# Speckle Spectroscopy - X-ray Photon Correlation Spectroscopy

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**Grenoble, September 2, 2005**

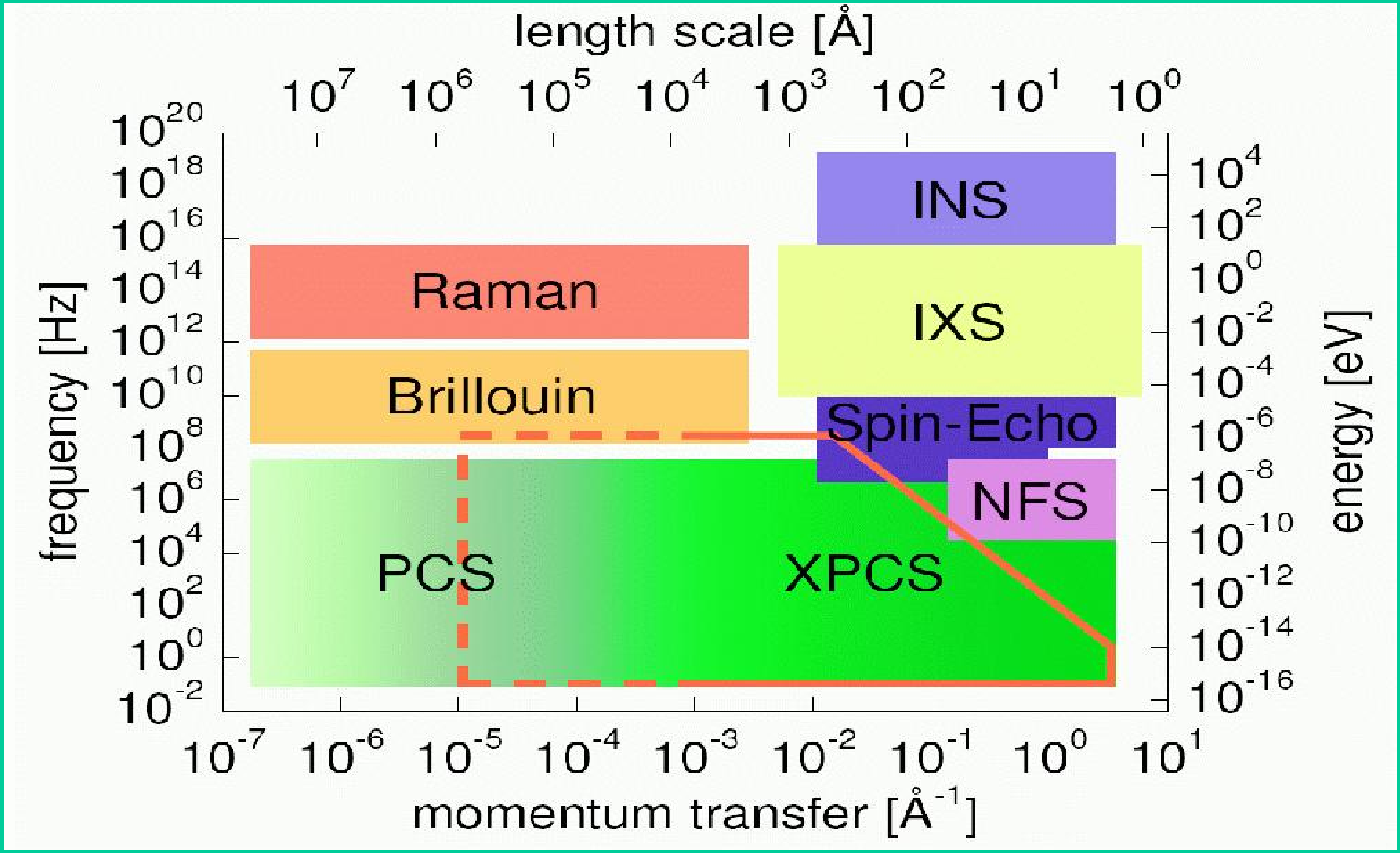
# Layout

- Introduction to coherent scattering
- Dynamics in different time scales
- Coherent Imaging
- Detectors - state of the art and future concepts
- Summary

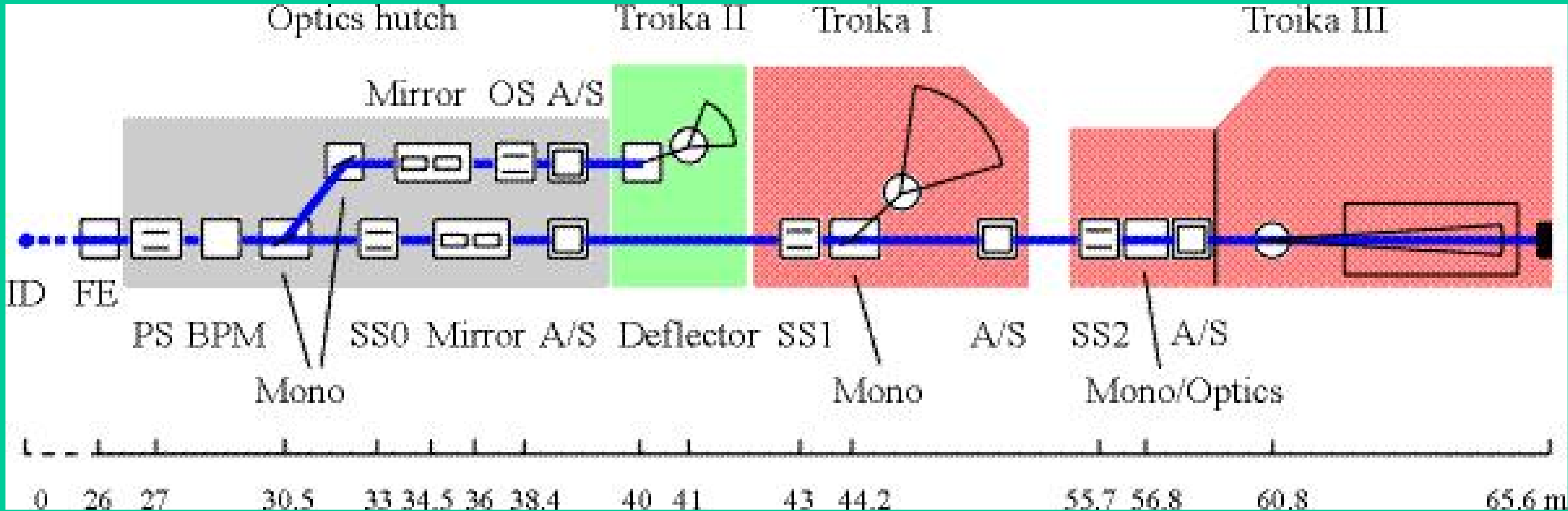
# XPCS - Overview

- XPCS is a scattering technique probing dynamics on nanoscaled systems
- XPCS uses coherent synchrotron radiation
- High degree of coherence only in a small beam cross section defined by an aperture ~ 10 μm
- It involves small angle X-ray scattering (SAXS)
- 2D detectors with high spatial resolution ~ 20...200 μm
- Scanning 0D detectors; Q-resolution by slit/pinhole

# XPCS - Overview



# ESRF ID10 - Overview




**Coherence lengths:**

$\xi_{trans} = \lambda/2 * R/s \sim 10...100\ \mu m$

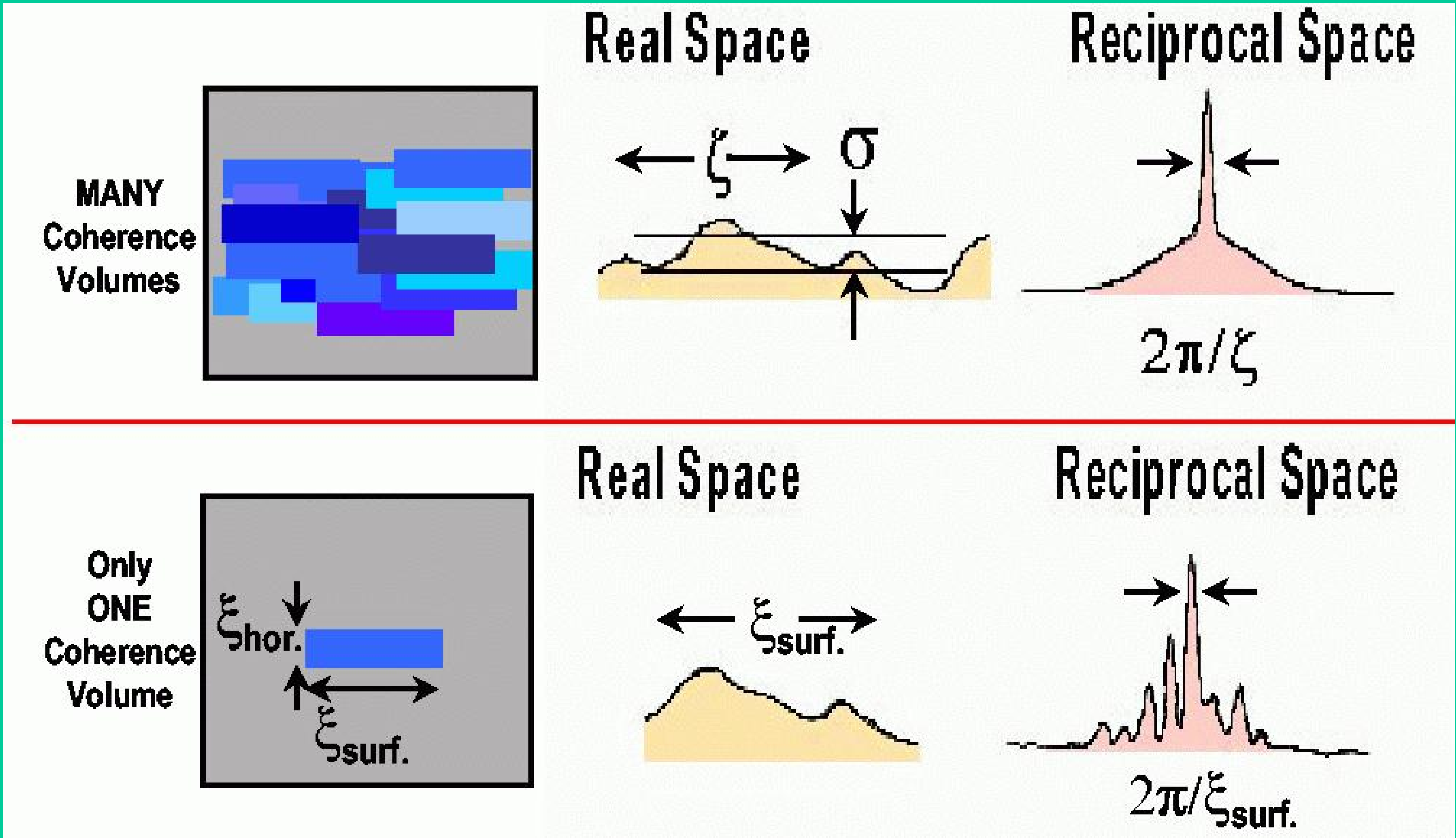
$\xi_{long} = \lambda*(\lambda /\Delta\lambda) \sim 10nm...10\ \mu m$

