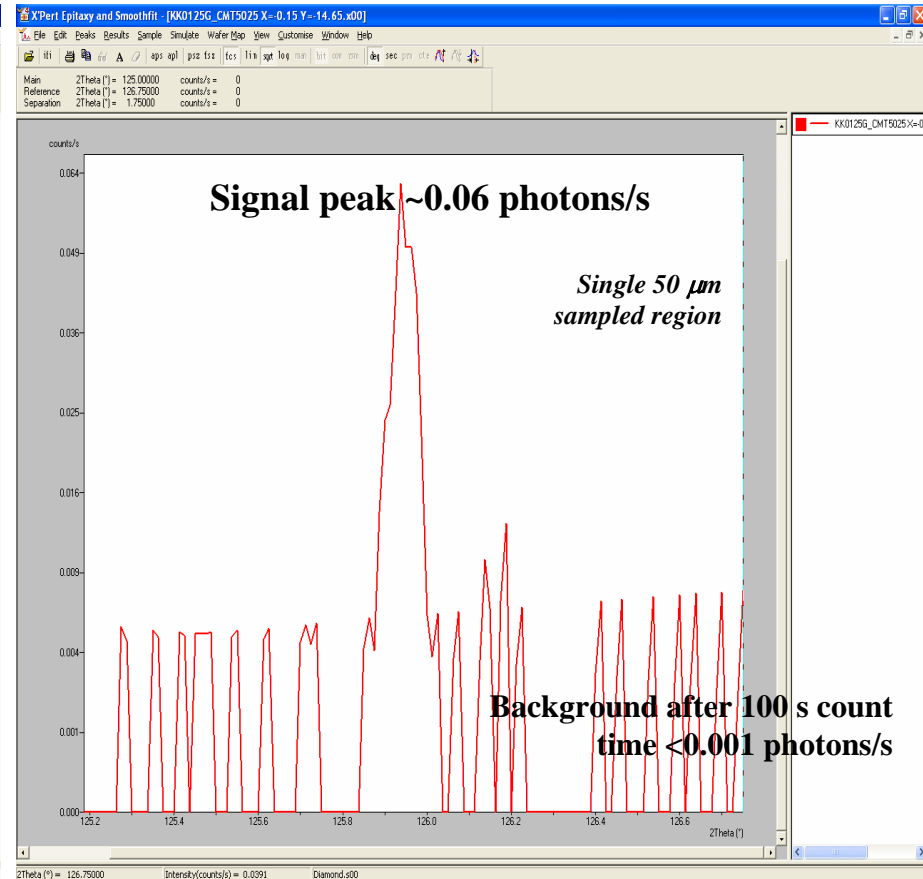
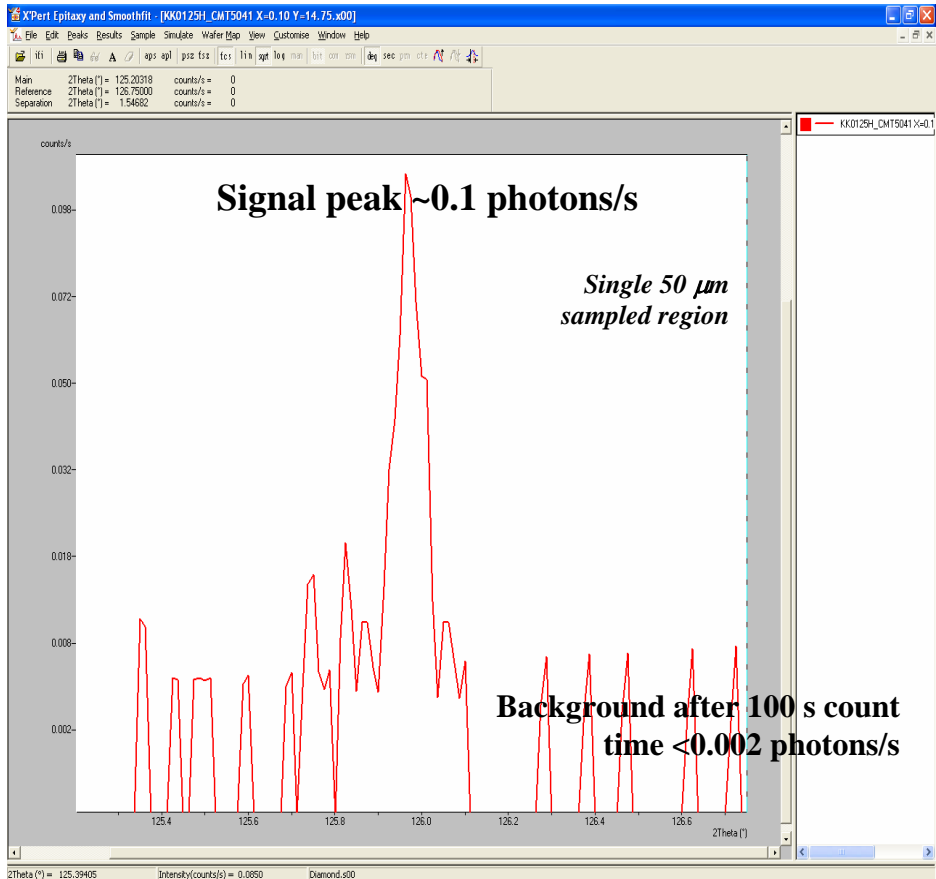


