Performance of micro pixel gas chamber in small angle X-ray scattering experiments

Kaori Hattori[1,2]

Chihiro Ida[1,2], Kazuki Ito[2], Kotaro Fujii[3], Hidetoshi Kubo[1,2], Kentaro Miuchi[1,2], Masaki Takata[2,4,5], Toru Tanimori[1,2], Hidehiro Uekusa[3]

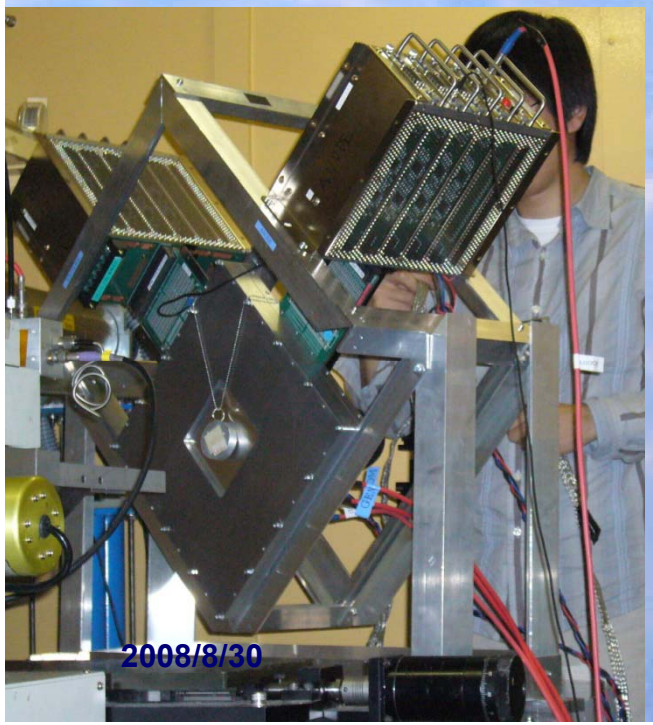
[1]Department of Physics, Kyoto University, Japan

[2] Structural Materials Science Laboratory, RIKEN Harima Institute/SPring-8 Center, Japan

[3] Department of Chemistry and Materials Sicence, Tokyo Institute of Technology, Japan

[4] SPring-8/JASRI, Japan

[5] Department of Advanced Materials Sciences, Graduate School of Frontier Sciences, The University of Tokyo



OUTLINE

- Development of detectors for structural determination
- Requirements for photon counting detectors
- Novel photon counting detector,

  mico-pixel chamber (μ-PIC)
- Time resolved experiments
- Small angle X-ray scattering (SAXS) experiments
- Summary

To provide powerful methods for structural determination

- **High speed**

Structural analysis of biological macromolecules (protein) materials

→ radiation within a couple of minutes

- **High precision**

→ wide dynamic range of $>10^7$

→ realize high precision measurements

→ Structural determination of materials with light elements

- **Time resolved**

active dynamics

photon-induced phase transition

record continuum transition with a time resolution of sec to sub-msec

repeated measurements will provide better time resolution

To satisfy these conditions

Photon counting detectors with good position resolutions are suitable

Requirements for two-dimensional photon counting detectors

1. Position resolution better than 100 μm
2. Counting rates > $10^7 mm^{-2}$, >1000 × MWPC (irradiated locally)
3. Large active area of > 150 × 150 mm^2
4. No dead region (ex. junctions)
5. Efficiency difference < 1 %
6. Image distortion < 1 %
7. Operation at room te
8. Easy maintenance
9. Low costs

Readouts without intervals
→CRP (continuous rotation photograph) method
High gain
→sensitivity to low energy X-rays of about 1 keV
Anomalous X-ray scattering of sulfur (2.3keV)

A photon counting area d

has realized 4, 6, 7, 8, and 9.

1, 2, and 5 are in progress.

3. A n active area of a μ-PIC currently in use is 100 × 100 mm^2

A μ-PIC with an active area of 300 × 300 mm^2 has proved stable runs.

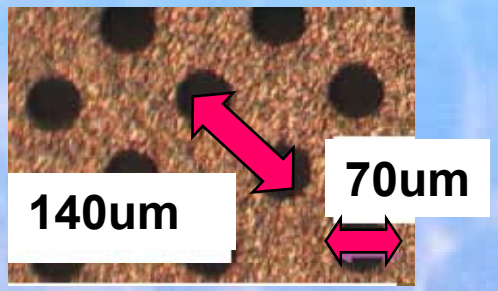
→ Verification experiments at a synchrotron radiation facility are being planned.

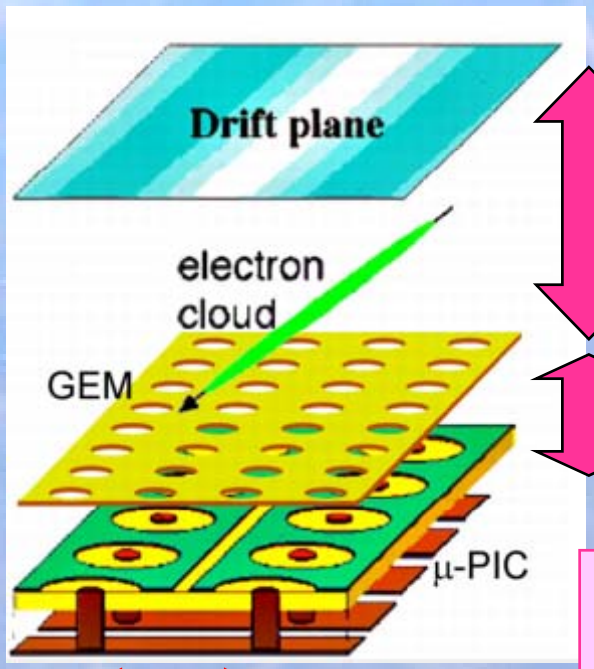
Novel gas detector
μ-PIC (micro-Pixel Chamber)

Mechanism for photon detection

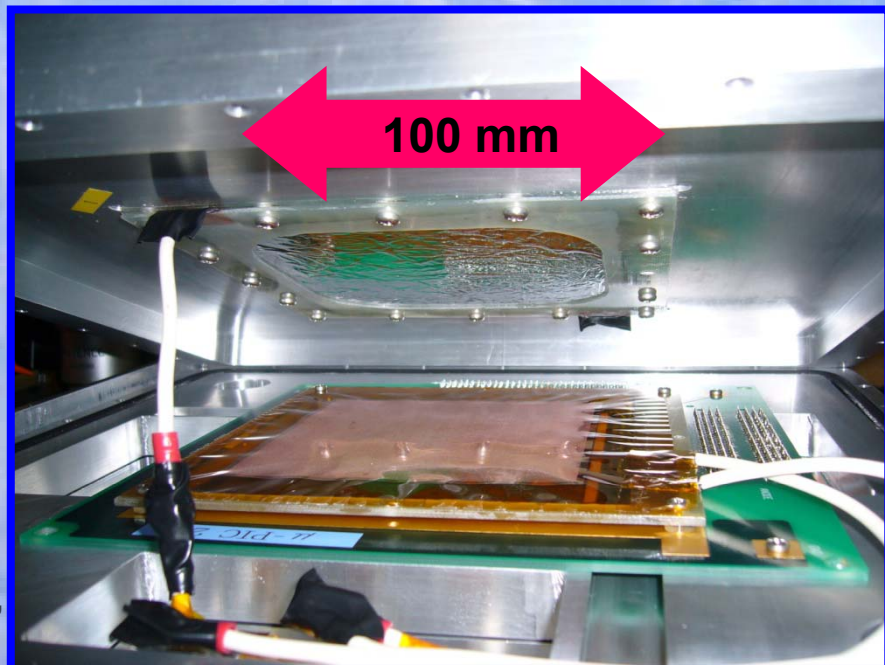
- ✓Photoelectric effect in a gas
- ✓Emitted electron runs until it loses a kinetic energy
- ✓Ionizes atoms
- ✓Electron clouds are amplified by a GEM(gas electron multiplier, F. Sauli, 1997) , and μ-PIC

GEM
(gas electron multiplier)





Pixel pitch
400 μm



Gas gain

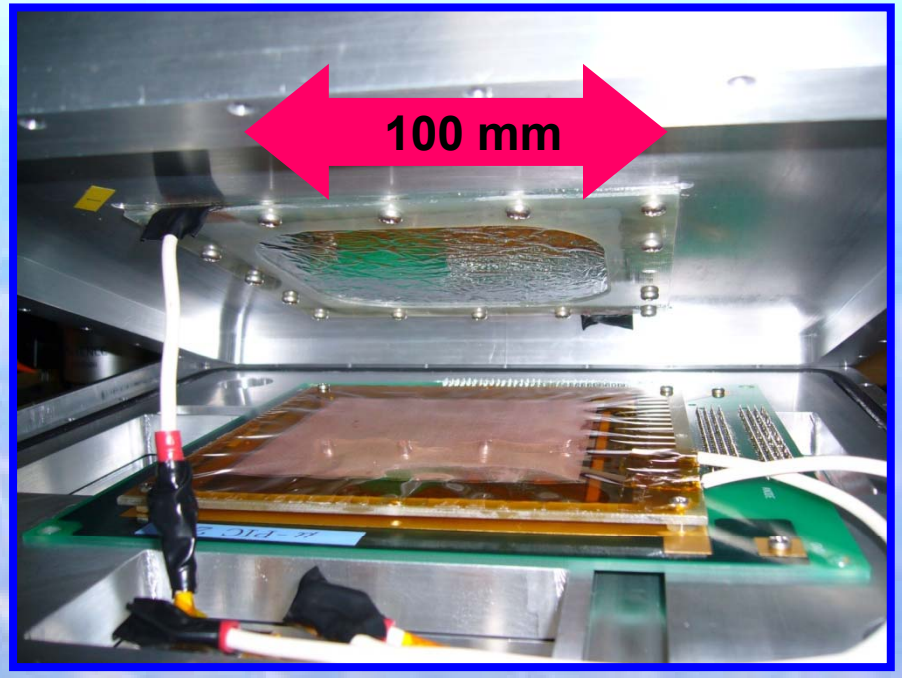
μ-PIC : 3×10^4 GEM: 3

IUCr 2008 Osaka,

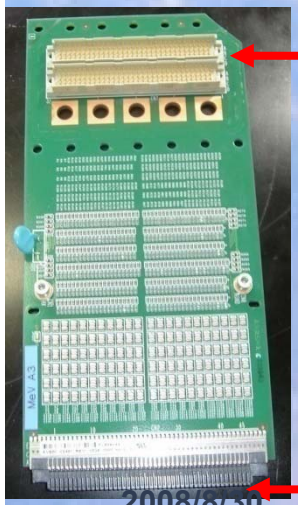
μ-PIC is kept in the sealed vessel

✓The μ-PIC is contained in a sealed vessel with a polyimide entrance window of 0.1-mm thickness.
✓The vessel is filled with Xe-C_2H_6(70:30) gas.
✓Stable operation without fresh gas supply for > 1 month