**Coherent flux:**

$F_c = (\lambda/2)^2 * B$

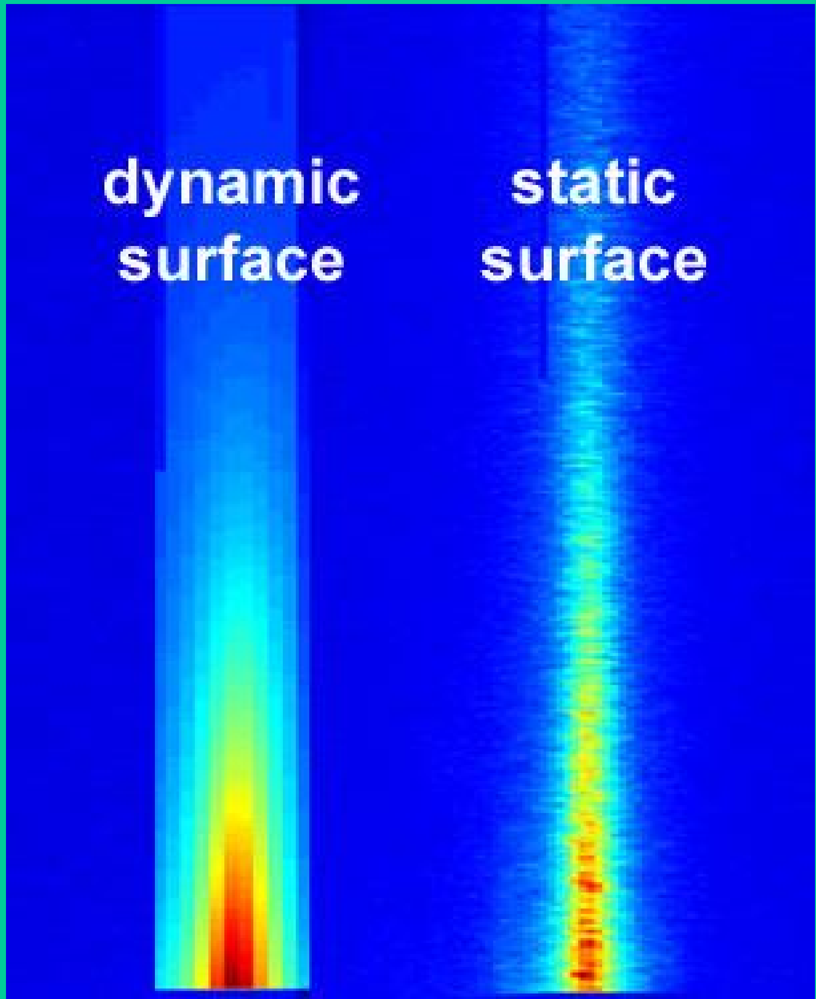
ID10: ~$10^{10}$... $10^{12}$ Hz

# Coherent Scattering




From Metin Tolan

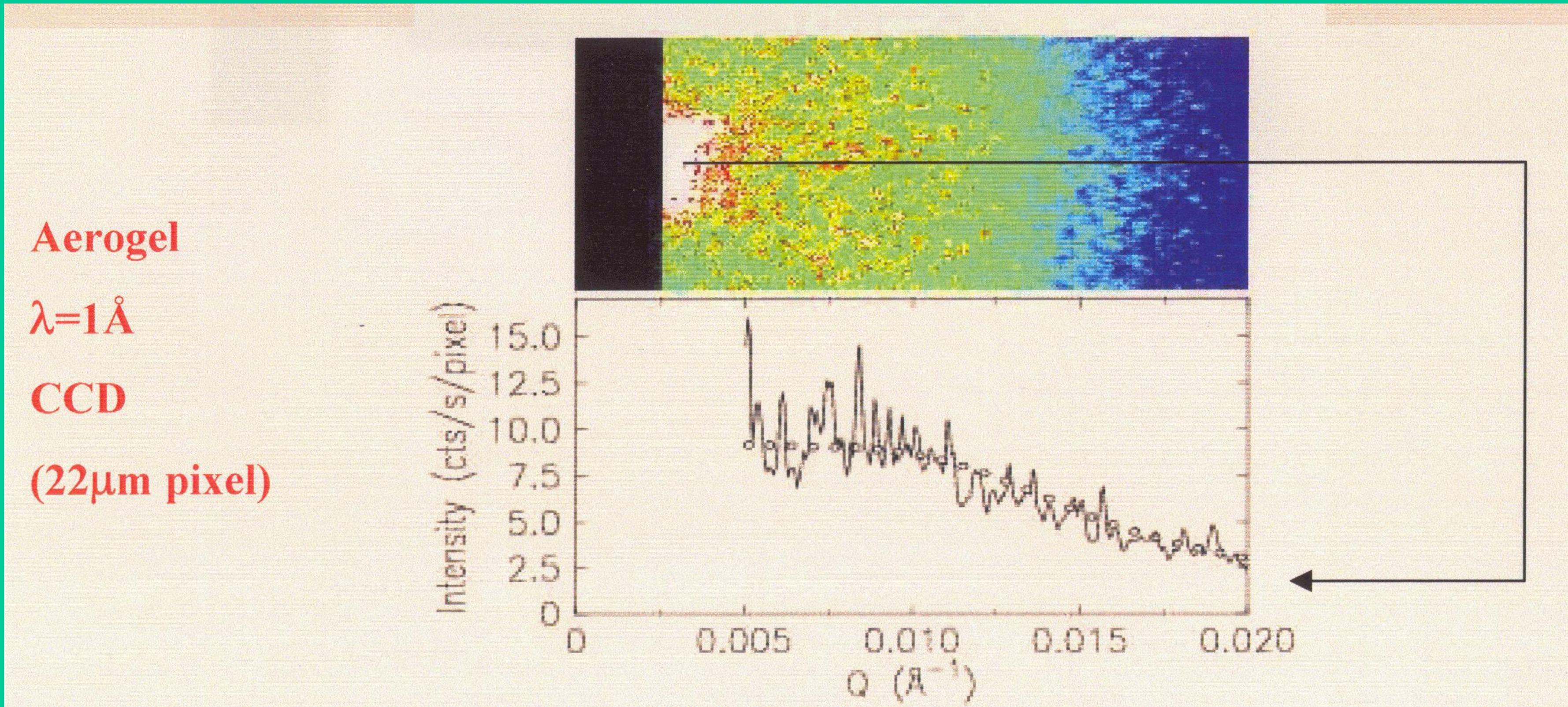
# Coherent Scattering




Polymer melt close to the glass transition

Speckle!

