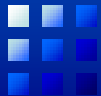


# X線回折顕微法における二次元X線検出器

西野 吉則 (SPring-8 / RIKEN)

RCNP研究会 'マイクロパターン検出器の開発と展望'  
Jan. 26 - 27, 2006

Yoshinori Nishino



# X線を用いた可視化手法(特に、二次元検出器を用いる手法)



## 吸収コントラストイメージング

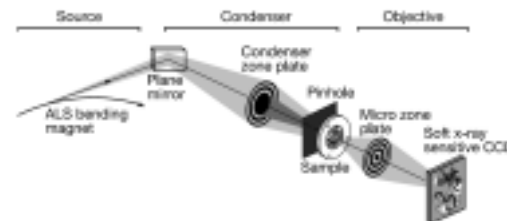
W.C. Röntgen,  
Nature **53**, 274-277 (1896)

プローブ深さ(非破壊透視)



## 位相コントラストイメージング

S.W. Wilkins *et al.*,  
Nature **384**, 335 (1996)



## 結像型顕微鏡

W. Chao *et al.*, Nature **435**, 1210 (2005)

光学(可視光)顕微鏡よりも  
高い空間分解能



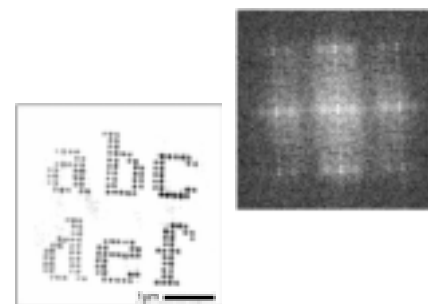
## ホログラフィー

S. Eisebitt *et al.*,  
Nature **432**, 885 (2004)



## X線結晶構造解析

C. Toyoshima *et al.*,  
Nature **405**, 647 (2000)



## X線回折顕微法

J. Miao *et al.*, Nature **400**, 342 (1999).

位相回復 (散乱の逆問題)

コヒーレントX線の利用

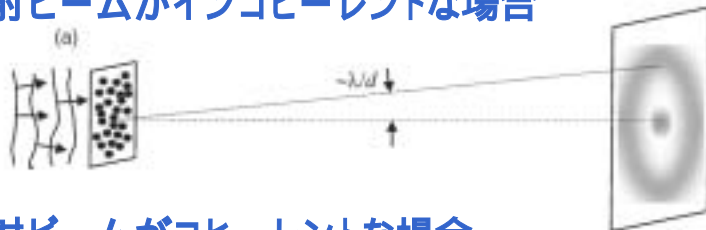


# コヒーレントX線による構造解析

散乱過程は、コヒーレント(弾性)散乱が主要

電子の静止質量エネルギーはX線のエネルギーはよりも大 電子の反跳は無視できる

## ● 入射ビームがインコヒーレントな場合



$$I(K) \propto N$$

散乱強度分布は  
試料の平均構造にのみ依存

## ● 入射ビームがコヒーレントな場合



$$I(K) \propto |\text{FT}[\rho(\mathbf{r})]|^2$$

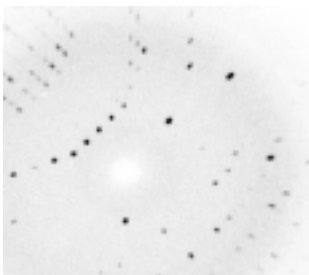
散乱強度分布は  
試料の構造の違いに敏感

N: the number of targets

F. van der Veen, F. Pfeiffer, J. Phys: Condens. Matter **16**, 5003 (2004).

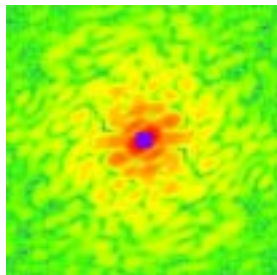
angular size of a speckle  $\sim \frac{\lambda}{a}$  e.g.  $\frac{0.1 \text{ nm}}{1 \mu\text{m}} = 100 \mu\text{rad}$

**結晶試料**



規則的な回折点

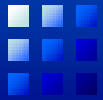
**ランダム試料**



スペックル(斑点)