Anode 256ch + cathode 256ch
Signals from the μ-PIC are sent via the printed circuit boards

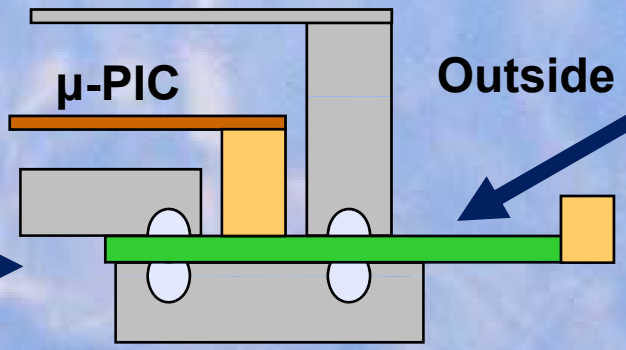


Sealed vessel



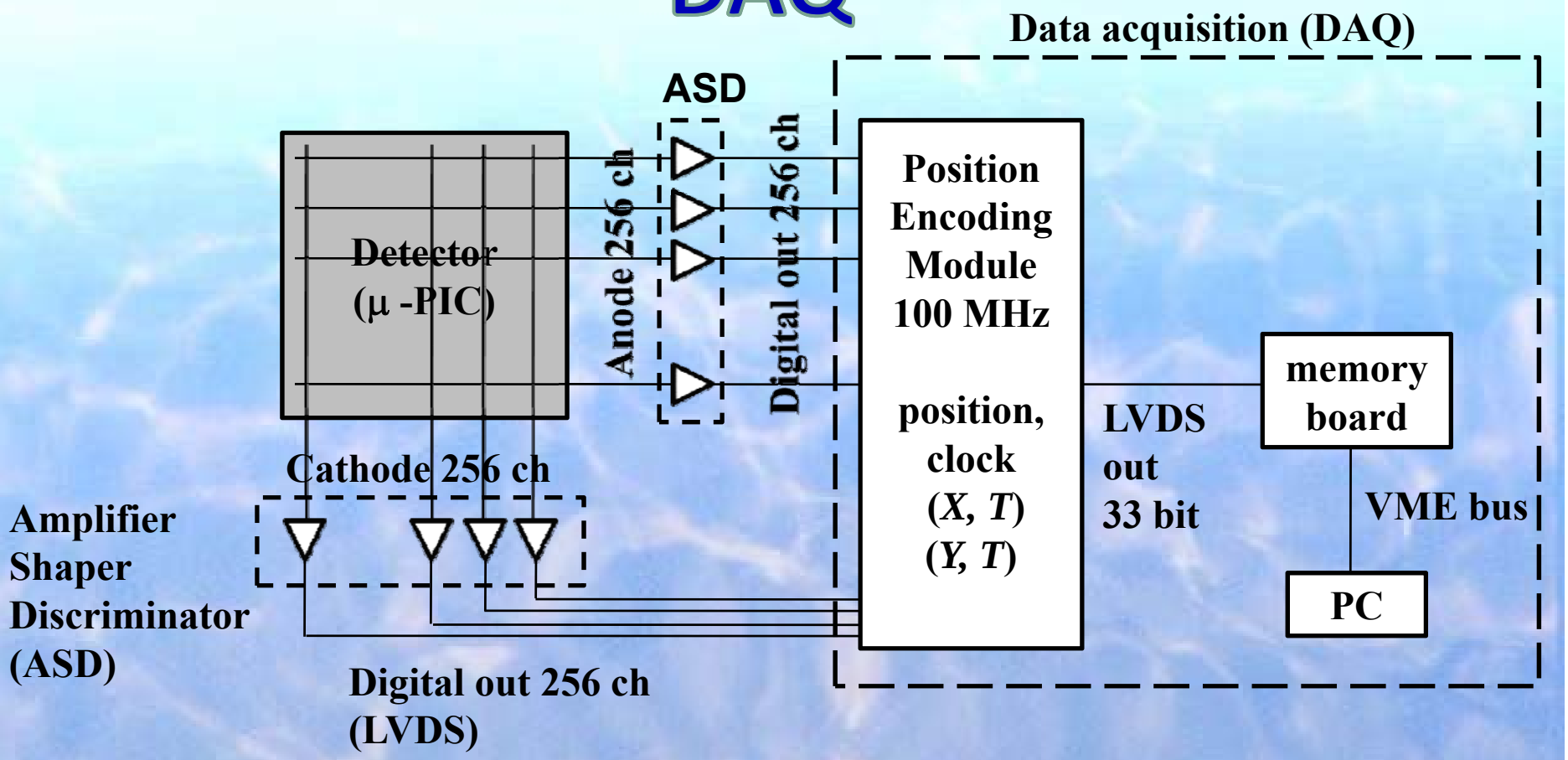
from μ-PIC

256ch per board



to pre-amplifiers

DAQ

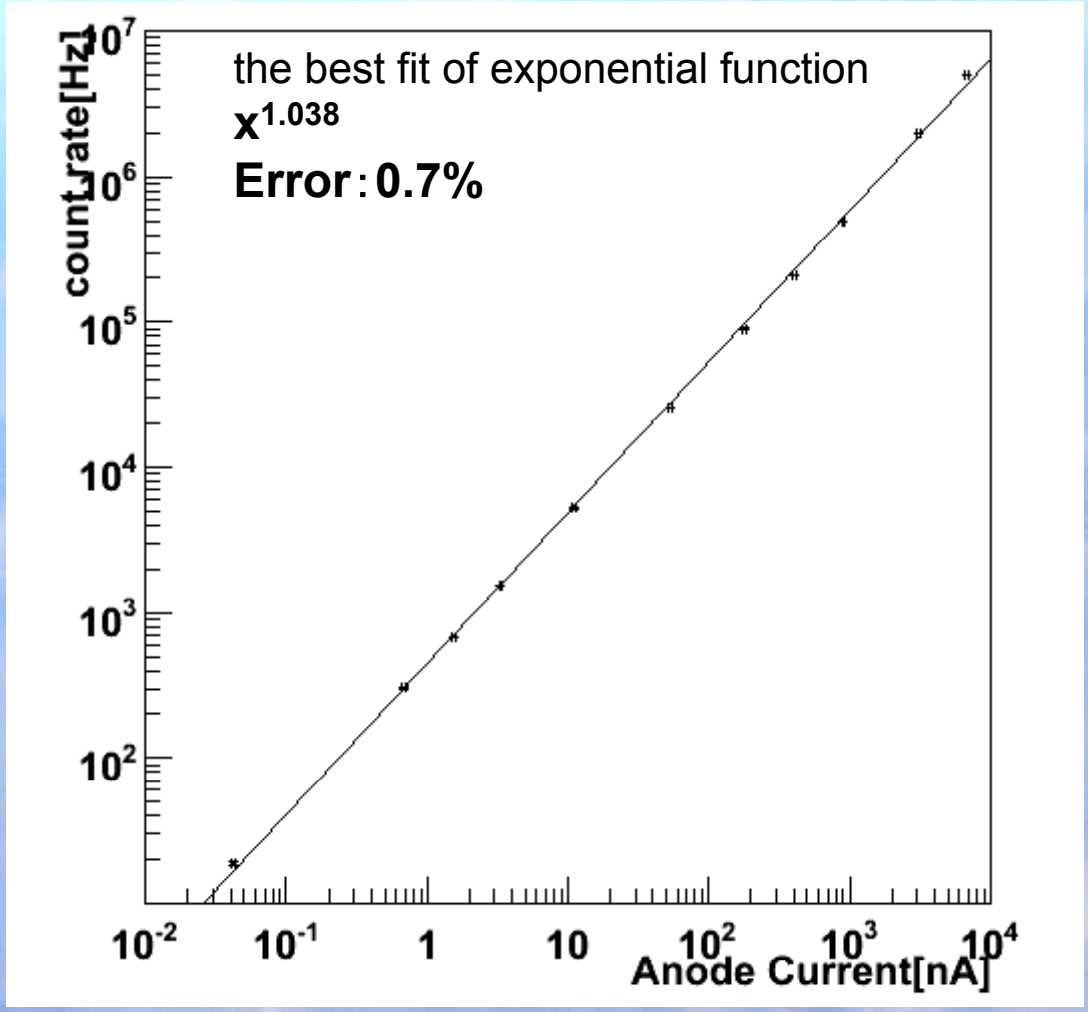


The output charges of the 256+256 channels are parallel pre-amplified, shaped, and discriminated by the ASD chips → completely digitized Digital signals are sent to the position encoding module with an internal clock of 100 MHz, allowing the recording of position (*X* or *Y*) and the timing *T* in the memory module

Advantages of digital readouts

- Simple
- Low cost
- Easy adjustments for detectors with large active area
- Fast readout
- Characteristic less depends on counting rates
- Good counting rate capabilities
- μ-PIC > 1 MHz ⟷ charge division < 1 MHz
  delay line

Linearity in data acquisition rates



Irradiated scattering from a piece of glassy carbon 0.9 Å

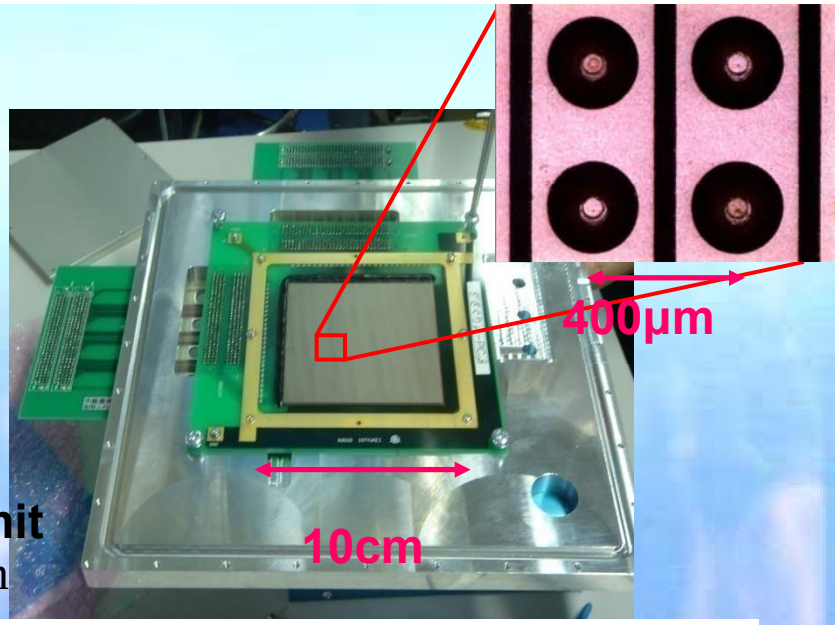
good linear correlation from 20 cps to 5 Mcps