Signal peak  $<0.25$  photons/s



- Discrimination on every photon separates very weak scattering from random noise in measurement
- Signal peak gets enhanced with counting time
- Random residual noise statistically cancels with counting time
- Results in an enhanced dynamic range

# Still weaker signals; very small residual noise



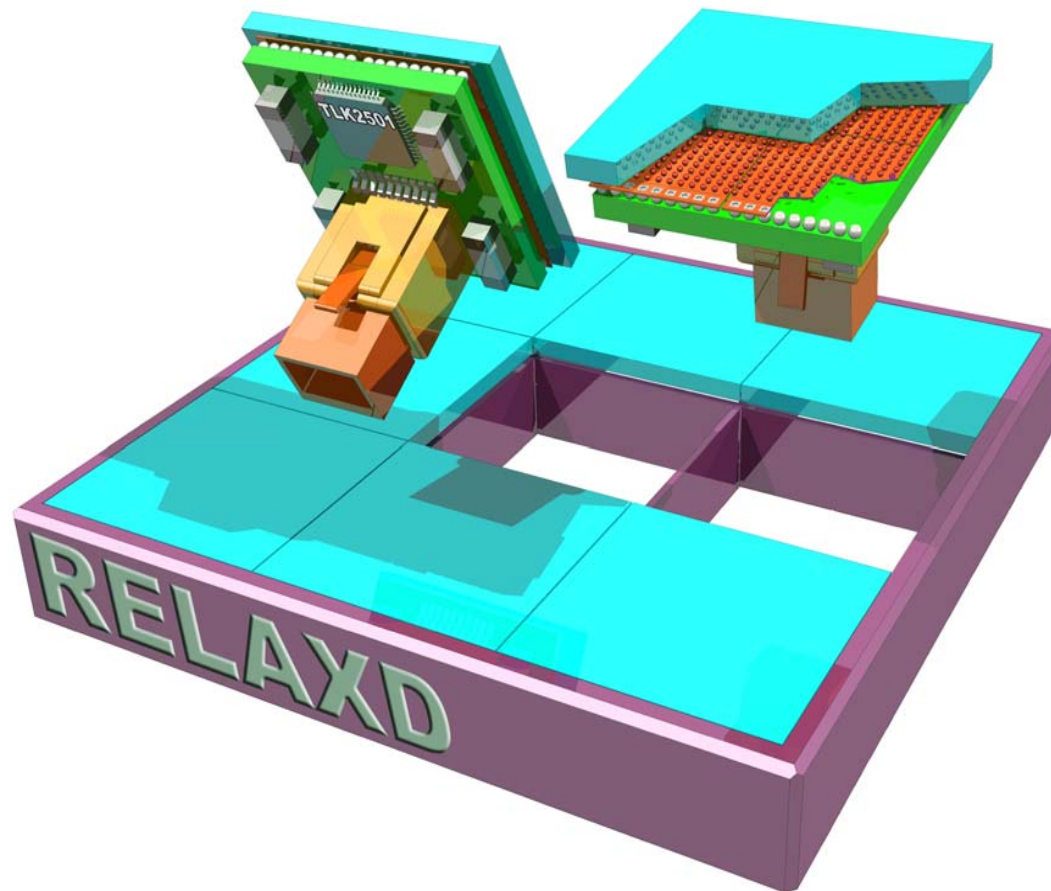


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# RELAXD

High REsolution Large Area X-ray Detector



## RELAXD Consortium

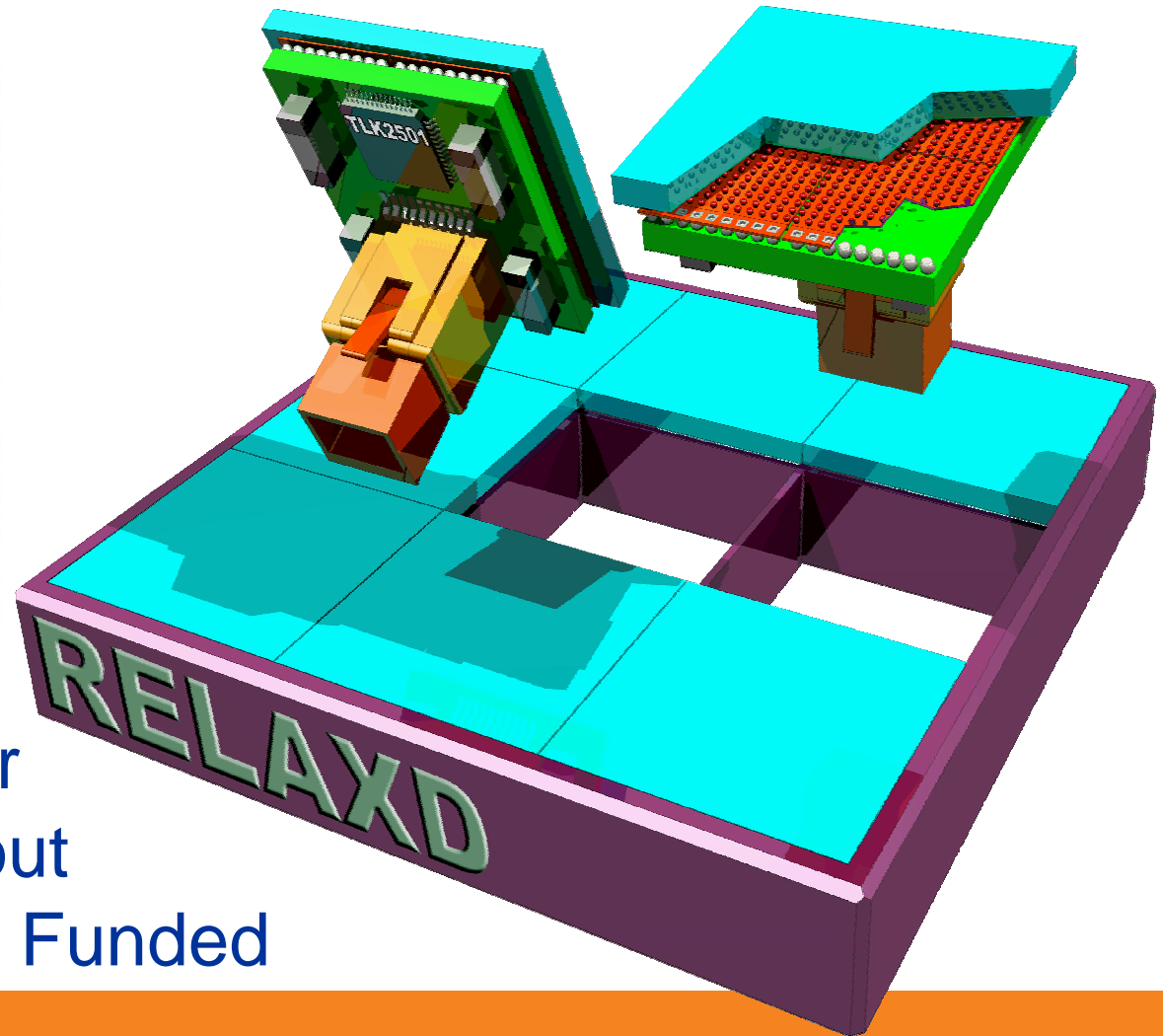
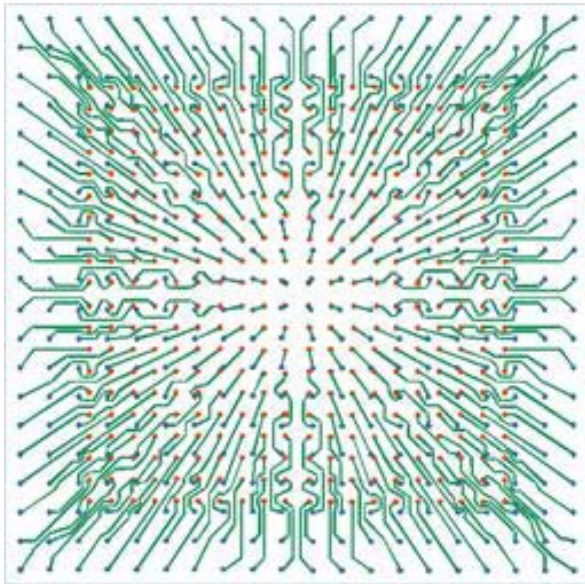
- CANBERRA Olen, Belgium
- IMEC Leuven, Belgium
- NIKHEF Amsterdam, Netherlands
- PANalytical as Penholder:  
Almelo, Netherlands

- **Cost & Subsidy (plan! not yet officially approved!)**
  - 2690 kE total project
  - 1138 kE PAN 60% funding => 455 KE own & **683 kE IS**
  - 922 kE NIKHEF 553 kE IS
  - 633 kE CANBERRA 331 kE IWT
  - 0 IMEC funded by CAN mainly and also by PAN
  - If you go larger (area) => the s/w r/o and acquisition algorithms have to be done anyway!

- **5 elements**

- Fan-out structure – pitch adaption
- Minimized dead spaces
- Through Si via technology – 4 side buttability
- High-speed Gbit/s read-out
- Software for data acquisition, homogenization of large area pixel response calibrations, control software  
=> X-ray detector ('low' energies)