X線回折顕微法における  
二次元検出器への要請

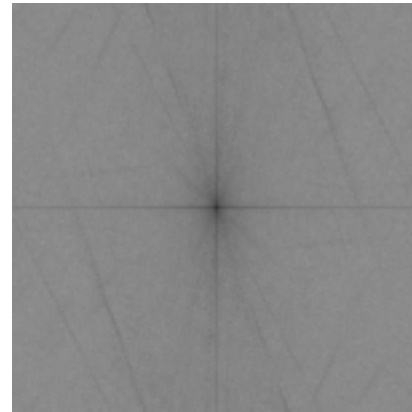
- |           |             |
|-----------|-------------|
| 連続的に分布    | 高い空間分解能     |
| コントラストが高い | 広いダイナミックレンジ |
| 顕微法の高分解能化 | 高い量子効率      |
|           | 大ピクセル数      |



# Simulation



Original Image



Calculated Diffraction Pattern

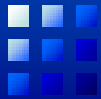
total 2500 x 2000 image 800 x 512 iteration 0



Iterative Image Reconstruction

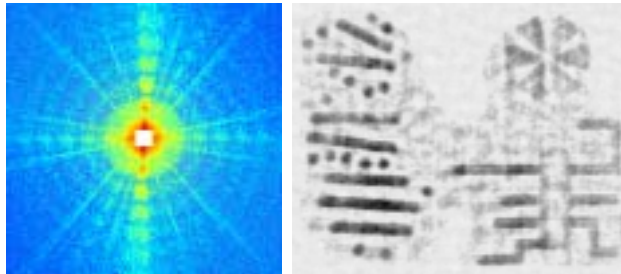


reconstructed image after 5000 iteration



# X-ray Diffraction Microscopy Experiment at SPring-8

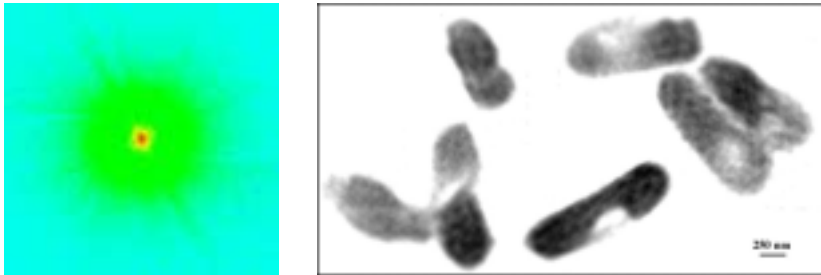
## 硬X線回折強度パターンのみからの世界初の試料像再生



ナノ構造を持つ金製のパターン試料  
( $2.5\ \mu\text{m} \times 2.0\ \mu\text{m}$ )

Y. Nishino, J. Miao, and T. Ishikawa,  
PRB **68**, 220101(R) (2003).

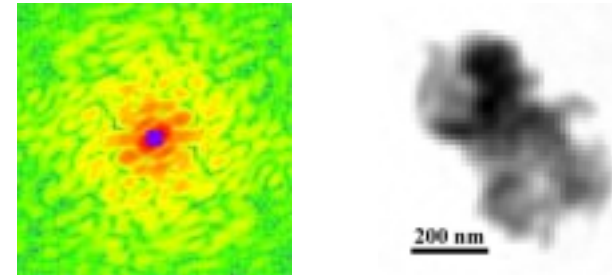
## 生物試料への世界初の応用



大腸菌

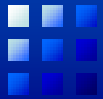
J. Miao, K.O. Hodgson, T. Ishikawa, C.A. Larabell,  
M.A. LeGros & Y. Nishino, PNAS **100**, 110 (2003).