Dynamic range of > 10^5
No saturation

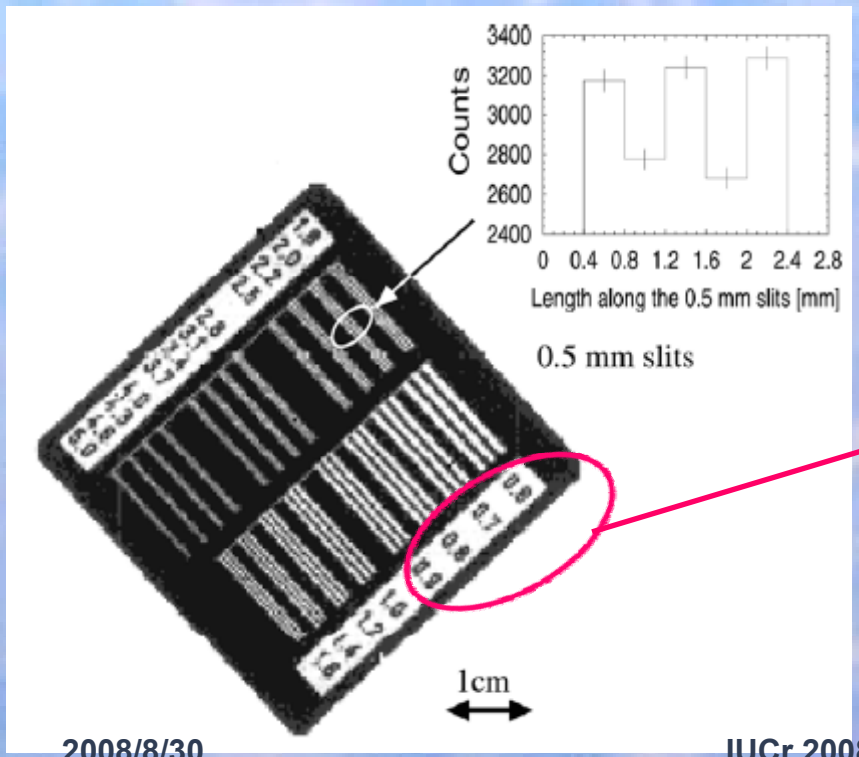
counting rates are limited by a high voltage module

Performance of μ-PIC

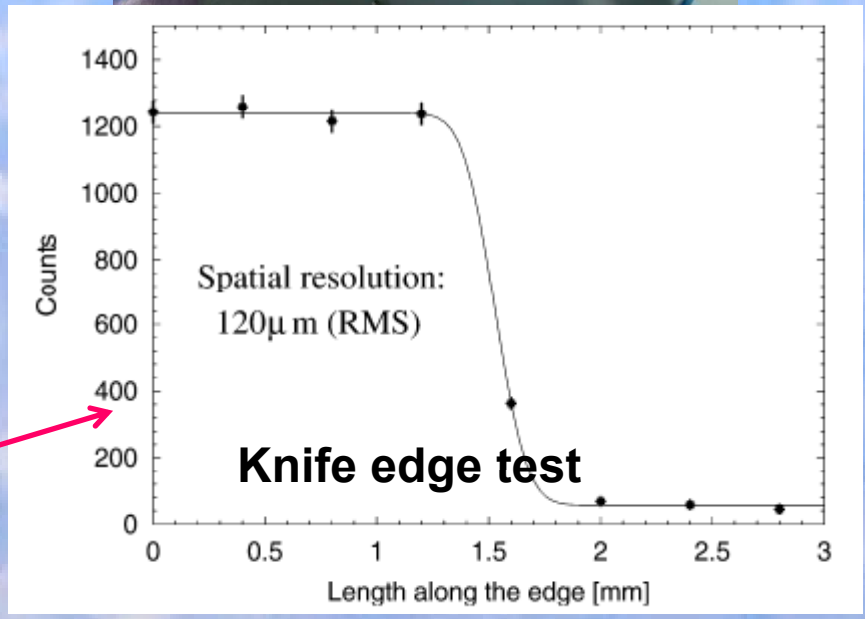
2-dimensional imaging gaseous detector
pitch 400μm,
size 100 mm × 100 mm, 300 mm × 300 mm



position resolution ~ 120μm ⟵ **Theoretical limit** $\sigma = d/\sqrt{12} = 115\mu\mathrm{m}$



X-ray image of test chart and the projected image along 0.5 mm slits

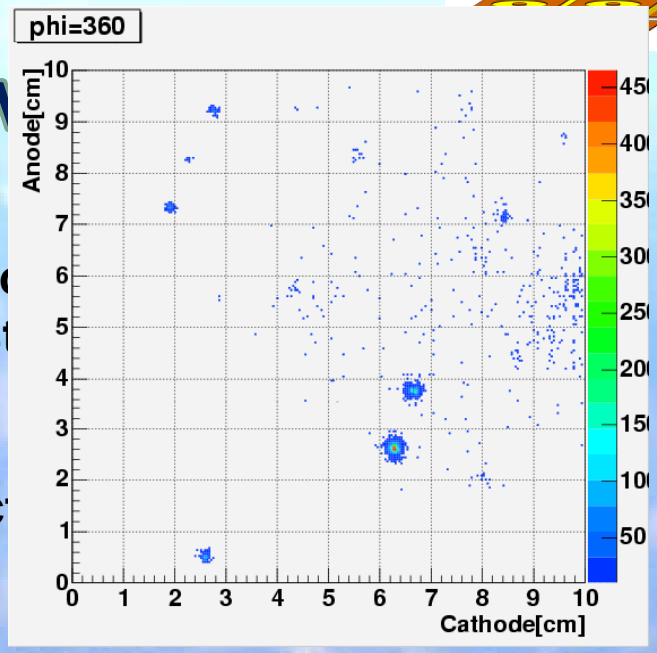


Projected image of the test chart edge and the best fit of the error function

A. Takeda et al., IEEE Transactions on nuclear science, Vol.51, No.5, (2004)

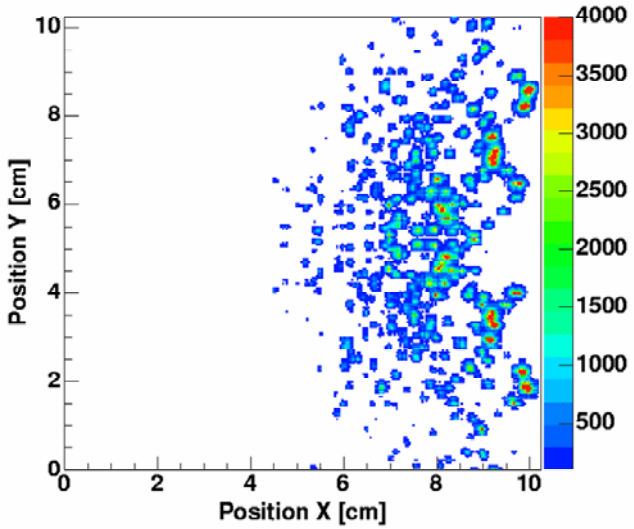
μ-PIC as a time-resolv

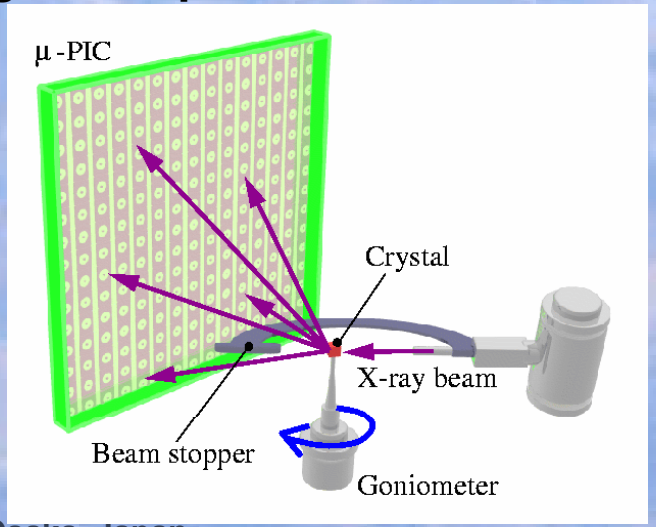
CRP (continuous rotation photograph) meth
Movie of diffraction spots from rotating cryst
a crystal rotated by a goniometer
timings of incident photons
converted to rotation angles of diffrac



Reducing the measurement time
Strong background reduction using a new parameter, rotation angle

- integrated diffraction spots

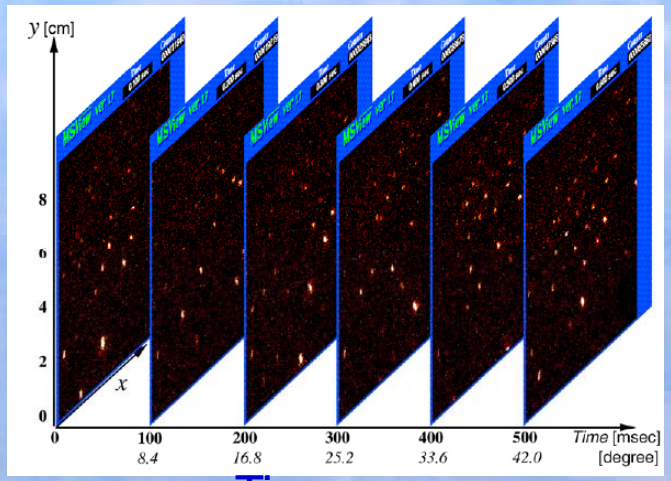




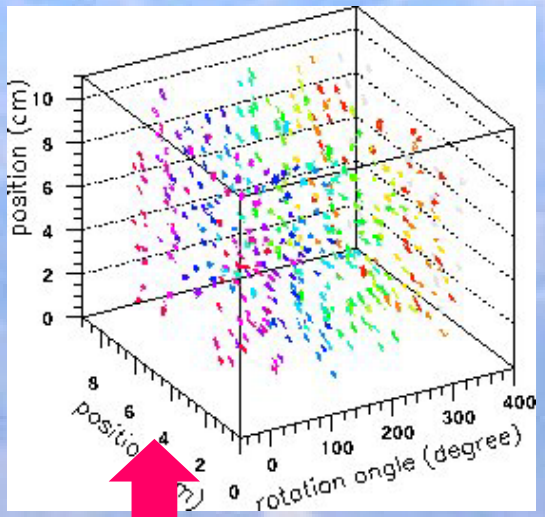
Time Resolved X-ray Crystal Structure Analysis(1999)