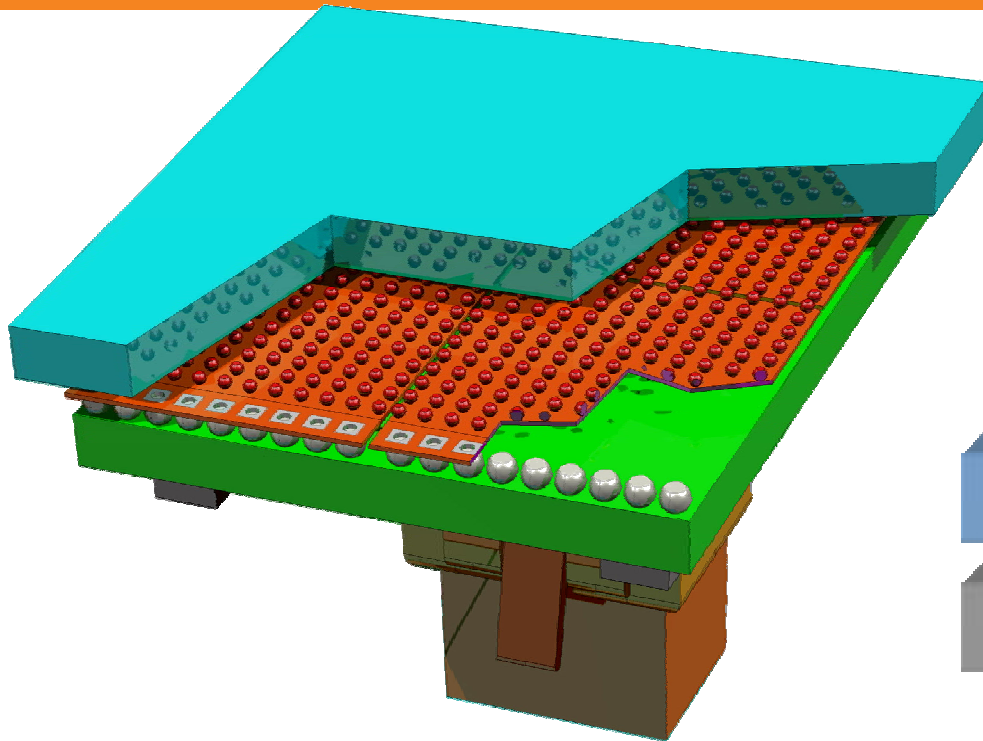
## Fully tiled X-ray imager



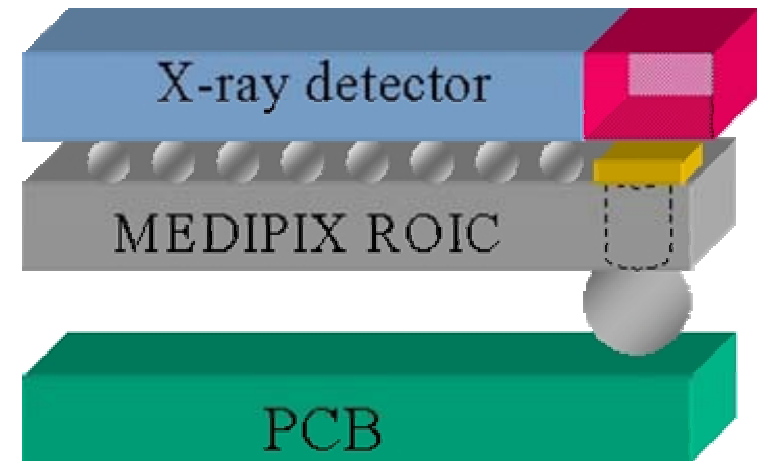
need pitch adapter  
Gbit/s serial readout  
Innovation Project Funded