## 材料科学への応用



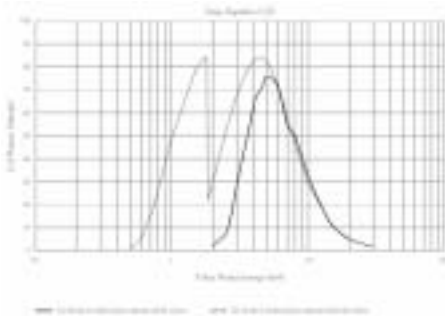
青色発光ダイオードの材料  
窒化ガリウム

J. Miao, Y. Nishino, Y. Kohmura, B. Johnson, C. Song,  
S.H. Risbud & T. Ishikawa, PRL **95**, 085503 (2005).



# Deep-Depletion Direct-Illumination CCD

**量子効率が高い** Deep-Depletion



**空間分解能が高い** Direct Illumination

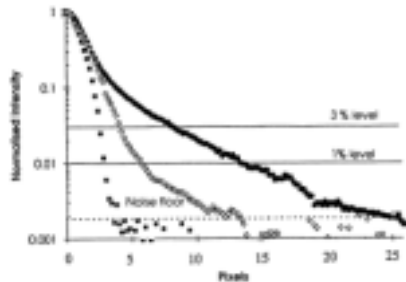
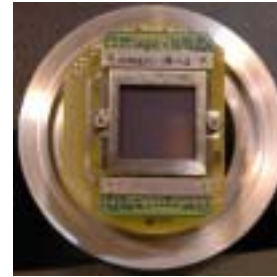


Fig. 2. Point spread functions for etched and sputtered scintillator face-plates. For comparison, the PSF of a direct detection CCD is also shown. Scintillator thickness is 40  $\mu\text{m}$ .

C.M. Castelli, N.M. Allinson, K.J. Moon, D.L. Watson  
NIM A348, 649 (1994)

Princeton Instruments  
PI-LCX:1300  
EEV CCD 36-40 1300  $\times$  1340F



**光子数容量が小さい**

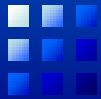
full potential well  $\sim 400 \text{ ke}^-$   
5 keV photon creates 1370  $e^-/\text{hole}$  pairs.

**読み出し速度が遅い**

$\sim 1.8 \text{ s} / \text{full ROI} @ 1 \text{ MHz ADC}$

**大面積化が比較的困難**

顕微鏡の高空間分解能化に必須



# In-Vacuum Imaging Plate Detector

larger area detector  
for higher spatial resolution microscopy

extremely slow in reading (~5 min/full ROI)  
and erasing (>2 min)

Imaging Plate (in Vacuum) }  
Reader and Eraser (in Air) } two sets

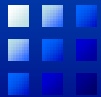
taking data with an imaging plate  
while reading & erasing the other



**R-AXIS VIII (Rigaku Inc.)**

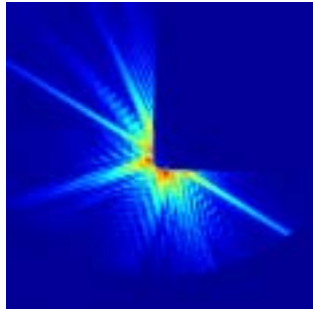
A CCD detector can be mounted downstream of  
the IP detector for quick alignment of the sample and optical components.

	R-AXIS VIII	PI-LCX CCD
Total Area	125 mm square	26 mm × 26.8 mm
Pixel Size	25 μm square	20 μm square
Total Pixel	5000 × 5000	1300 × 1340
Photon Capacity	~10 <sup>5</sup>	~10 <sup>2</sup> (direct illumination w/o phosphor)



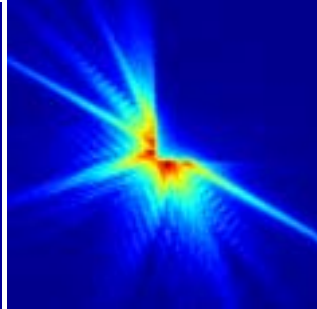
# Evaluation of the Imaging Plate Detector