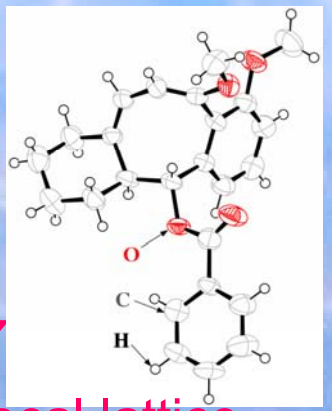
	Crystal	Ref. #	R-factor (I > 2σ)	time (sec.)
	$C_4H_9NO_6$	1,406	7.9%	2.1
	$C_{20}H_{37}CoN_6O_4$	4,361	9.8%	300
	$C_{25}H_{26}O_4$	4,565	8.4%	80

MSGC(Micro Strip Gas Chamber**)**

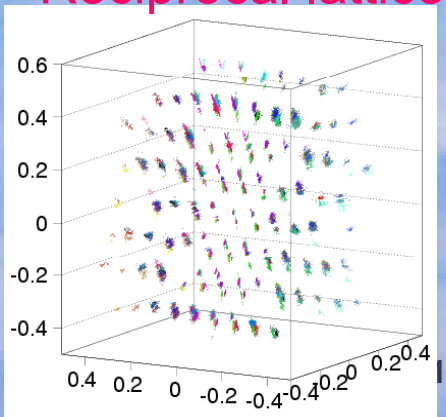


Time





Reciprocal lattice

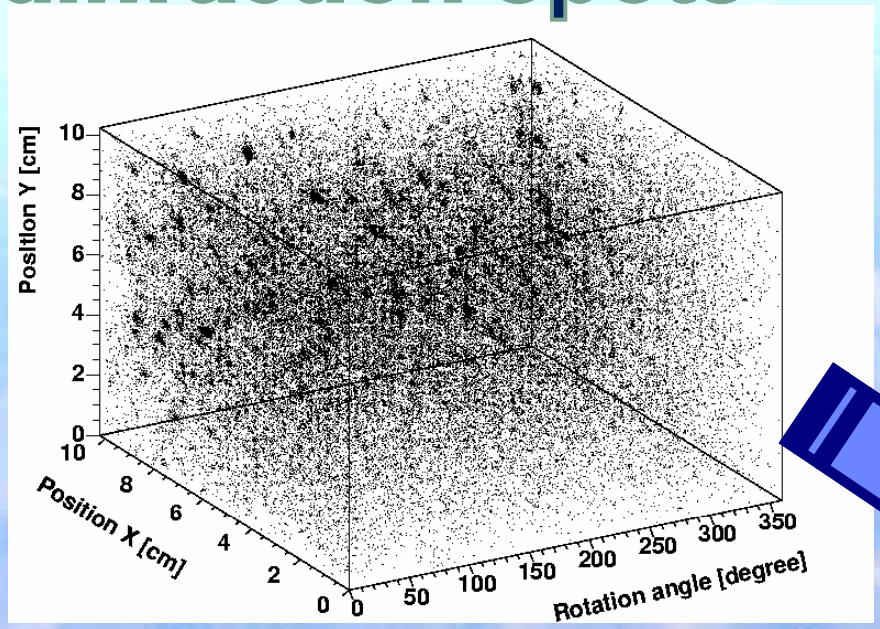


Movie varying 2θ continuously

Time resolution of ~ 100 ns for each X-ray

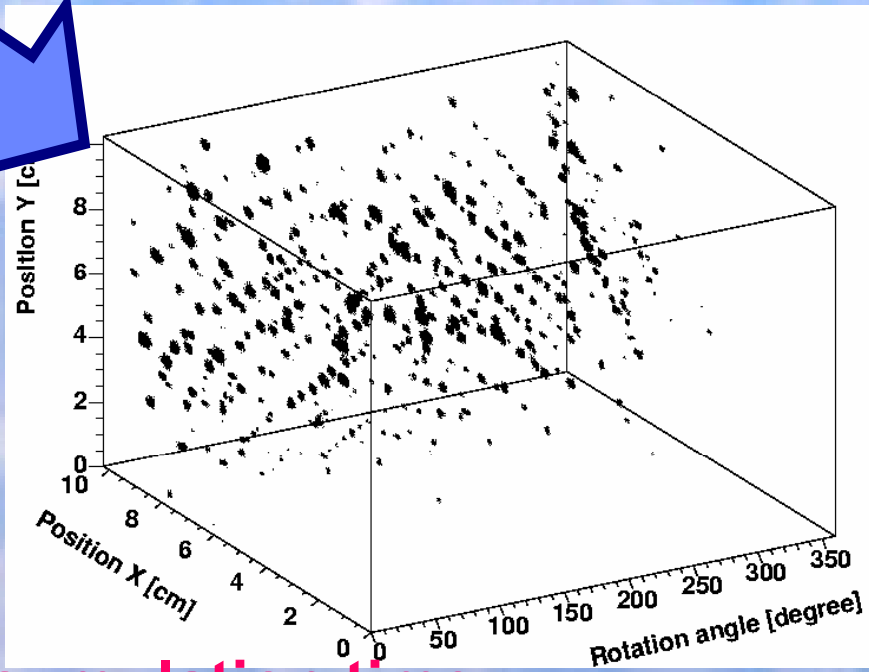
Much Information -> quick online analysis

μ-PIC: 3-dimensional image of diffraction spots



rotation speed : 4.89 sec/cycle
mesurement time : 3716 sec
counting rate : 1.05×10^4 cps

Applying the noise reduction using 2θ information

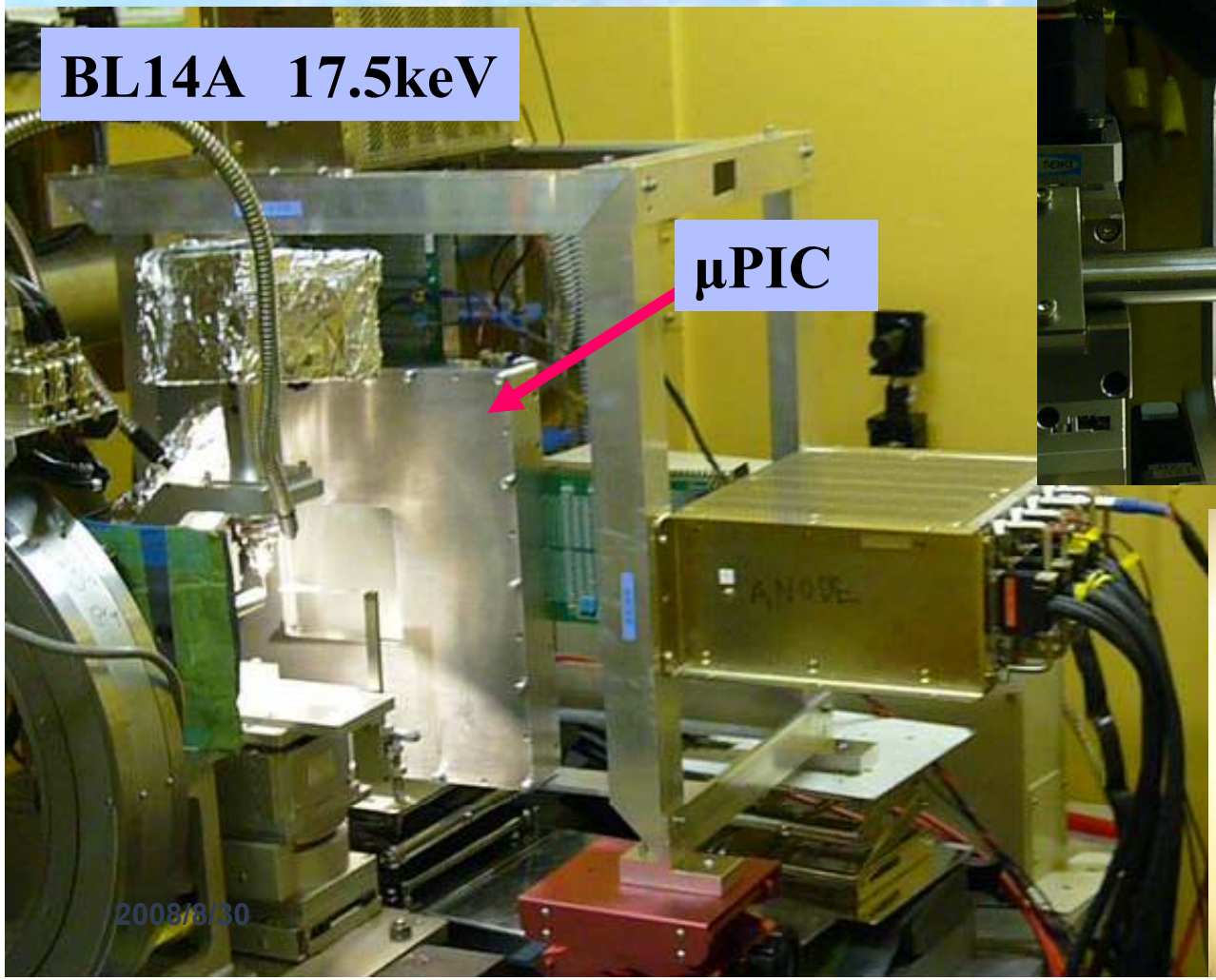


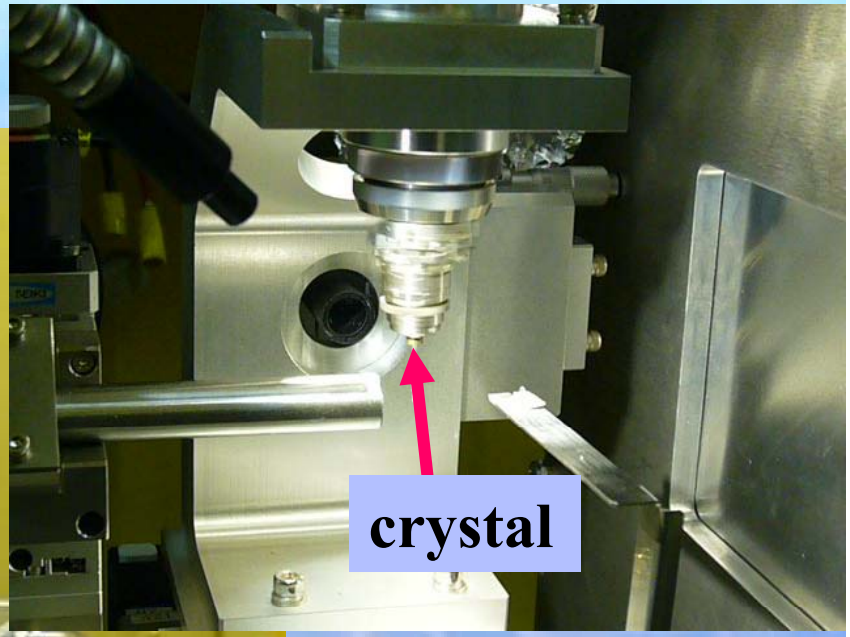
3716s $2\theta<49^o$
Reflections 1556 (331 unique)
Rint (internal agreement factor)3.7%