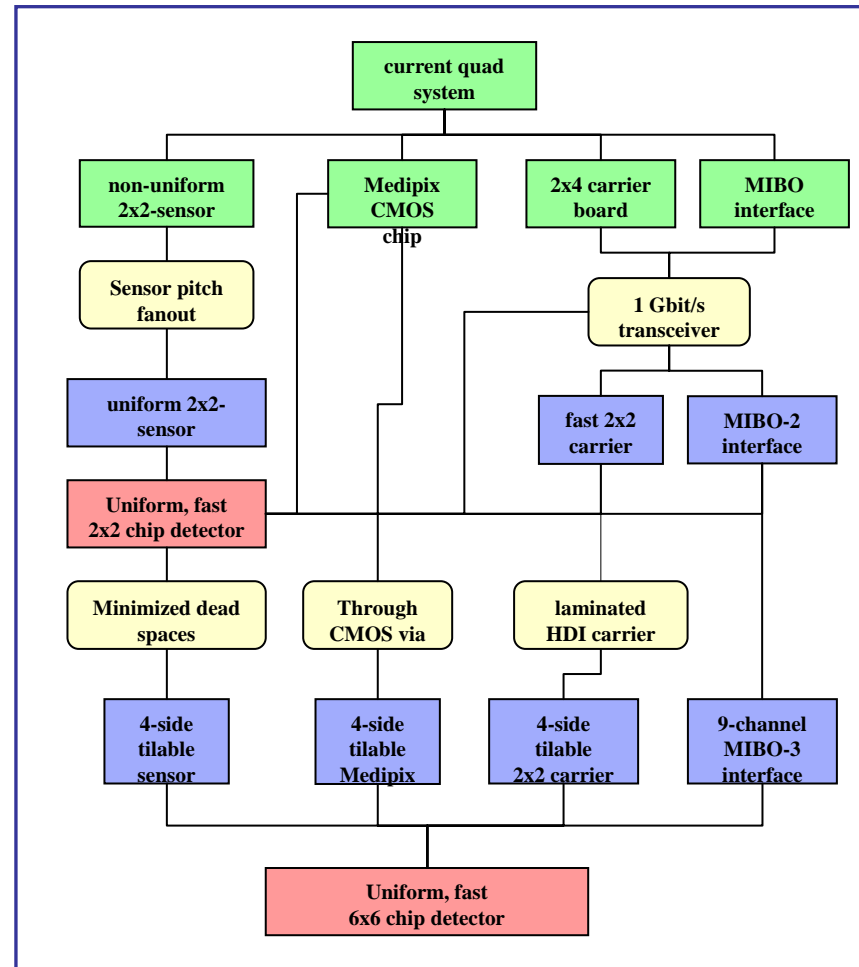
## The Future: Medipix2 tiling



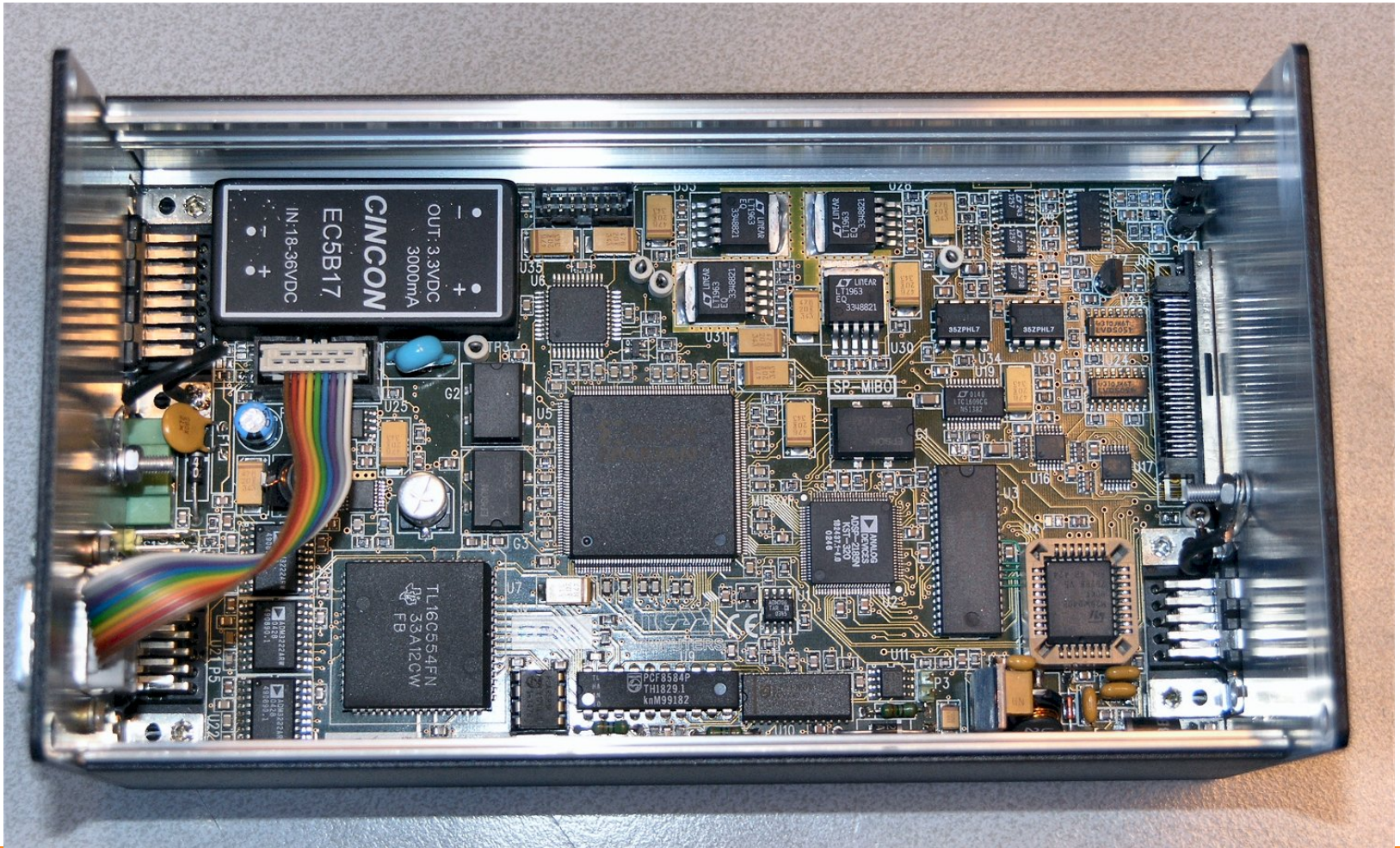
through-via etching  
wafer thinning  
3D stacking