# Coherent Scattering

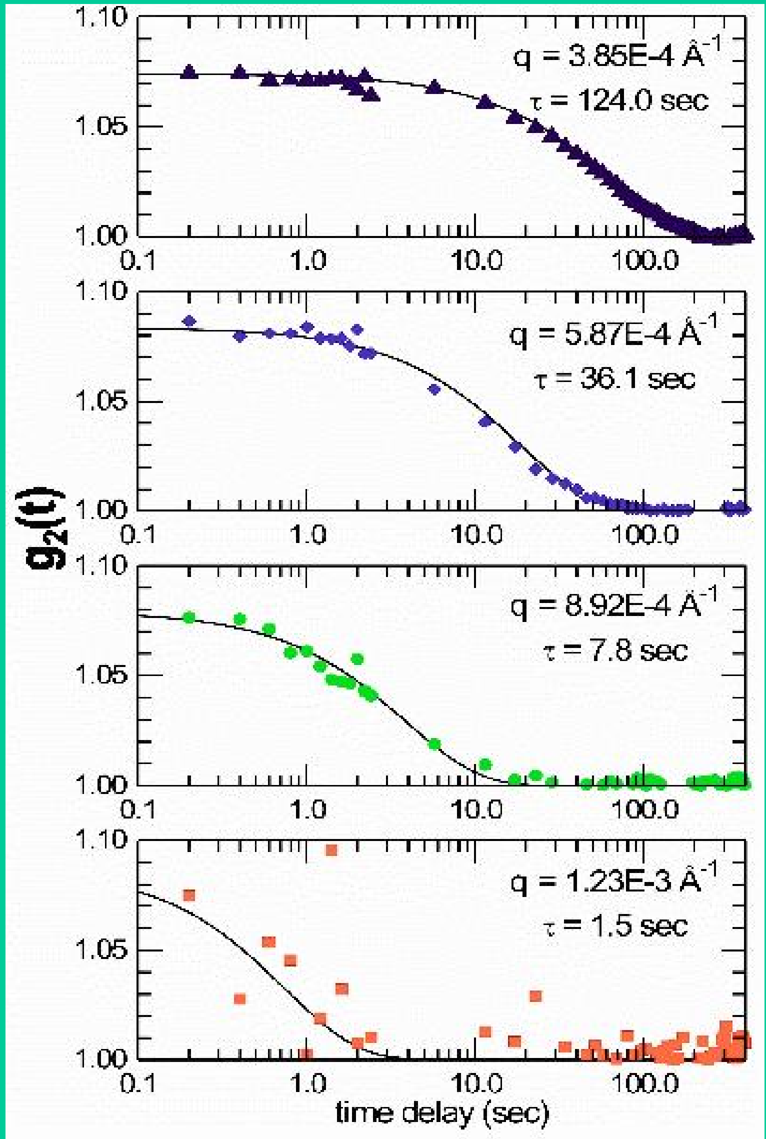



Aerogel sample

full line: coherent illumination
circles: incoherent illumination

D.Abernathy et al. JSR 5(1998), 37

# XPCS - slow dynamics




Polystyrene film on Si measured in reflection geometry

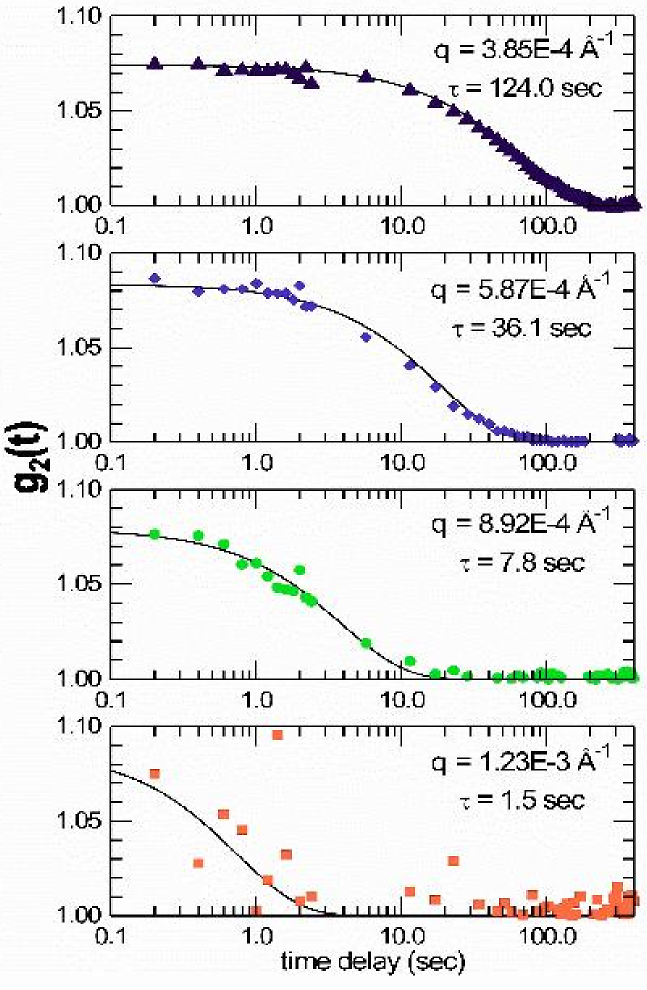
Autocorrelation function:

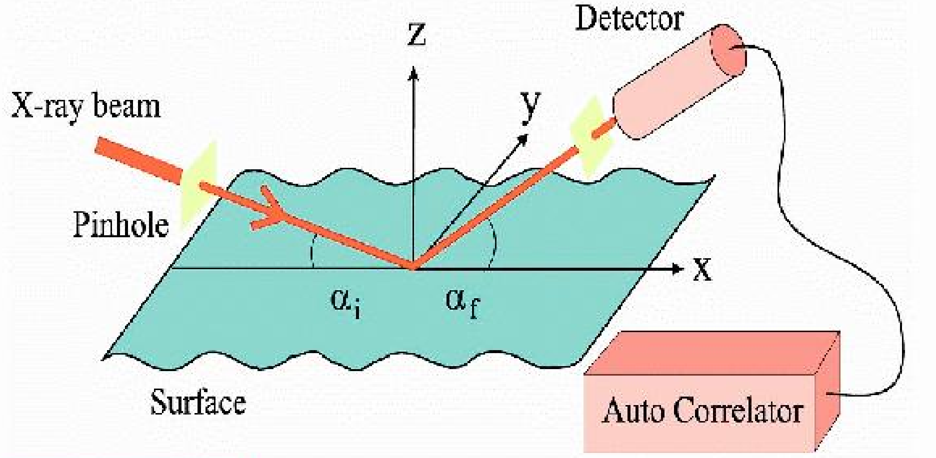
$$g_2(t) = \langle I(q,t')I(q,t'+t)\rangle \, / \, \langle I(q,t')\rangle^2$$

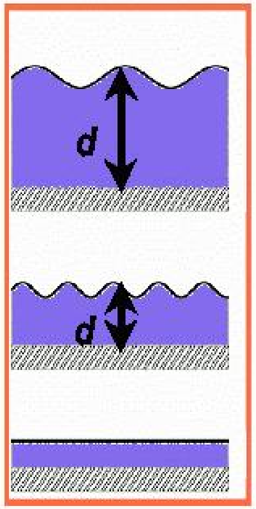
yields relaxation time

Kim et al. PRL 90(2003),068302

# XPCS - slow dynamics




**APS 8-ID:**

**Dynamics of thin film polymer *surfaces***

Hyunjung Kim, A. Rühm, L.B. Lurio, J.K. Basu, J. Lal, D. Lumma, S.G.J. Mochrie, S.K. Sinha; *Phys. Rev. Lett.* 90, 068302 (2003).