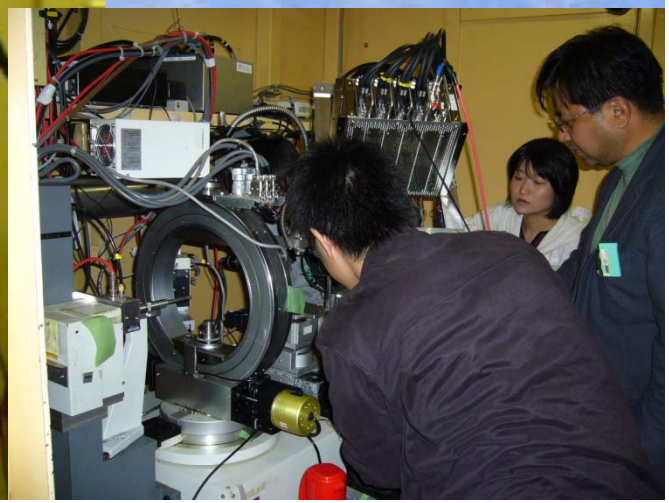
Rint of 1% will achieve
with ten times the length of accumulation time

Takeda et al. J. Synchrotron Rad. (2005) 12, 820-825

Single crystal diffraction and powder diffraction study performed at KEK-PF, Japan

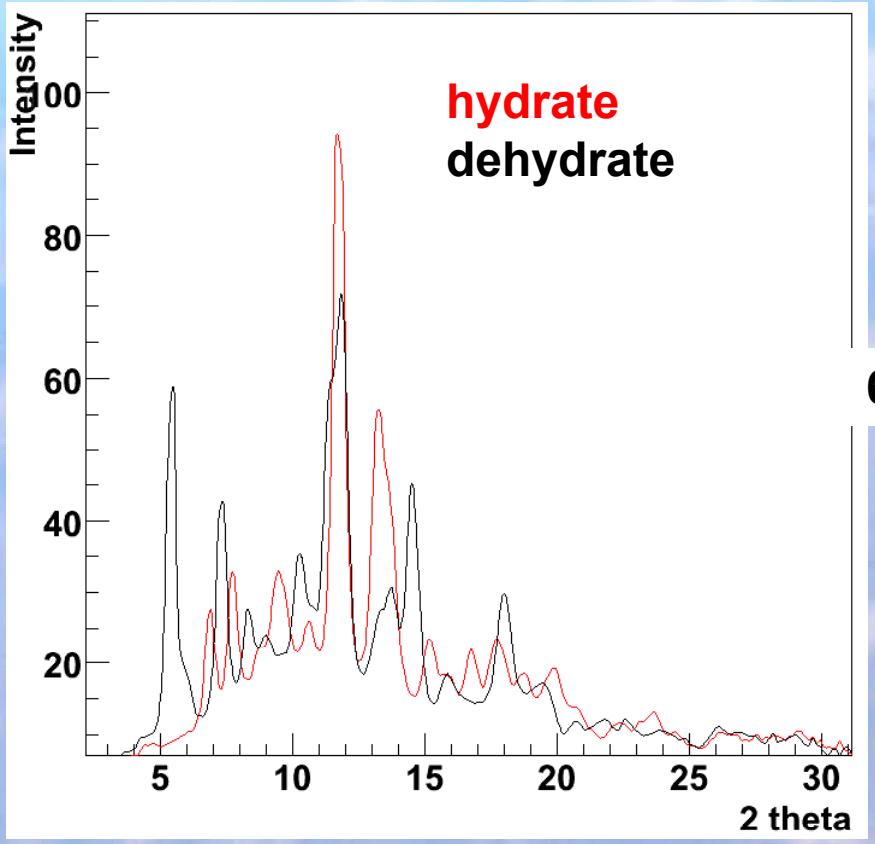


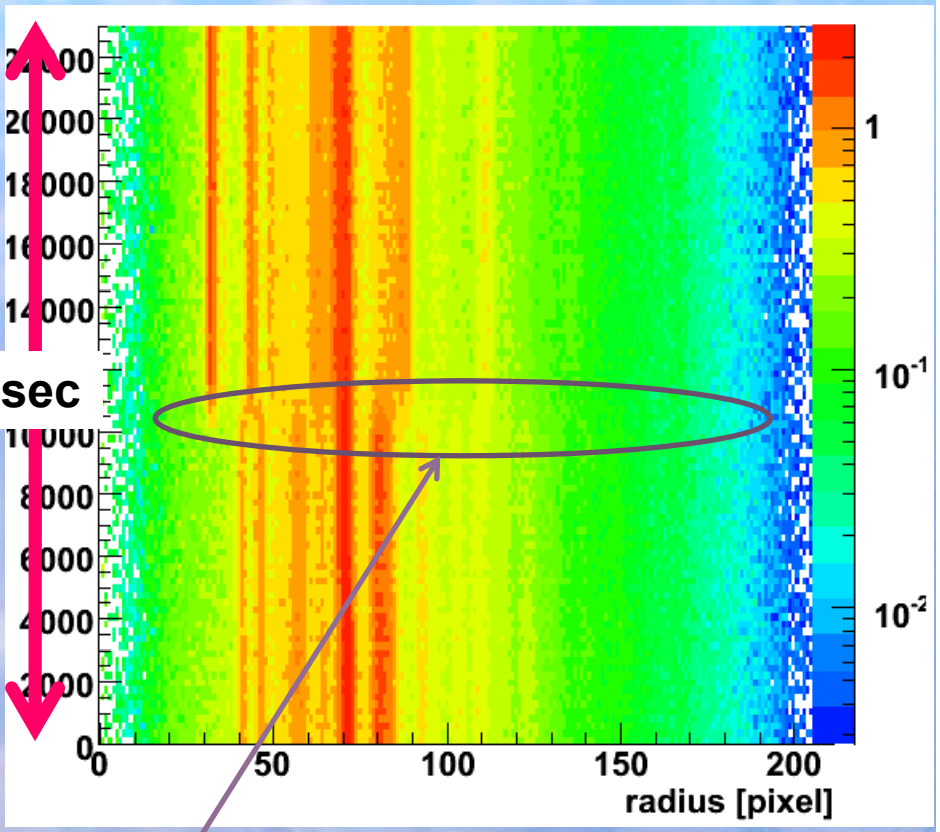




Powder diffraction changing temperature (1)

KEK Photon Factory 0.7 Å
Dehydration reaction of a pyromellitic acid hydrate occurs while heat is applying (140℃)