## Project Flow

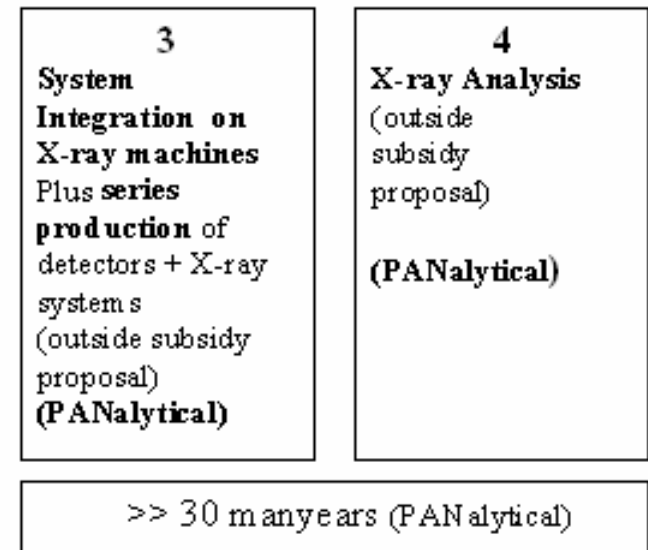
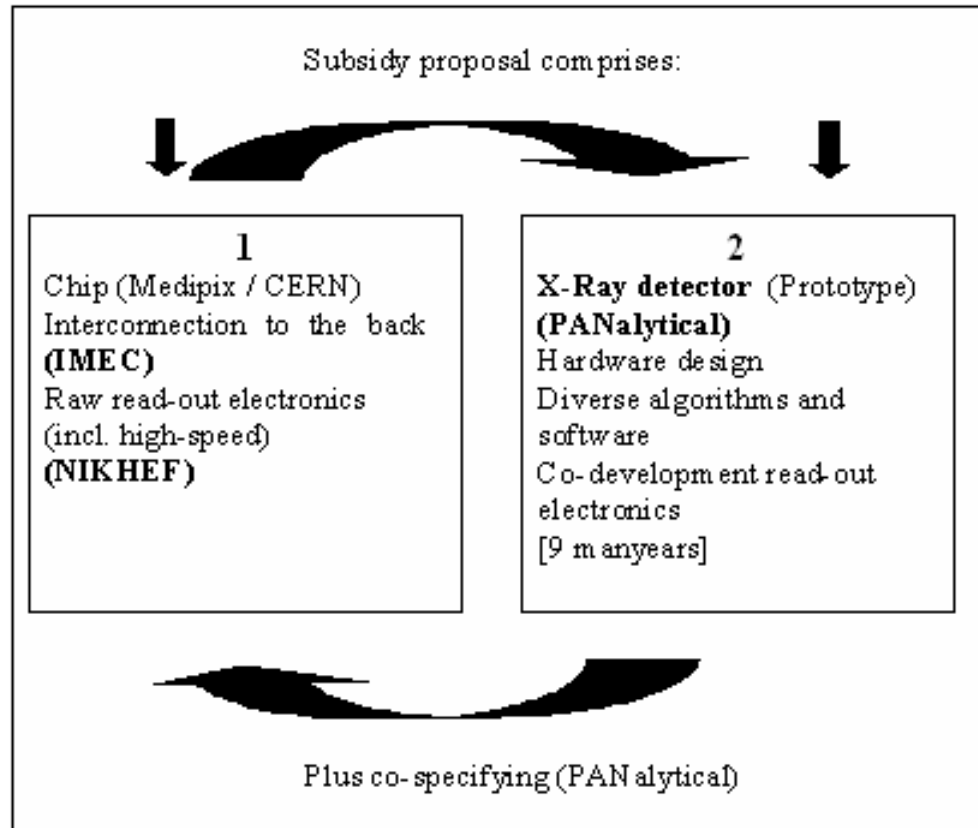