From Metin Tolan

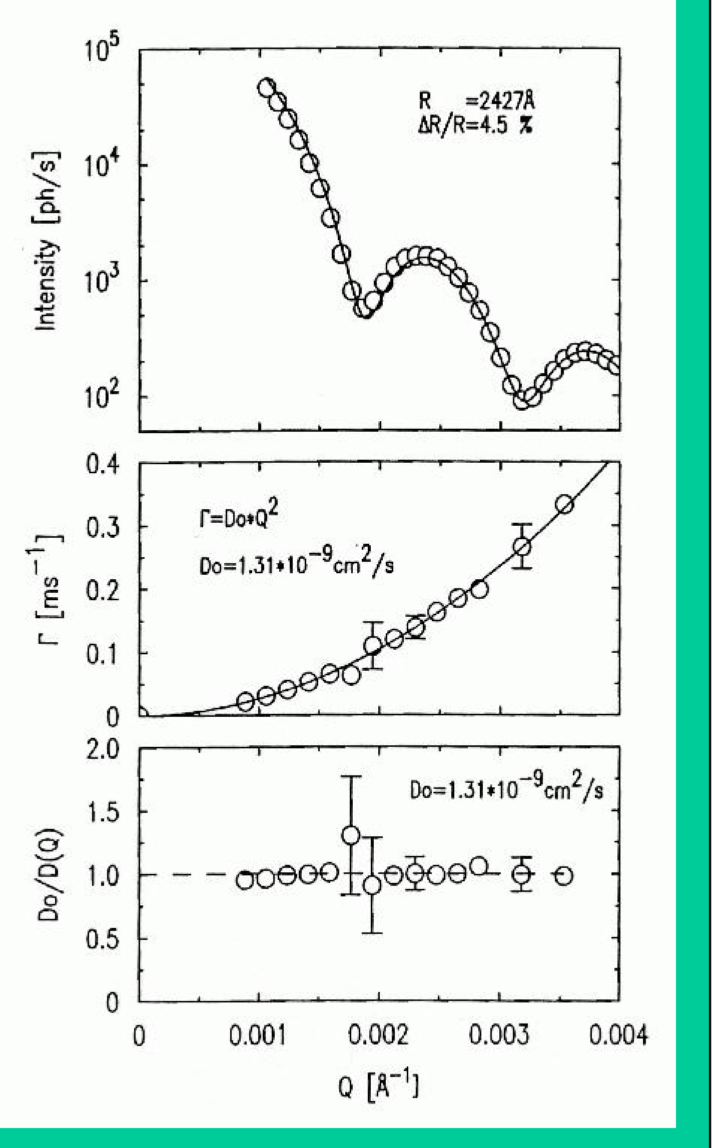
# XPCS - slow dynamics

SAXS pattern of 500nm colloidal silica in water/glycerol mixture

Relaxation rate determined from intensity correlation functions at various Q

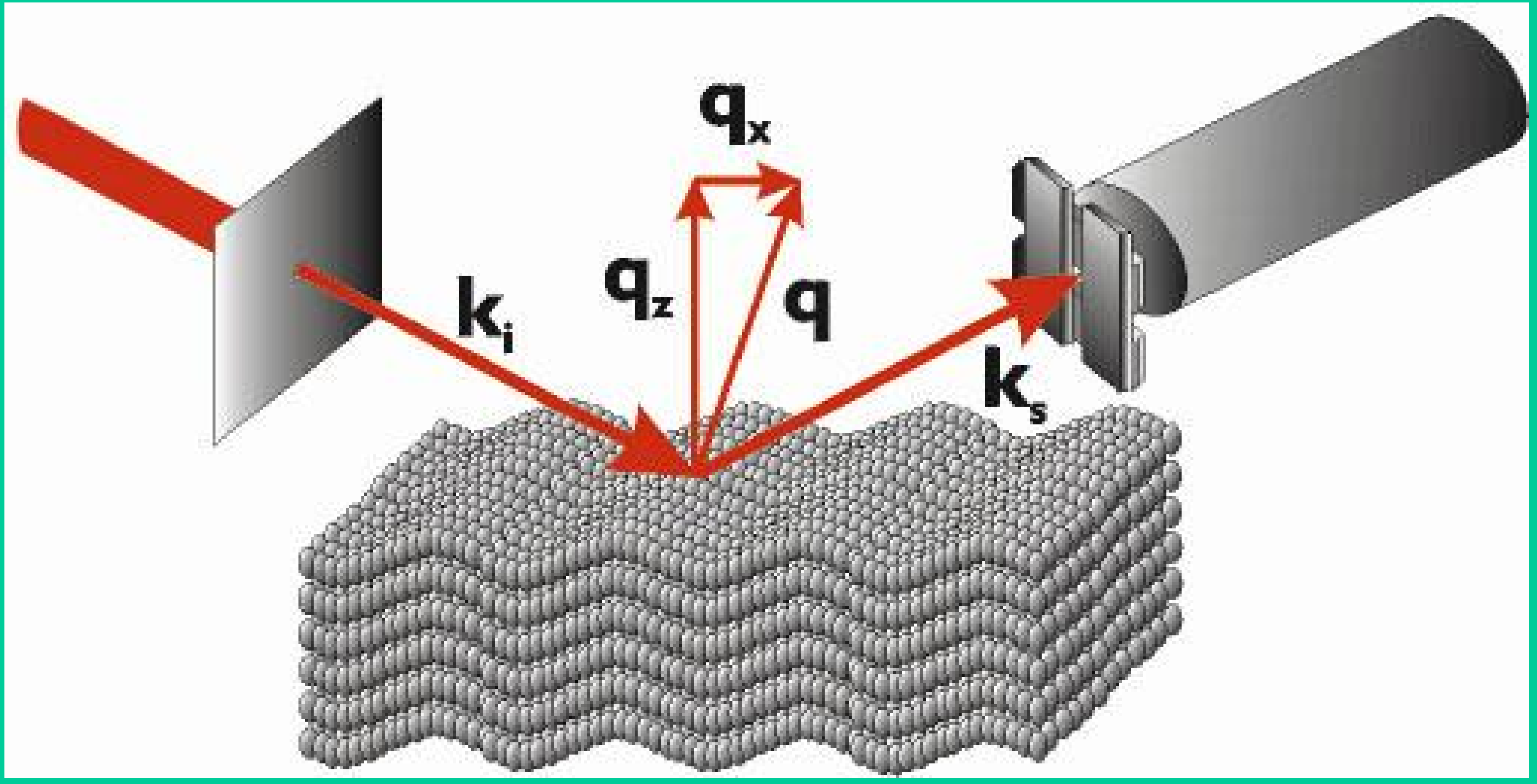
$D_0/D(Q)$ is Q independent
no interaction of particles

G.Grübel et al. (1999) in *Slow Dynamics in Complex Systems*



# XPCS - slow dynamics

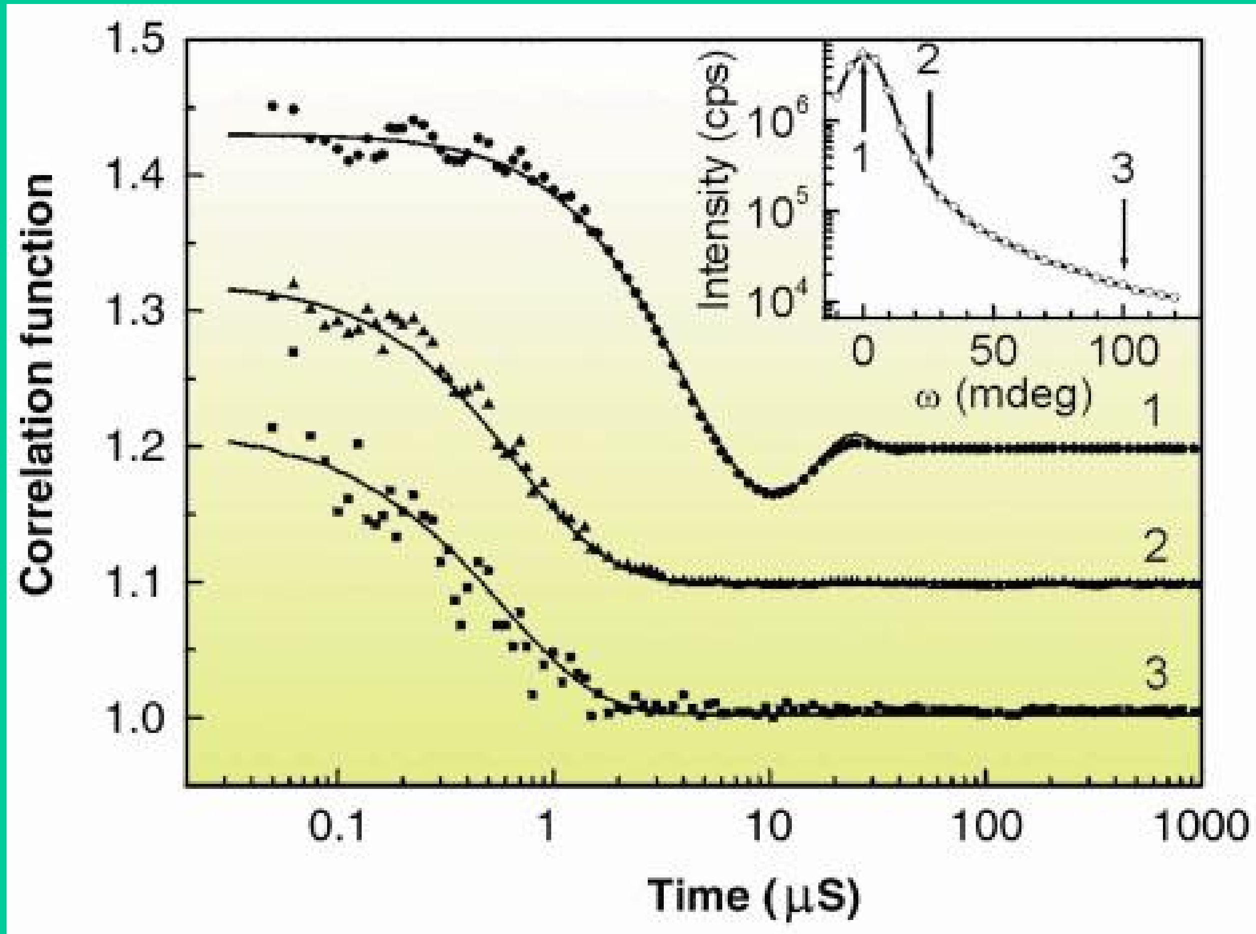
Membranes in Motion




A.Fera et al. , ESRF Highlights 2001

# XPCS - slow dynamics

## Membranes in Motion




A.Fera et al. , ESRF Highlights 2001

# Summary on XPCS

Time correlation functions are determined

either

by measuring continuously frames with 2D detector and software processing: characteristic times > 1s