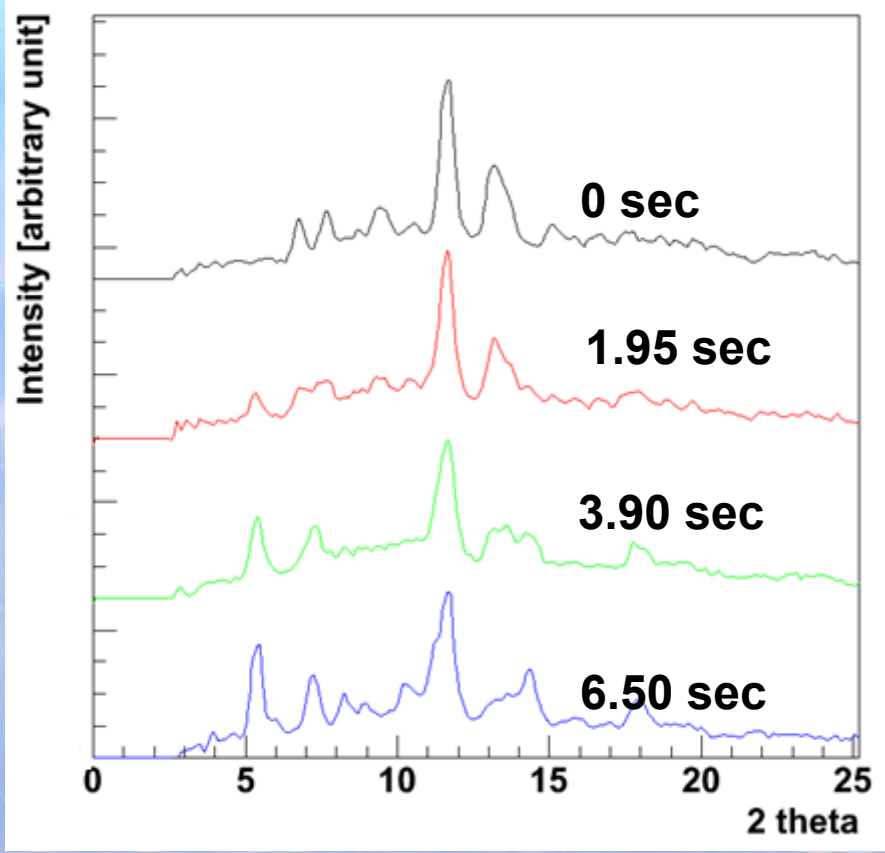


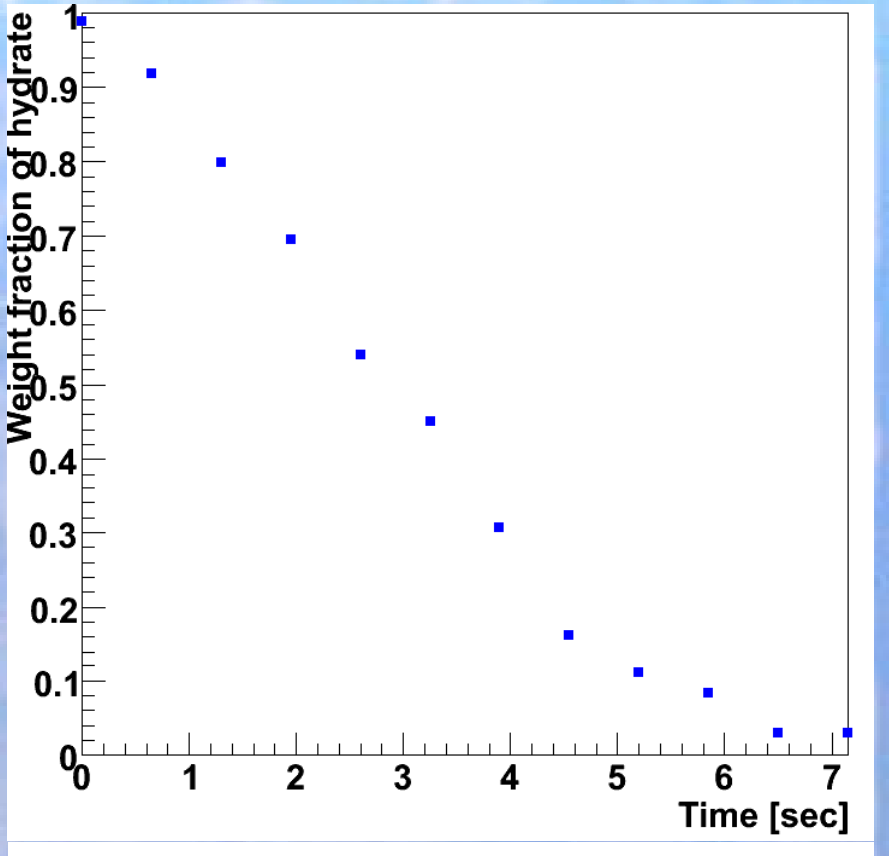


Change in a diffraction pattern in 7 sec

Powder diffraction changing temperature (2)

The intensity $I(2\theta, t)$ is expressed as $I = xI_d(2\theta) + (1-x)I_h(2\theta)$, where $I_d(2\theta)$, $I_h(2\theta)$ is the intensity of the dehydrate, the hydrate, respectively, including a background

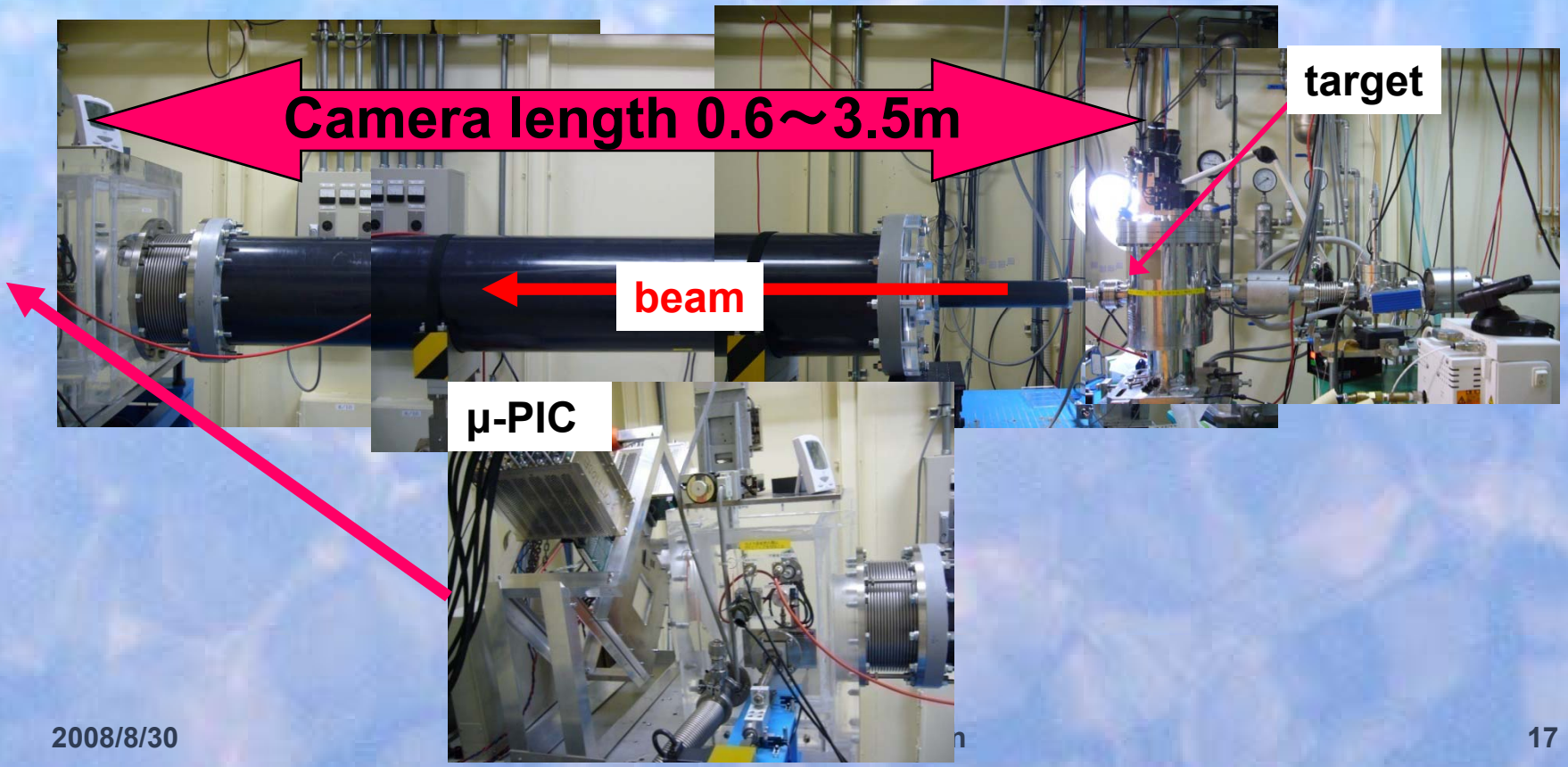




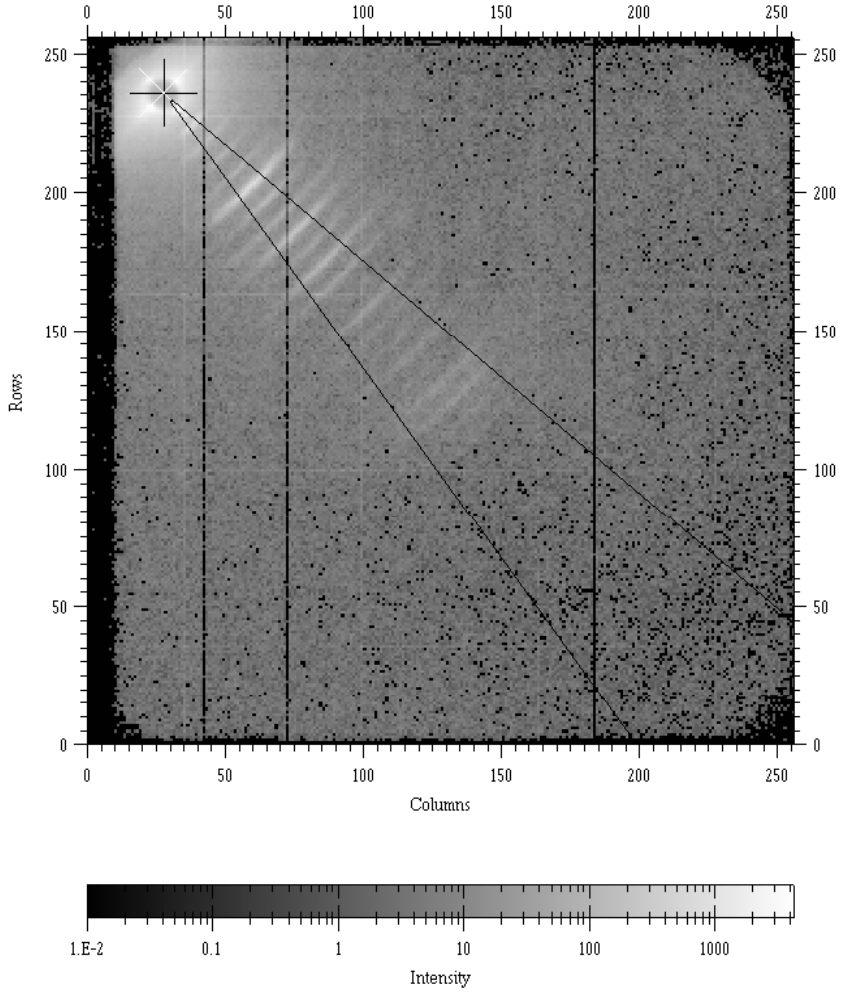
4.3×10^4 events / 0.65 sec

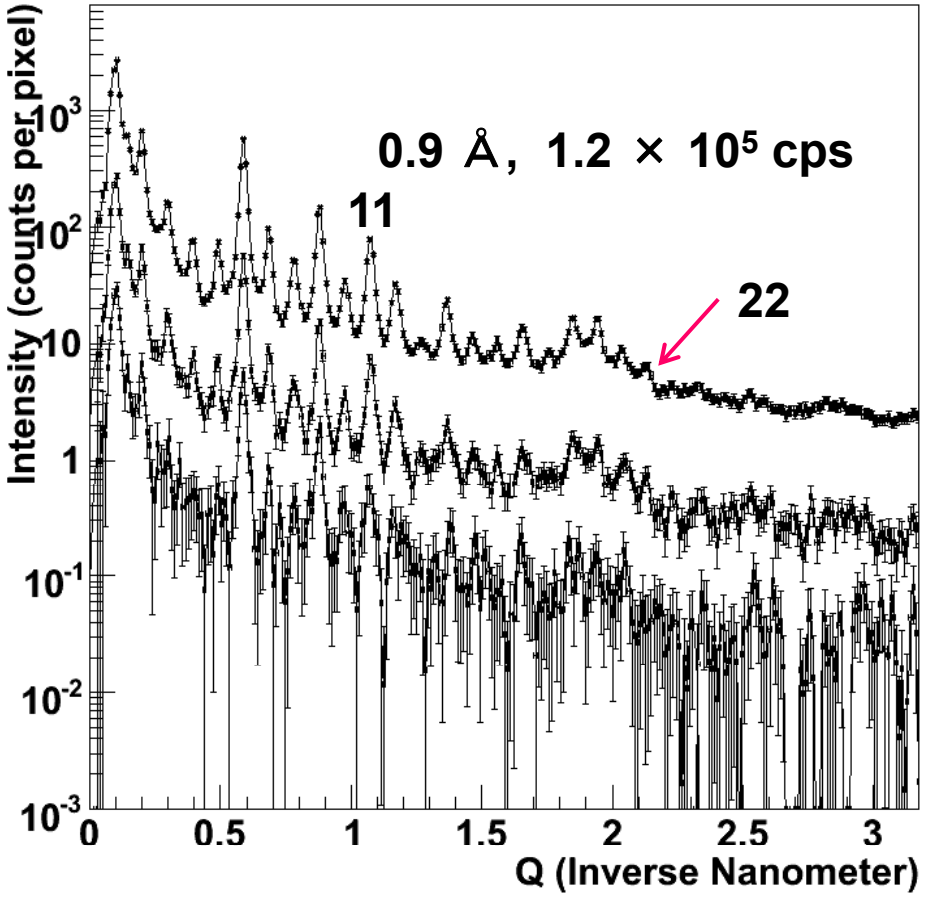
Time resolution will be expected to about 4 msec with a count rate of 10MHz