試料: Pt 単一粒子、粒径  $\sim 4 \mu\text{m}$   
X線エネルギー = 5 keV



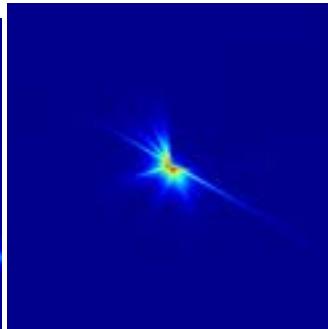
CCD検出器

1001 × 1001 pixels  
0.01 s × 500 = 5 s



IP検出器

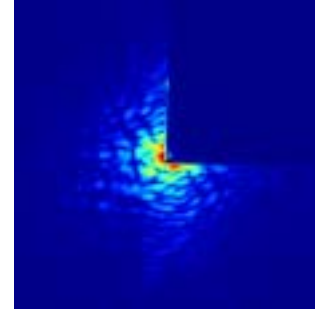
555 × 555 pixels  
10 s × 1 = 10 s



IP検出器

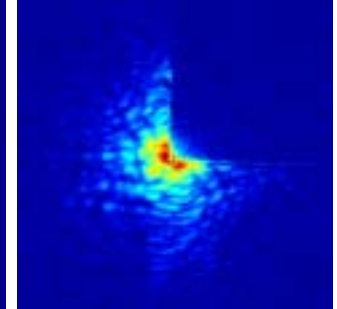
2001 × 2001 pixels  
10 s × 1 = 10 s  
(左と同じデータ)

試料:  $\text{Ge}_{18}\text{Te}_{82}$  単一粒子、粒径  $\sim 1.5 \mu\text{m}$   
X線エネルギー = 5 keV



CCD検出器

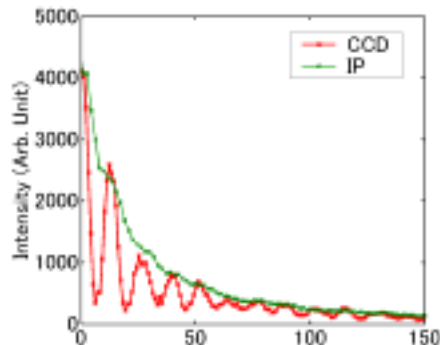
1001 × 1001 pixels  
0.15 s × 500 = 75 s



IP検出器

555 × 555 pixels  
75 s × 1 = 75 s

長く伸びるストリーク上の強度プロファイル

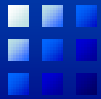


空間分解能が低い

左図: IP位置での強度変動の空間周期  
 $\sim 178 \mu\text{m}$  ( $\sim 7 \text{ pixel}$ )が分解出来ない

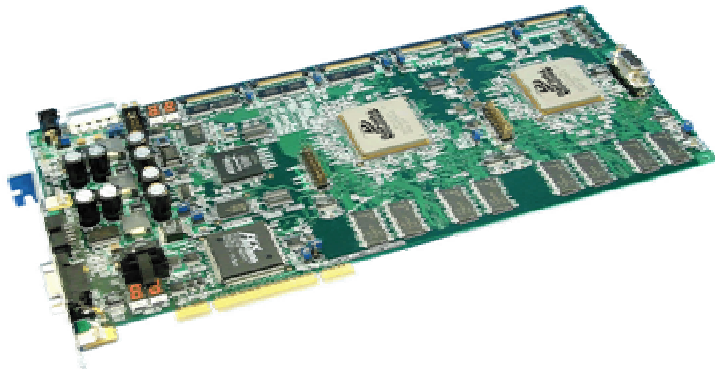
量子効率が低い  
ノイズが大きい



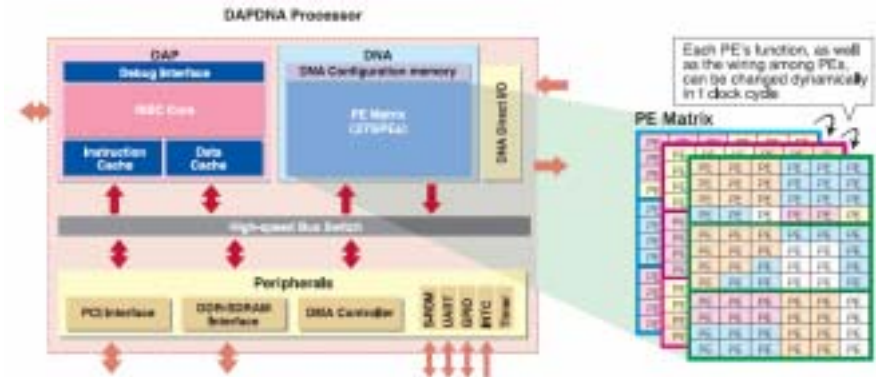


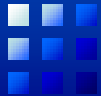
# Dynamic Reconfigurable Processor

- von Neumann architecture ← most computers
  - general purpose hardware
  - application specific software
- ASIC (Application Specific Integrated Circuit)
- FPGA (Field Programmable Gate Array)
  - static reconfigurable
- Dynamic Reconfigurable Processor