or

by point detectors (APDs) and hardware autocorrelators till μs

# Coherent Imaging and Reconstruction

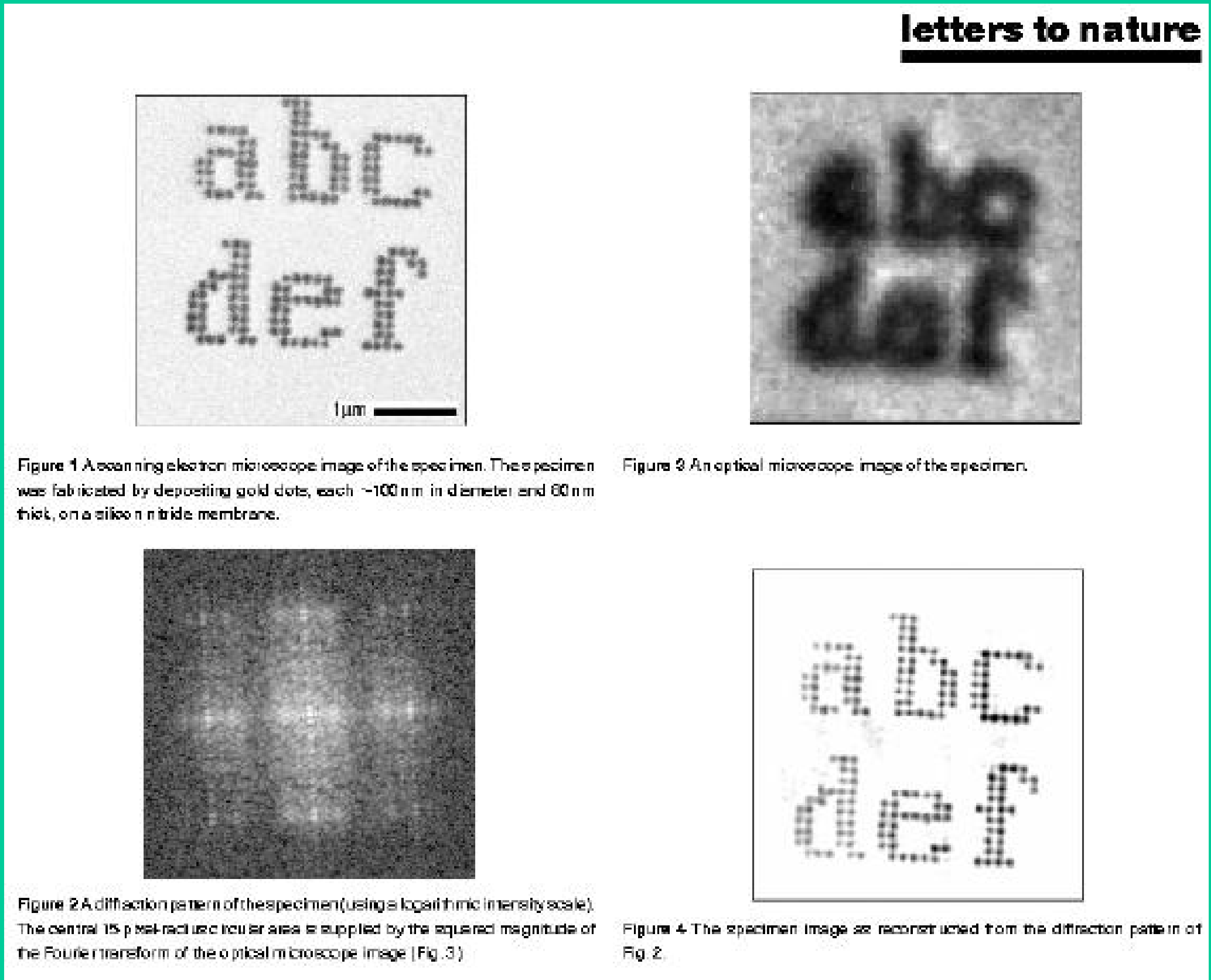

letters to nature



Figure 1 A scanning electron microscope image of the specimen. The specimen was fabricated by depositing gold dots, each ~100 nm in diameter and 80 nm thick, on a silicon nitride membrane.

Figure 3 An optical microscope image of the specimen.

Figure 2 A diffraction pattern of the specimen (using a logarithmic intensity scale). The central 15 pixel-radius circular area is supplied by the squared magnitude of the Fourier transform of the optical microscope image (Fig. 3).

Figure 4 The specimen image as reconstructed from the diffraction pattern of Fig. 2.

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

# Coherent Imaging and Reconstruction

Oversampling technique

Spatial frequency on the detector has to be at least twice as large as the smallest scattering feature

Reconstruction is performed in an iterative way starting from random phases

Uses a „support“, which should be defined by the sample size

# Coherent Imaging and Reconstruction

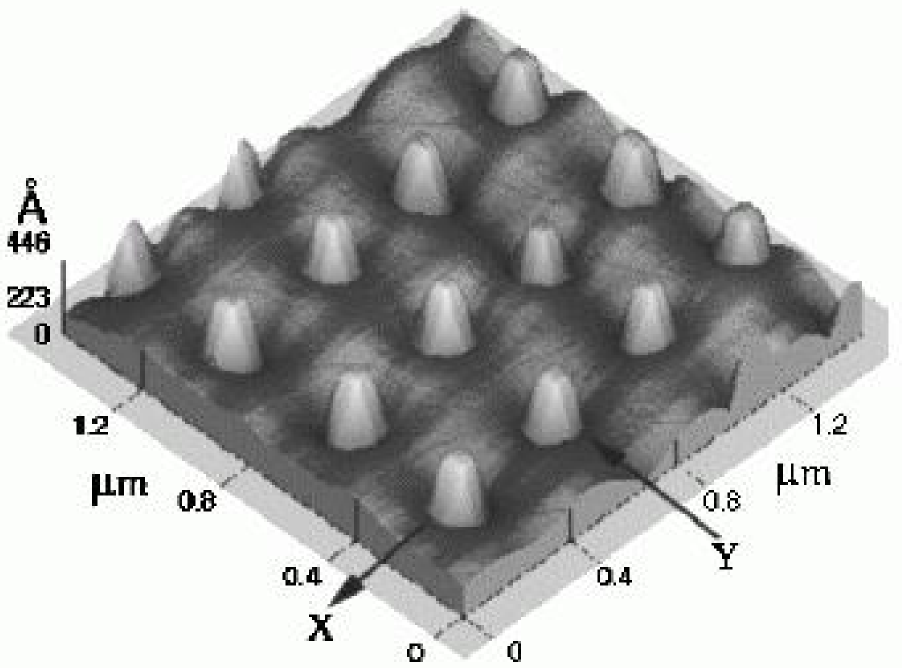
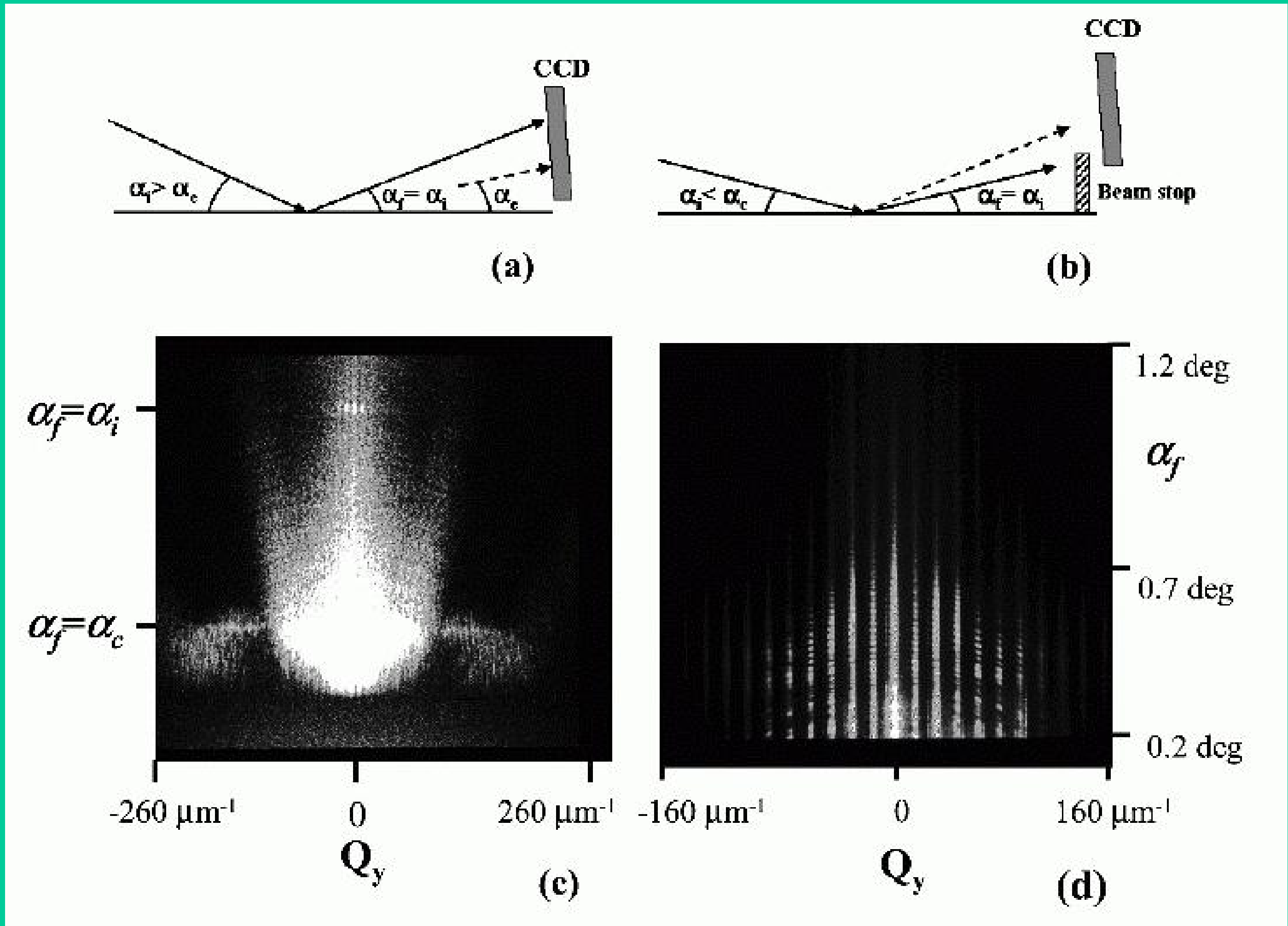



FIG. 1. AFM micrographs of 2D ordered Ge islands.