Small Angle X-ray Scattering experiments conducted at SPring-8 BL45-XU SAXS station



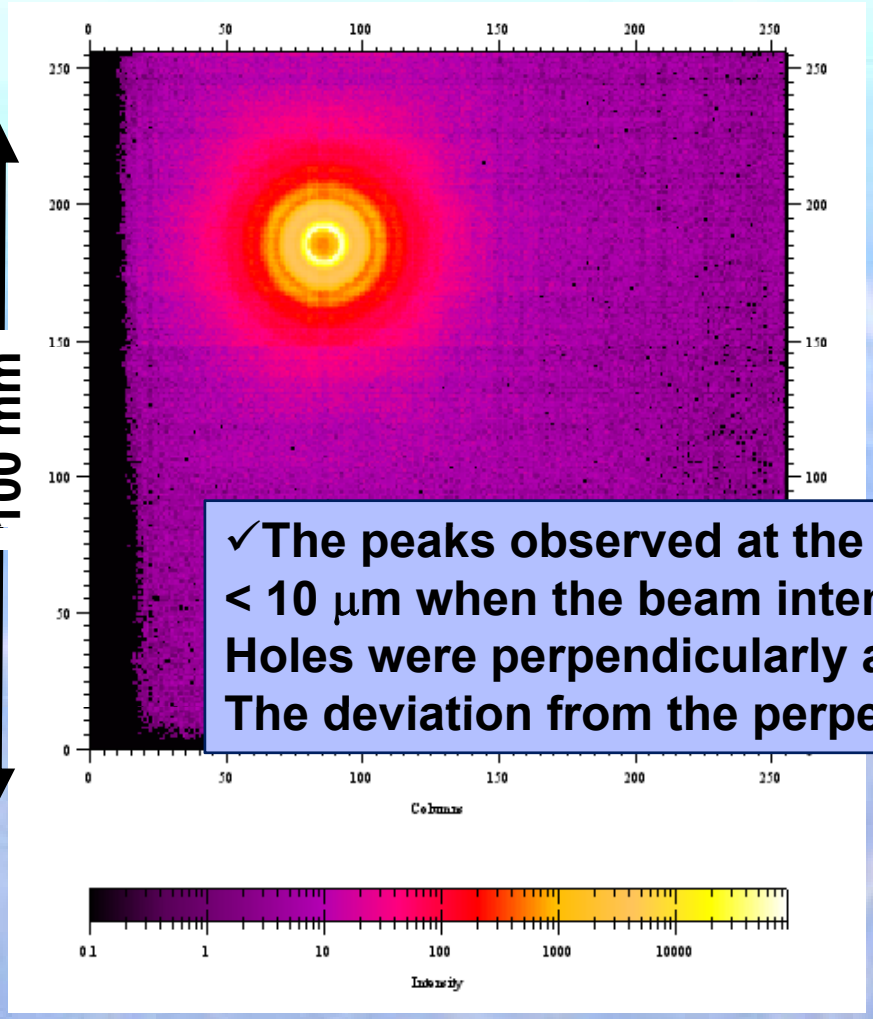
Diffraction pattern of collagen

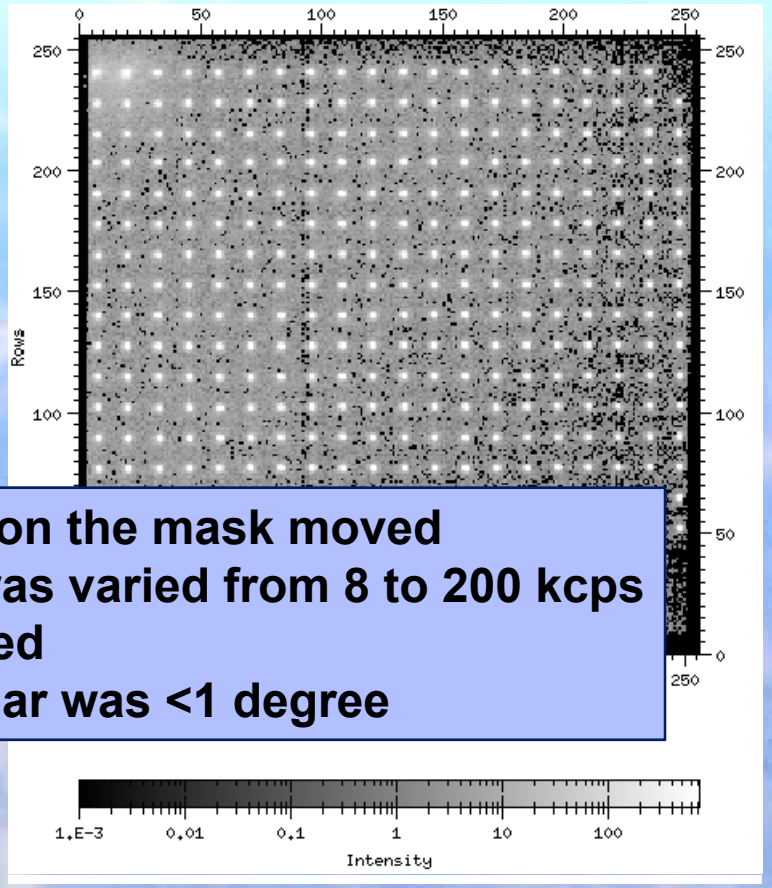




Diffraction patterns of collagen with a μ-PIC X-ray imaging system with an accumulation of 10^6 events, 10^5 events, and 10^4 events, respectively. Signal-to-noise ratio in the background of the diffraction pattern was improved

Powder diffraction in SAXS





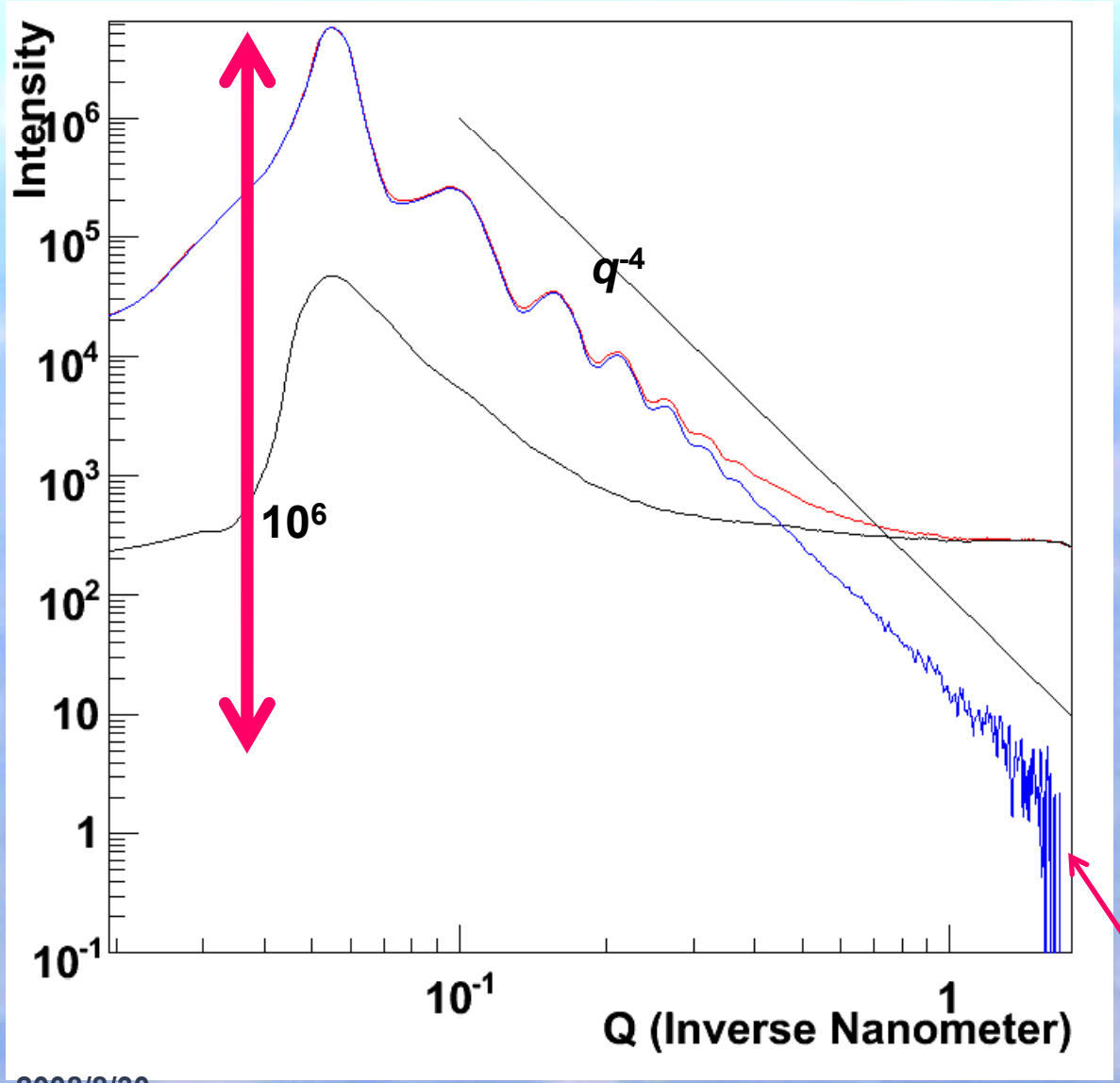
✓The peaks observed at the holes on the mask moved < 10 μm when the beam intensity was varied from 8 to 200 kcps
Holes were perpendicularly arranged
The deviation from the perpendicular was <1 degree