## Pictures of the MIBO



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## Role PAN





## Roadmap Partners &amp; PANalytical

**Potential Partners**



**Tech & Application  
Center**  
**PANalytical**  
*Product / Integration /  
Software*



**Core Partners**

**CERN / NIKHEF /  
CANBERRA / IMEC**  
*Chip design / Electronics /  
Software*



**Output => PLATFORM**

*Basic detector modules  
Tiling- RELAXD  
Mainstream  
+ spin-offs*

**Suppliers**

**IBM  
MCNC  
Electronics companies  
Outsourcing**

**Relevance: PAN Detector center**

**Pixel intelligence**

**CERN**

**Medipix 2**

**Detector chip production**

**CMOS process (0.25  $\mu\text{m}$ )**

**Medipix + , ++**

**0.13  $\mu\text{m}$ , 0.09  $\mu\text{m}$  CMOS**

**....Super-chip,  
>RELIABLE<**

**CANBERRA**

**IMEC**

**NIKHEF**

**PANalytical**

**(MCNC)**

**Large® area**

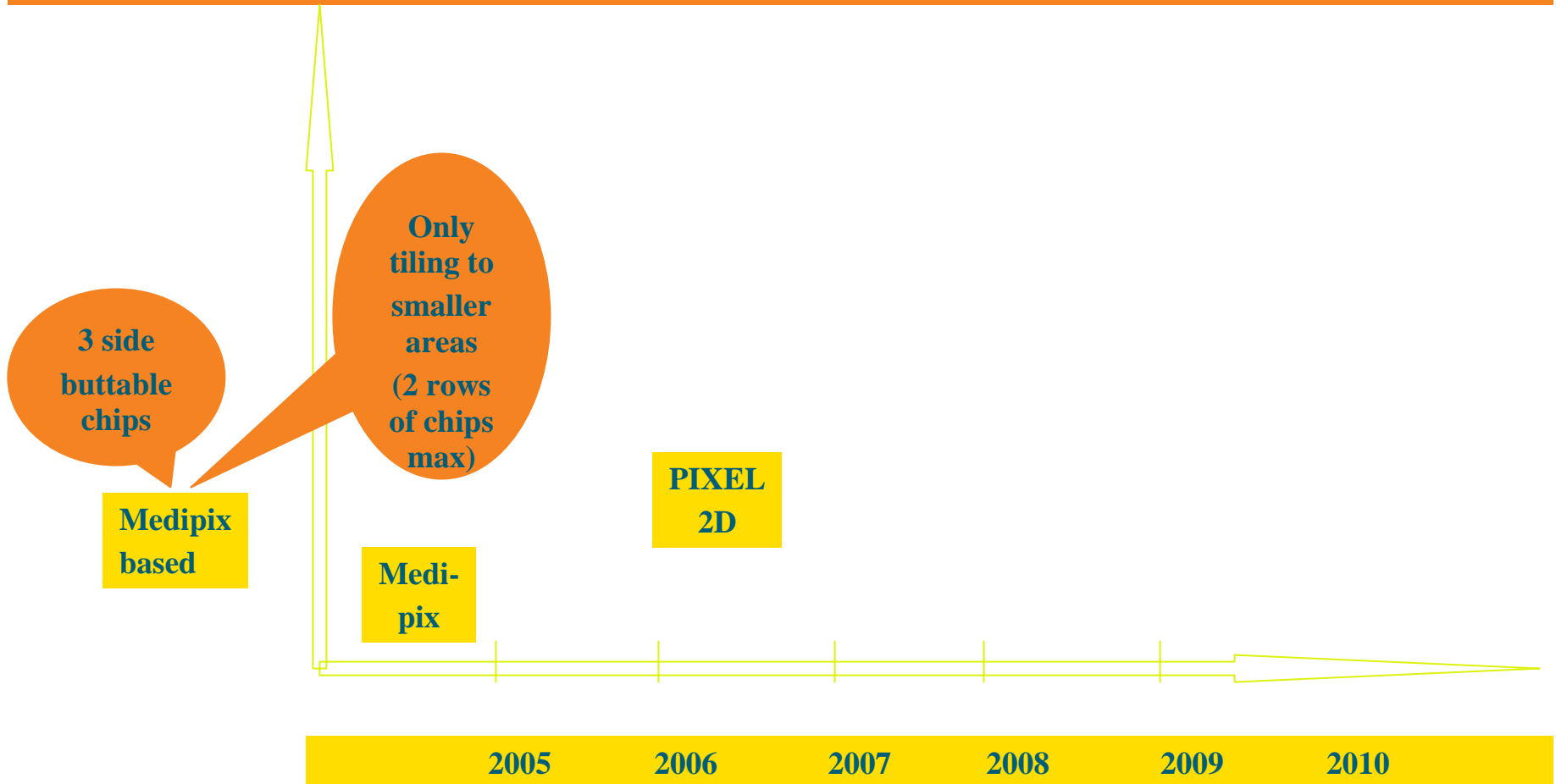