Ge Quantum Dots on Si

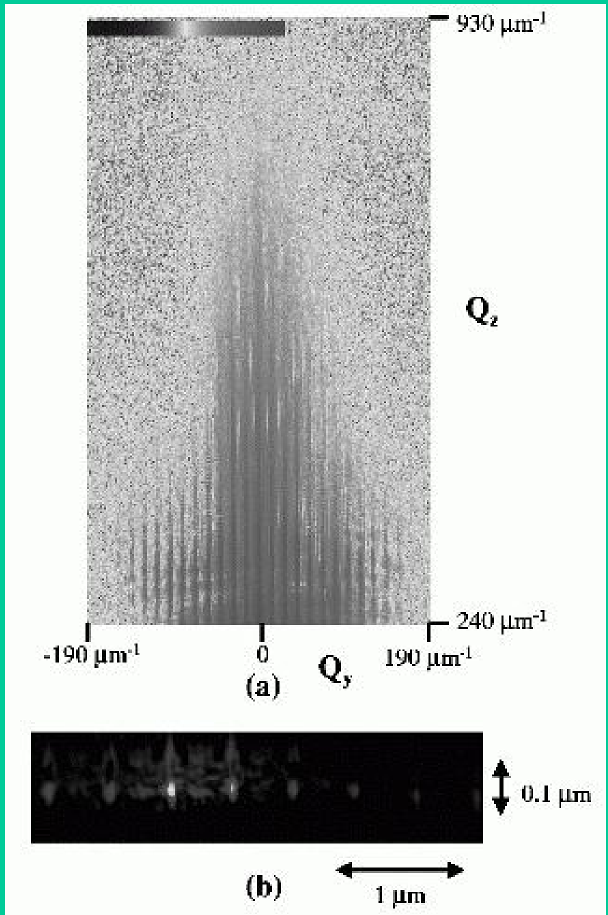
I. Vartanyants et al. PRB 71(2005), 245302

# Coherent Imaging and Reconstruction

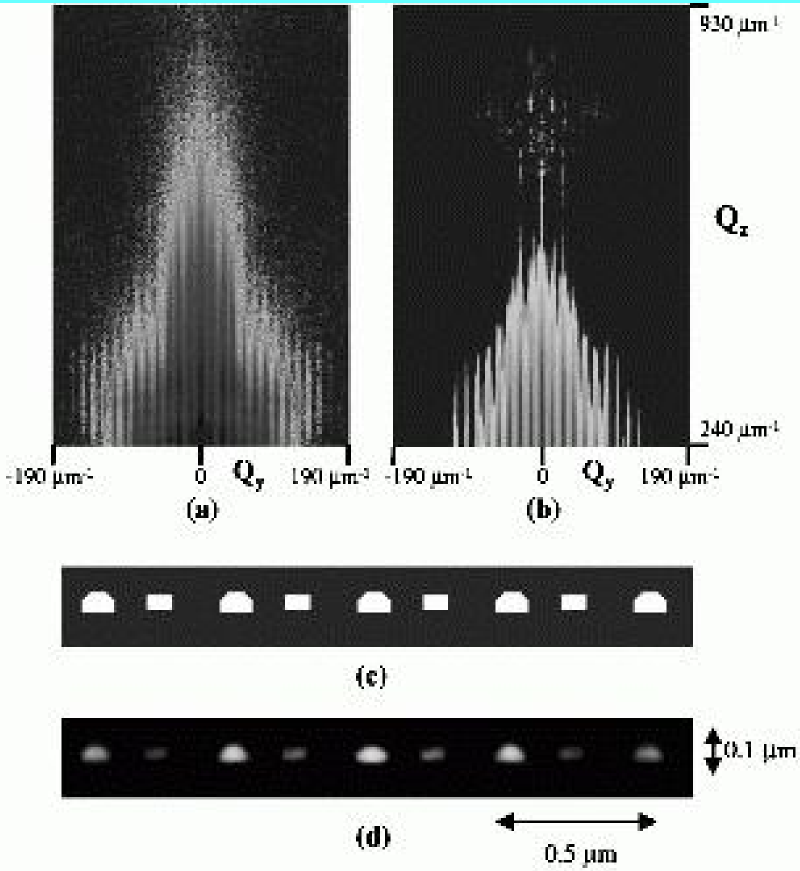



I. Vartanyants et al. PRB 71(2005), 245302

# Coherent Imaging and Reconstruction




exp. data reconstructed data




Top: reconstructed SAXS pattern
bottom: support + reconstruction

c: refined support
d: reconstruction

# Coherent Imaging and Reconstruction

Magnetic lensless imaging on Co/Pt-ML (Co L-edge) at Bessy using circularly polarized synchrotron radiation

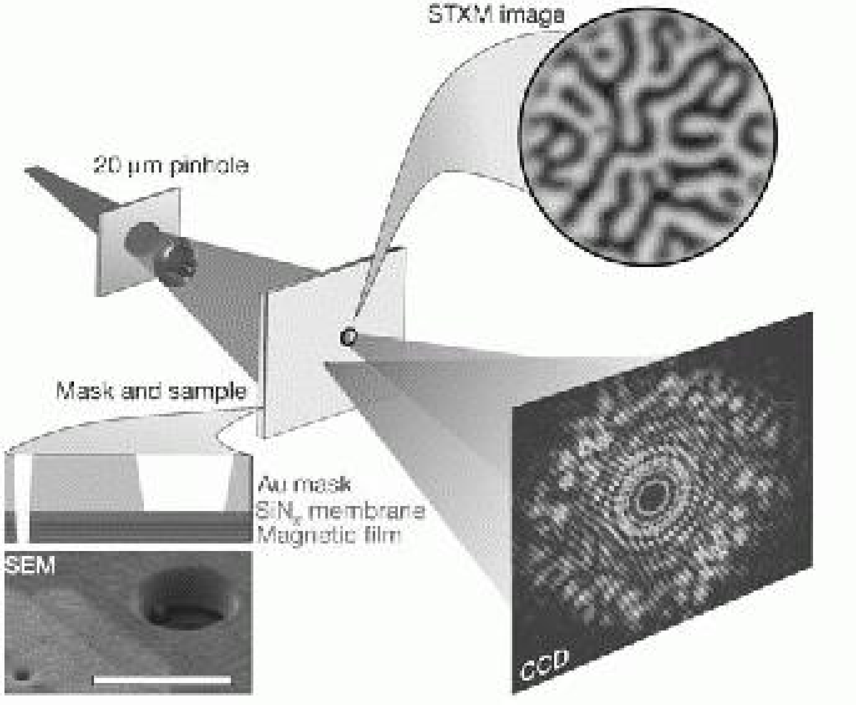
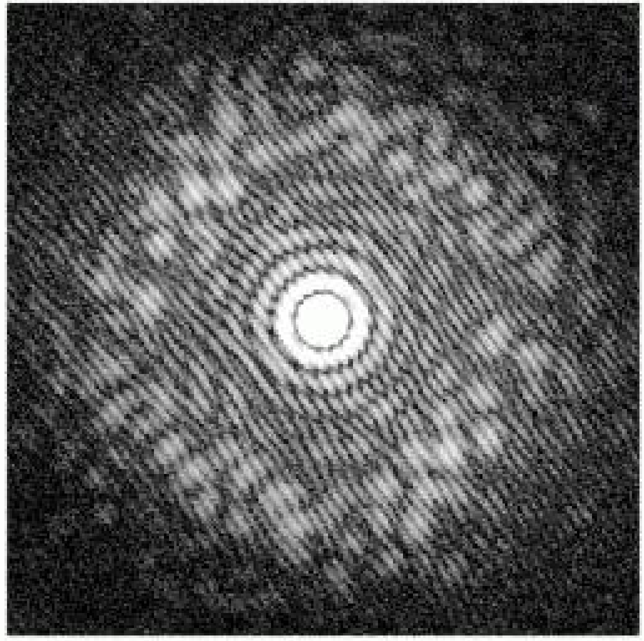
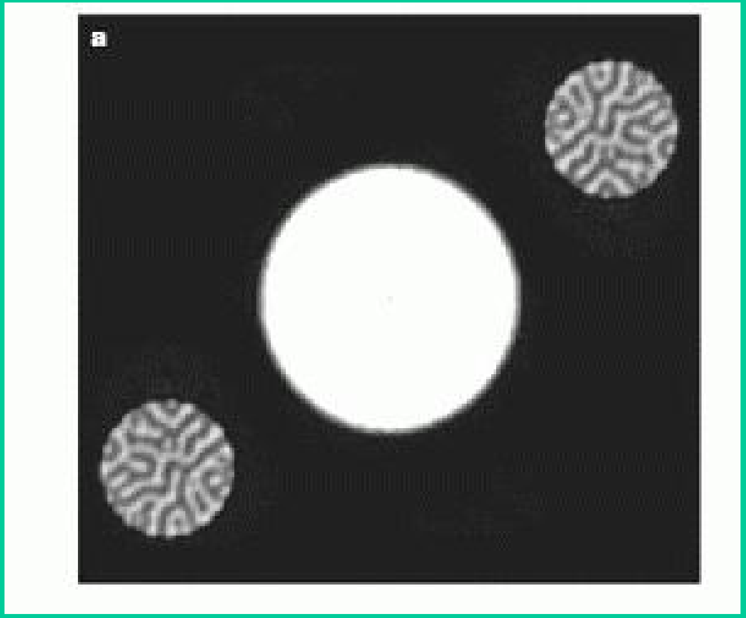