irradiating a grid mask with scattering from a piece of glassy carbon, 1.5 Å

solution scattering from polystyrene latex (110 nm, 5 mg / ml), 1.5 Å

No spatial distortion was observed

Dynamic Range



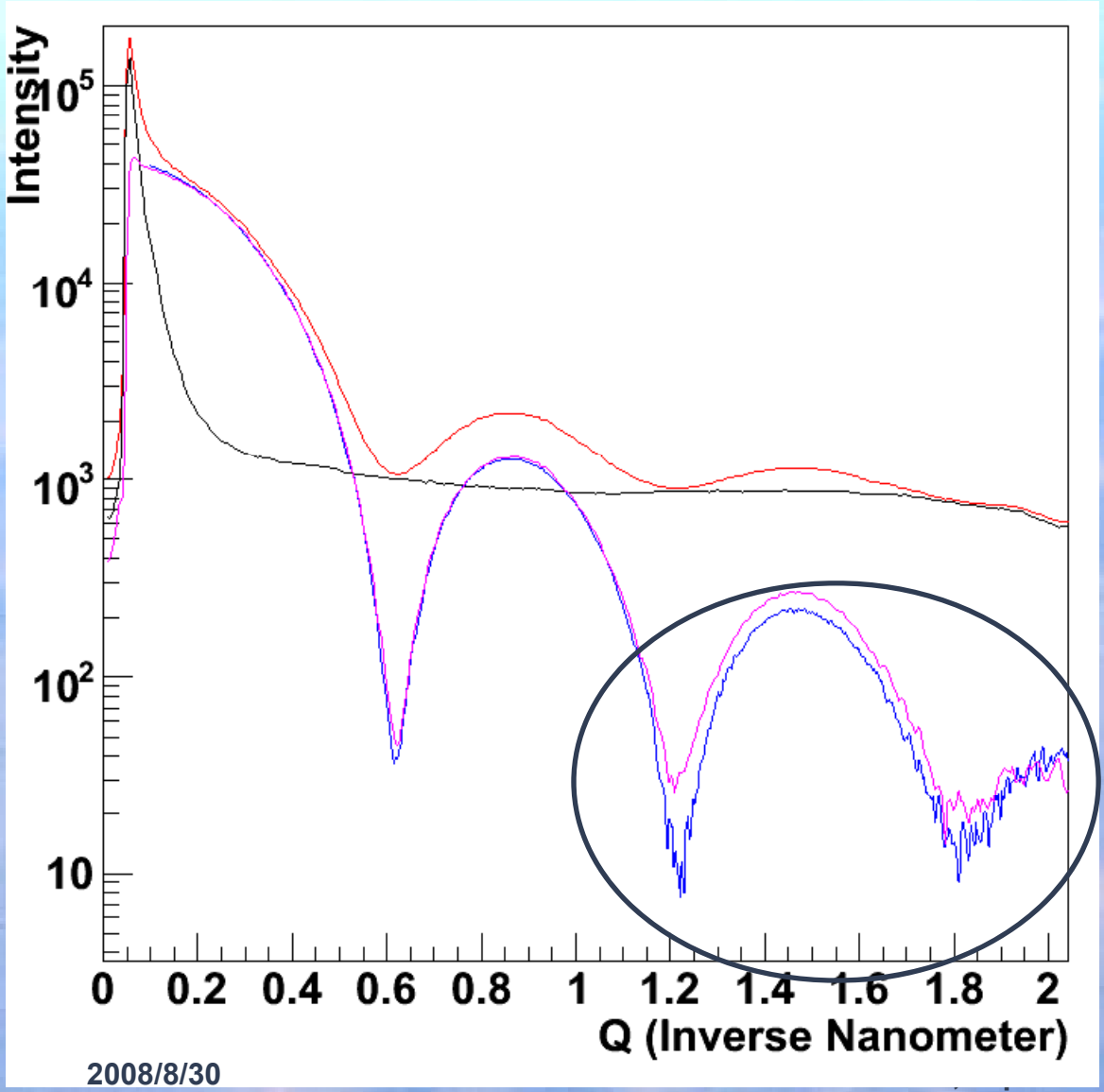
Polystyrene latex
0.04 weight %
solid spheres of 110-nm diameter
1.5 Å
exposure time : 154 sec

Incident photon flux
1.5×10^{11} photons / s

dynamic range
Six orders of magnitude
CCD: 10^4
Imaging Plate: $10^{5\text{-}6}$

Close to edge of the detector
Low detection efficiency

Solution Scattering from Apo-Ferritin



Apo-Ferritin
1.5 Å
exposure time : 436 sec

Solution (μ -PIC)
Water (μ -PIC)
Solution – water (μ-PIC)
R-AXIS (IP)

Incident photon flux
1.5×10^{11} photons / s

✓Deviation from IP was seen in high-q region.
✓Signal to noise ratio strongly effects on low-counting rates region.
✓Further studies are necessary

Detector characteristics

	current	goal
pitch	400 μm	200 μm
Number of electrodes	256 × 256	1500 × 1500
Active area	100 × 100 mm^2	300 × 300 mm^2
Gas gain	$5 \times 10^3 - 10^4$	$> 10^4$
Dynamic Range	$> 10^6$	10^7
Intensity Range(Global)	< 5MHz	10MHz
Efficiency uniformity	～several %	< 1%
distortion	No	No

Achieved by Gamma-ray camera based on a μ-PIC

Summary

- good linear correlation > 10^5 (20 cps – 5 Mcps)
- Position resolution of 120 um
- CRP method :

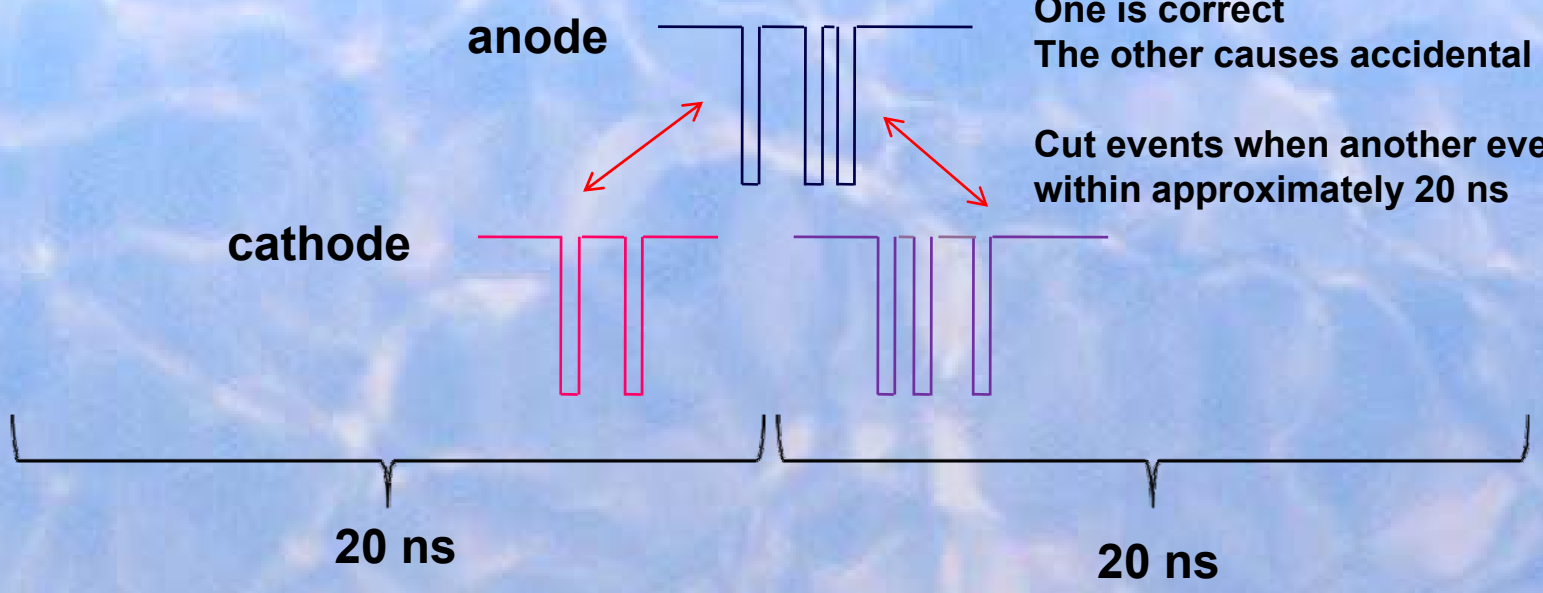
  Rint (internal agreement factor)3.7%
- Time resolved measurements
- Image without distortion
- Dynamic Range of > 10^6

Photon Counting Detector μ-PIC

A photon anode : a few signals for ~100 ns
cathode : a few signals for ~100 ns

coincidence

To avoid accidental coincidence



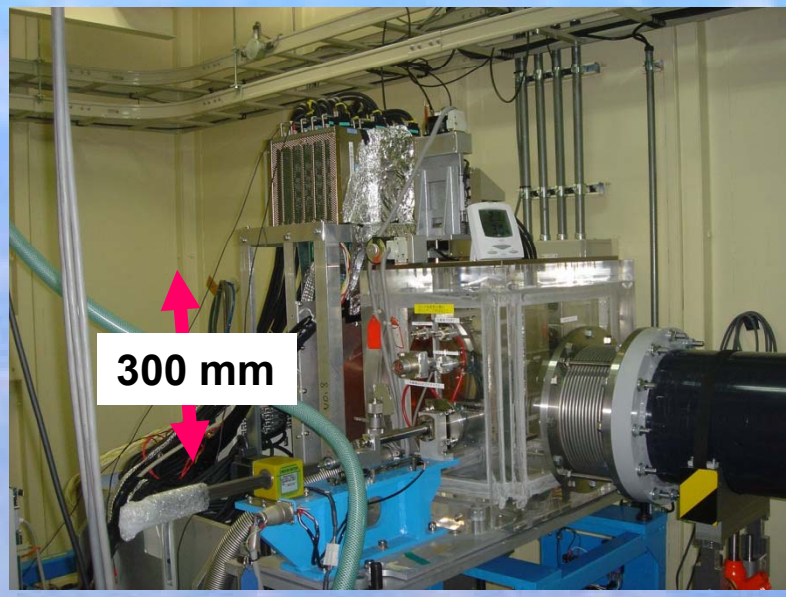
Choose between two possibilities:
One is correct
The other causes accidental coincidence

Cut events when another event comes within approximately 20 ns

Further improvements are necessary

Further Work

<schedule>
Small-angle neutron scattering at JRR-3, Japan in September
Solution scattering experiments
at Spring-8, Japan in October



Increase
detection efficiency
dyamic range
Confirm consistency under
high and low-count rate environments
Large μ-PIC with an active area
of 300 × 300 mm^2 in development