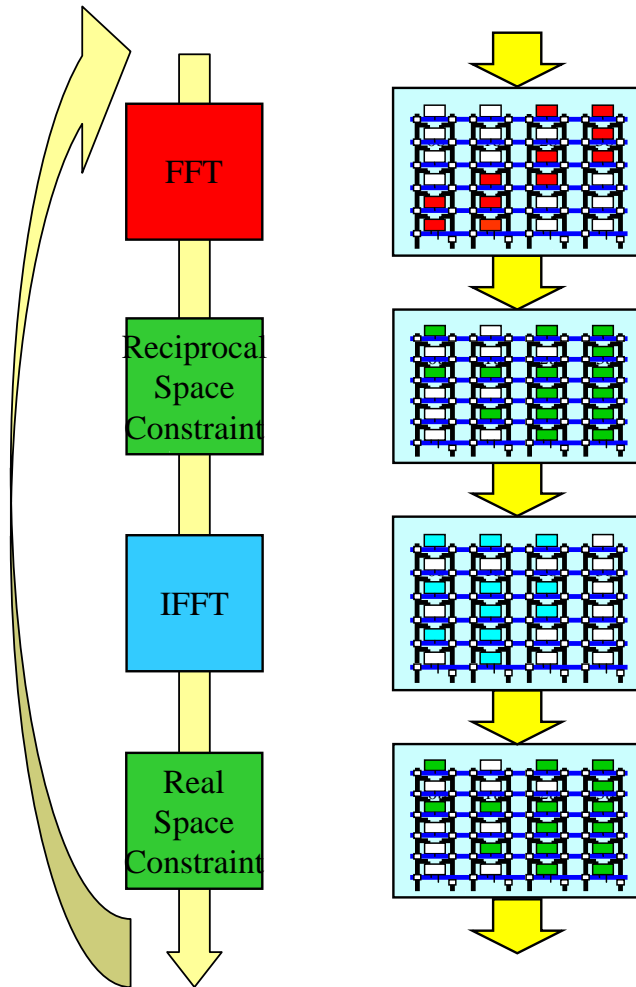


DAPDNA-EB4 (PCI card)  
IP FLEX inc.





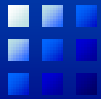
# Image Reconstruction with Dynamic Reconfigurable Processor



reconfiguration in one clock cycle  
~ 6 ns with 166 MHz clock frequency

**FFT / IFFT**  
~ 13 times faster  
than 3.60 GHz Pentium 4 Processor

It takes 87 sec for 1000 iterations of  
 $1024 \times 1024$  pixel image reconstruction



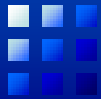
# X線FELでの利用を目指した二次元X線検出器

- Coherent
- Intense  
~  $10^{12}$  photons/pulse
- Short-Pulse  
sub-pico second pulse duration



試料構造のフェムト秒スナップショット/スナップショットムービー

**電荷積分型ピクセル検出器**を候補に検討を始める



# Collaborators

## Experiment at SPring-8 BL29XUL

Yukio Takahashi, Yoshiki Kohmura, Tetsuya Ishikawa (SPring-8/RIKEN)  
Jianwei Miao, Changyoung Song (UCLA)  
Bart Johnson (SSRL/SLAC)

## In-Vacuum Imaging Plate Detector

Yukio Takahashi, Masaki Yamamoto, Tetsuya Ishikawa (SPring-8/RIKEN)  
RIGAKU (<http://www.rigaku.co.jp/>)

## Dynamic Reconfigurable Processor

Kuniaki Koike, Toshikazu Ebisuzaki (RIKEN)  
Tetsuya Ishikawa (SPring-8/RIKEN)  
IP FLEX Inc. (<http://www.ipflex.com/>)

## X-ray Detector for X-Ray FEL (plan)

Atsushi Taketani, Hideto En'yo, Toshikazu Ebisuzaki, *et al.* (RIKEN)  
Hidenori Toyokawa *et al.* (SPring-8/JASRI)  
Tetsuya Ishikawa *et al.* (SPring-8/RIKEN)