**Figure 1** Scheme of the experimental set-up. Monochromatized and circular polarized

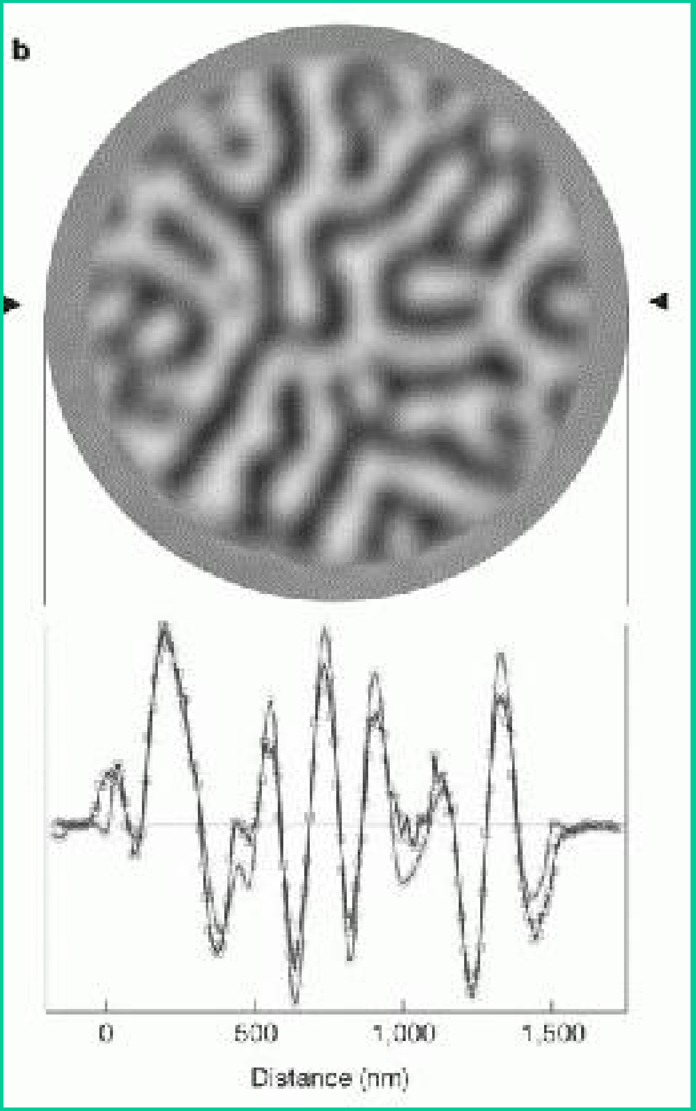


S. Eisebitt et al., Nature 432(2004), 885

# Coherent Imaging and Reconstruction




FT of the hologram




Reconstruction and comparison with STXM

# Summary on Imaging

Imaging always uses 2D detectors

Requirements on resolution:

small pixel size necessary to „see“ the whole sample

large number of pixels (large total size) to resolve small structures at high Q

Reconstruction by Oversampling (iterative technique)

Direct reconstruction after holographic recording;

lensless imaging of magnetic nanostructures

# Avalanche Photo Diodes

## Commercial Suppliers for APDs and Developers

APDs are manufactured by

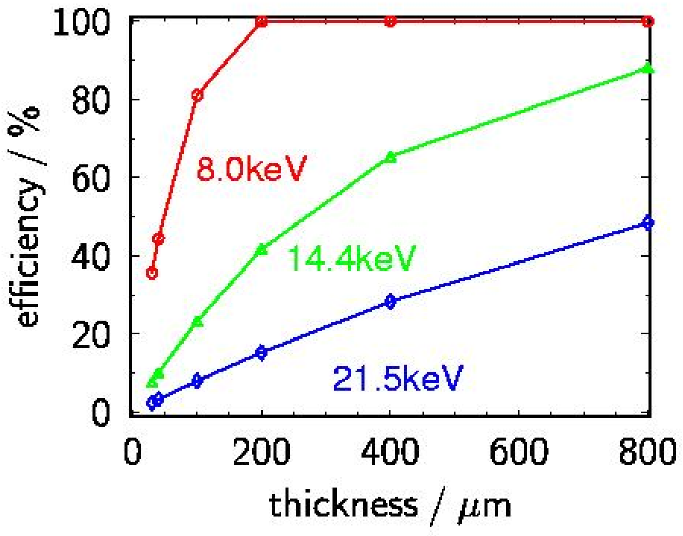
- Advanced Photonix (USA)
- Hamamatsu (Japan)
- Perkin Elmer Optoelectronics – former EG & G (Canada)
- Radiation Monitoring Devices – RMD (USA)

APDs were introduced in SR research by

- S. Kishimoto – Inst. of Materials Structure Science, Tsukuba
- A. Baron – SSRL, ESRF, Spring-8

# Avalanche Photo Diodes

## Ways to Increase Efficiency of APD Detectors



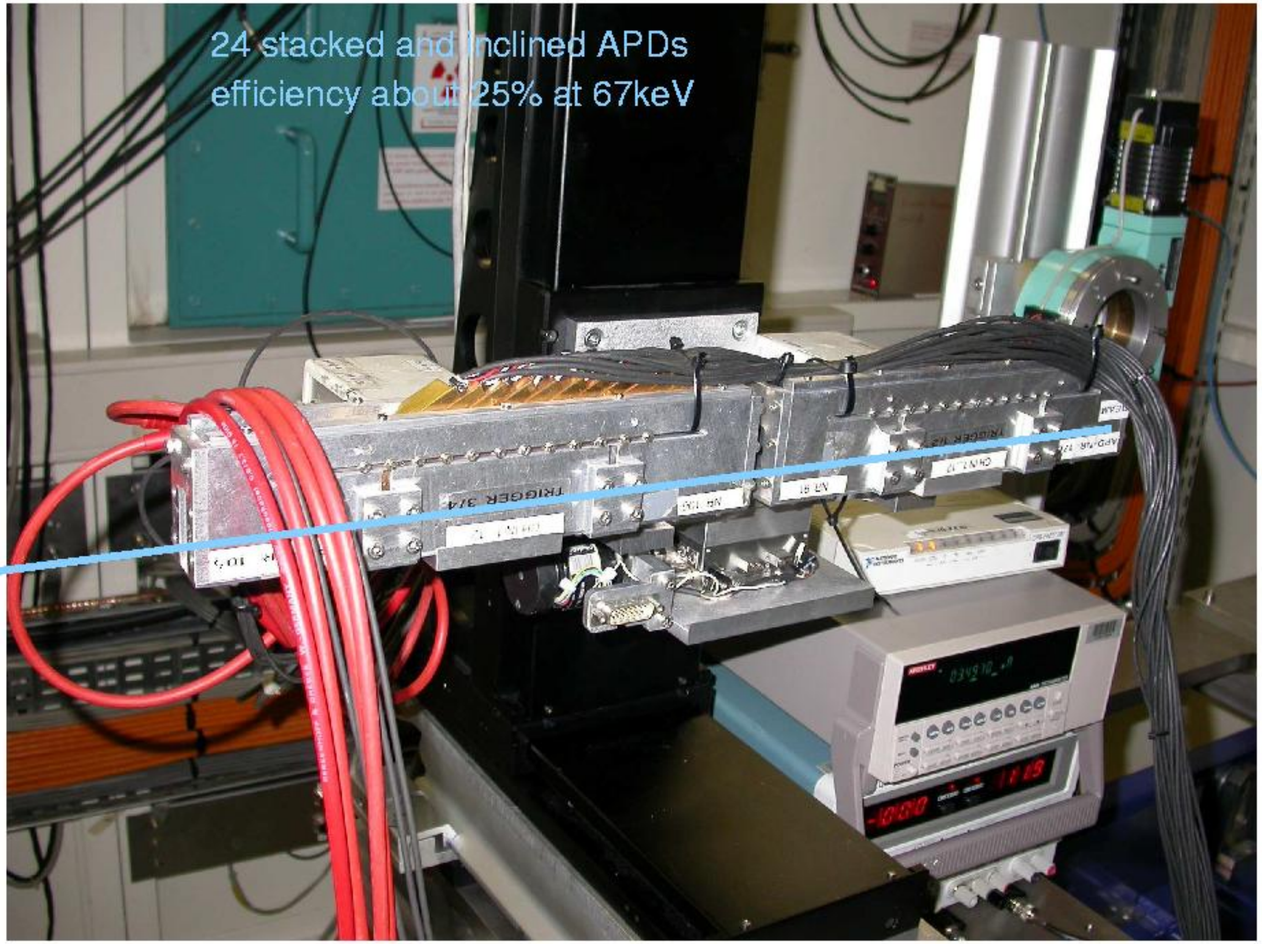

- 30 $\mu$m : Hamamatsu APD
- 40 $\mu$m : Advanced Photonix APD
- 100 $\mu$m : Perkin Elmer APD
- 200 $\mu$m : Perkin Elmer APD (inclined)
- 400 $\mu$m : 4 Perkin Elmer APDs stacked
- 800 $\mu$m : 4 Perkin Elmer APDs stacked and inclined

The last 4 are standard solutions at ID18 at the ESRF

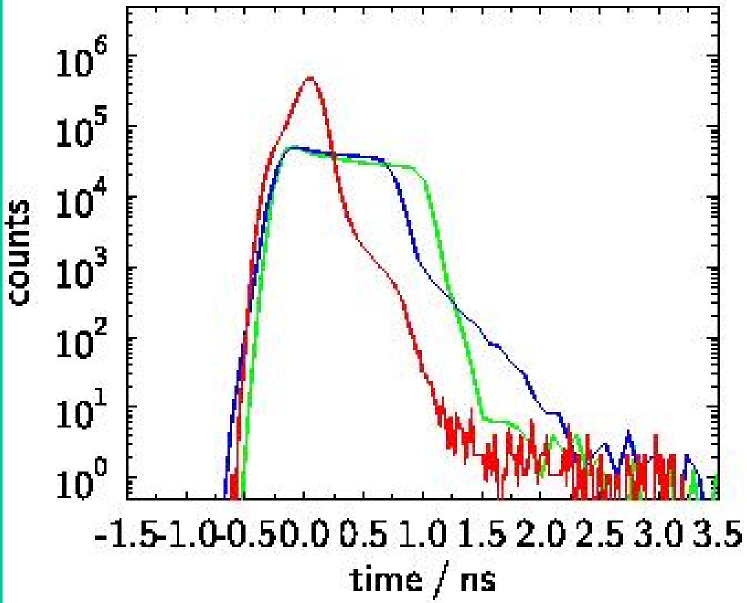
This will be a problem with arrays!