**Various shapes & sizes**

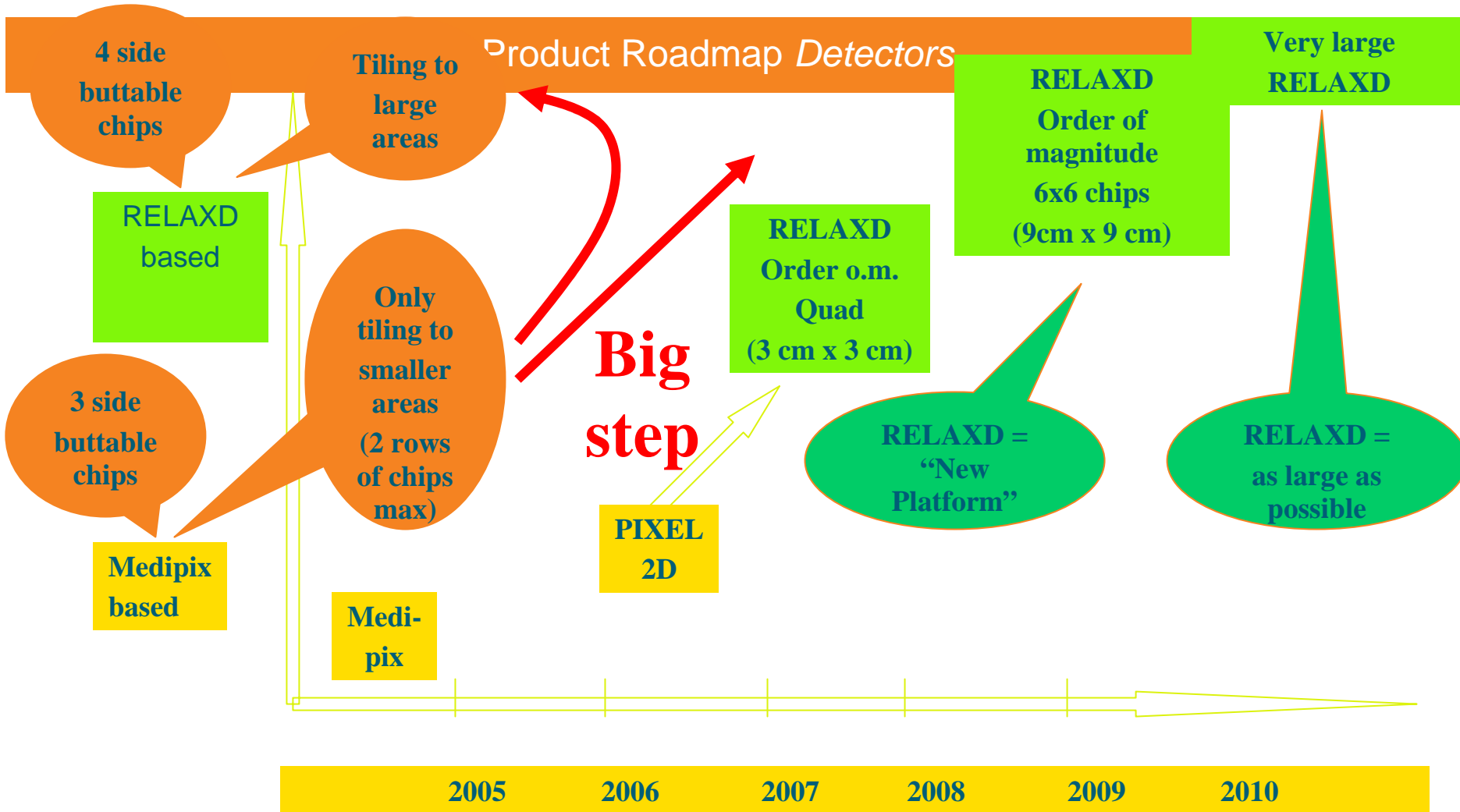
**Plus spin-offs**

**PANalytical  
(+CANBERRA)**



## Product Roadmap *Detectors*





## Detectors

- **Medipix**
  - Mature, may come soon
- **RELAXD**
  - Starts soon
- **RELA-XRD**
  - Prepared in time
- **Question**
  - Who has fun in a collaboration / (different chips)?
  - Who has enthusiasm for another subsidy project ?

## BACKGROUND in Medipix2 CHIP

### EXPOSURE at Panalytical Roelof de Vries

TRACK over ~ 140 PIXELS

RADIUS of CURVATURE  
 $r = 6.9 \text{ mm}$   
PROBABLY MUON  
BENT by EARTH FIELD ?

$B = 5 \times 10^{-5} \text{ T}$

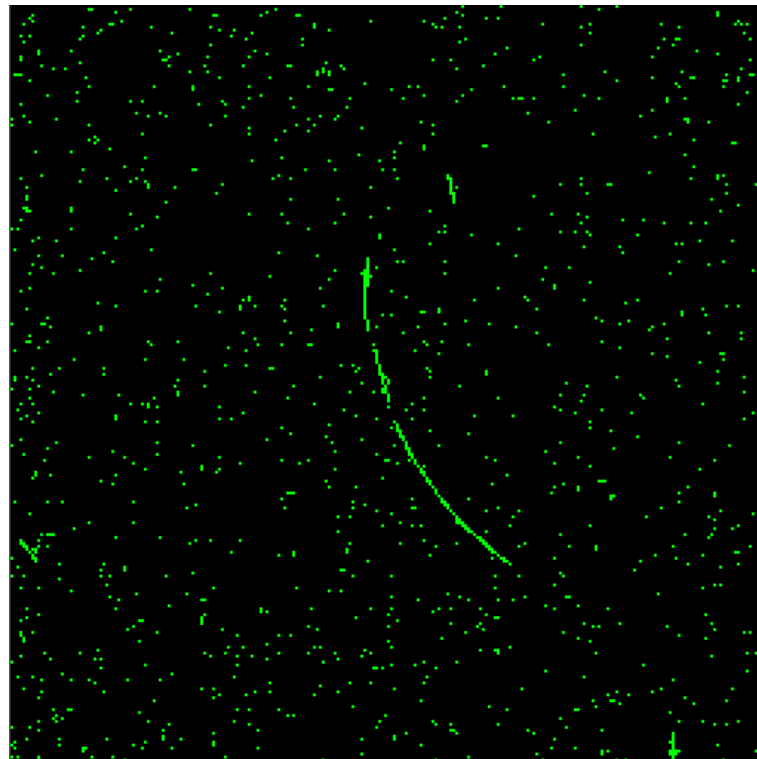
$p = 0.3 B r$

--> momentum

$p \sim 100 \text{ eV}$

(not possible ??)

10 MeV  $\mu$  has range ~5 mm)  
(in reality field higher ?)



Erik HEIJNE CERN PH

MIC Tutorial Seminar 12 January 2005