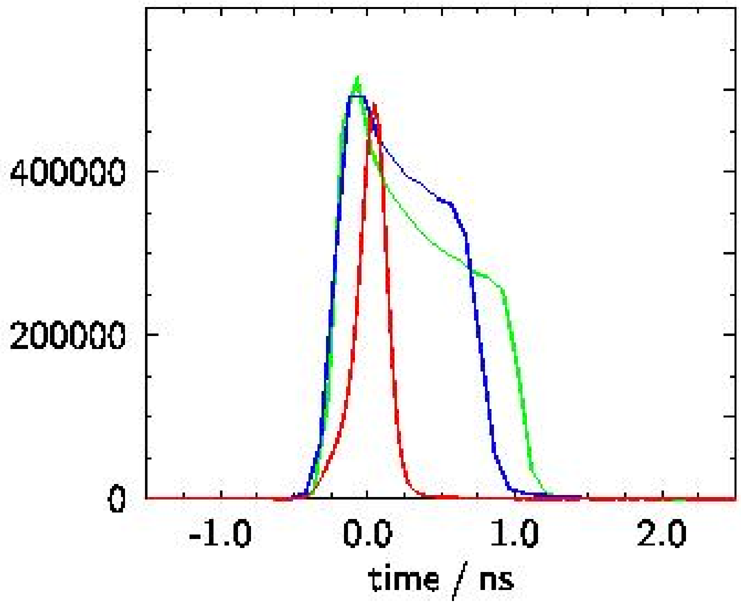
# Avalanche Photo Diodes

## Ways to Increase Efficiency of APD Detectors



# Avalanche Photo Diodes

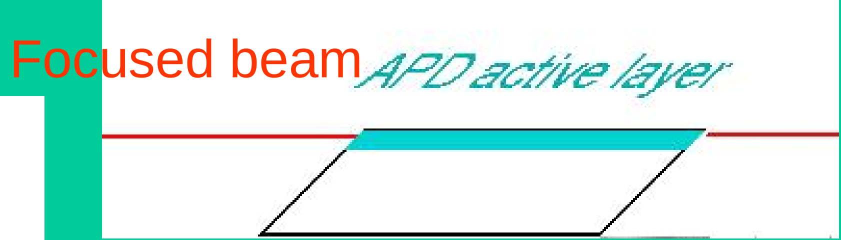



EG & G 5mm

Hamamatsu 3mm

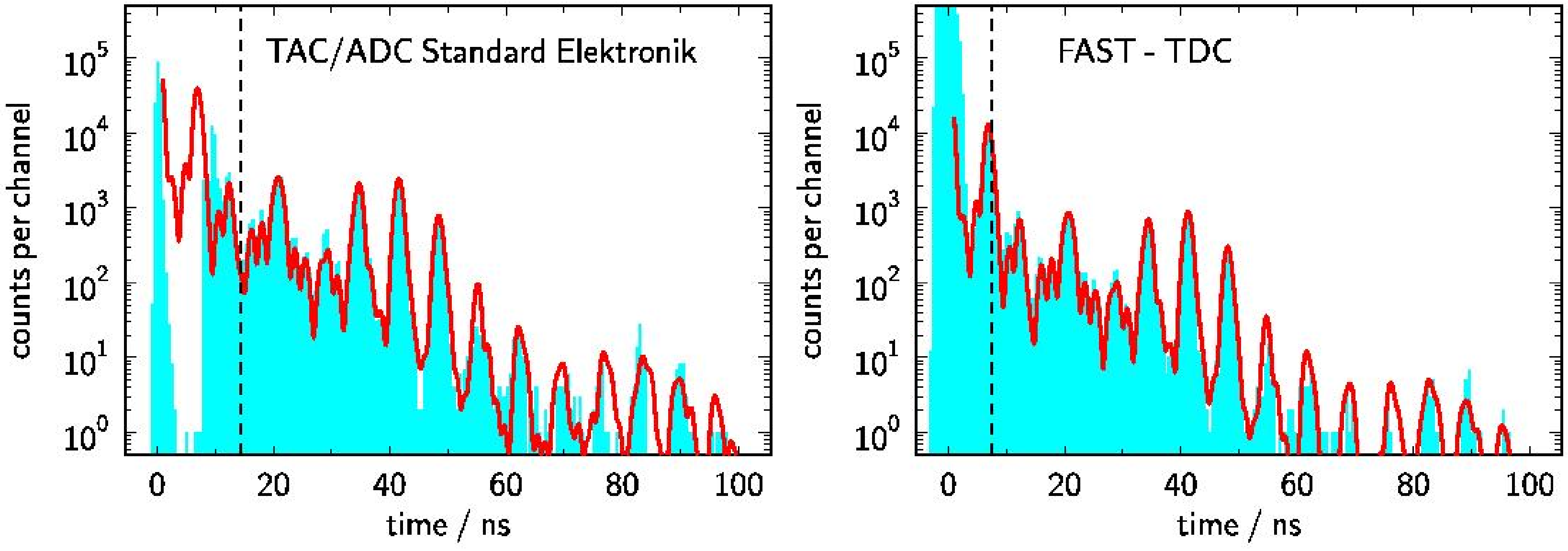
Hamamatsu 3mm perpendicular



# Avalanche Photo Diodes

EuS  T=7K H = 0

Nuclear Resonant Scattering time spectra – Perkin Elmer APD




Vertical dashed lines show begin of fit region

# Summary on point APDs

Manufacterer:

RMD, Advanced Photonix not recommended

Perkin Elmer, Hamamatsu very reliable

Perkin Elmer more efficient

Hamamatsu can be faster, especially in „perpendicular incidence“

# APD array

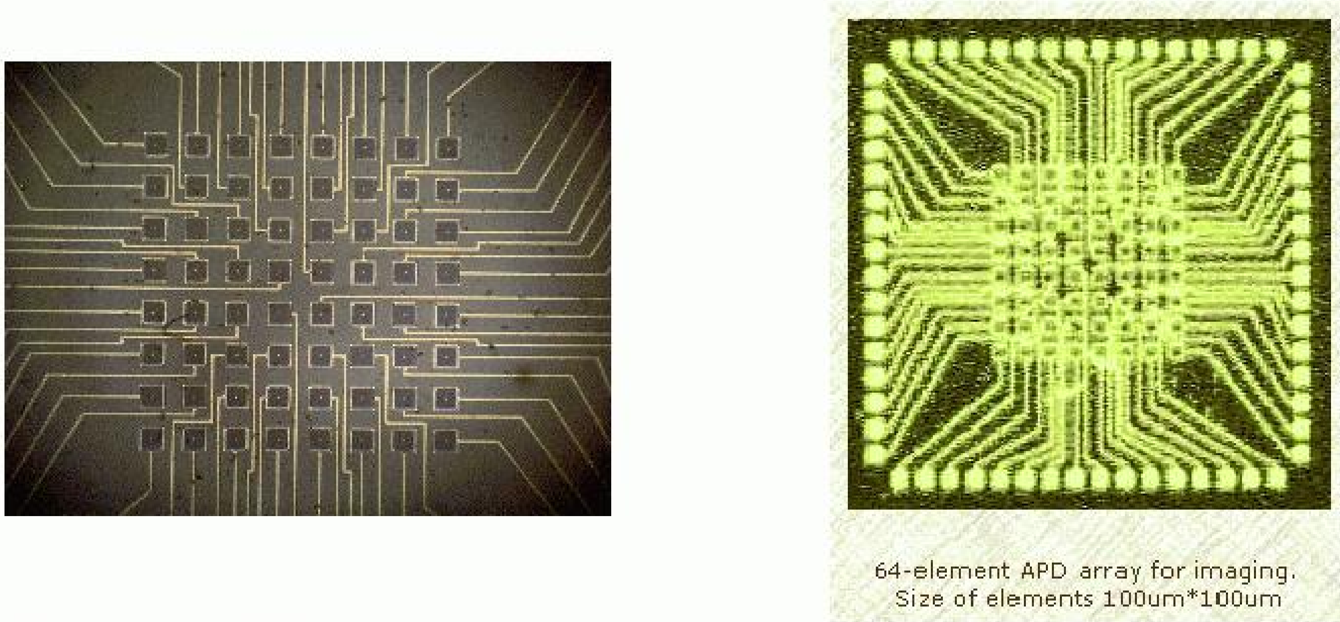



Fig. 1: Array of GMPD's. A microscopic view is shown left and the chip layout with pads right.

left: 8 x 8 array of APDs
right: APD array and chip layout
Disadvantage: only 3 μm thick

# Important points for APD arrays

Electronics „downstream“ has to be developed!

Collaborations: Medipix? PSI? Cornell? MPI Munich? ...

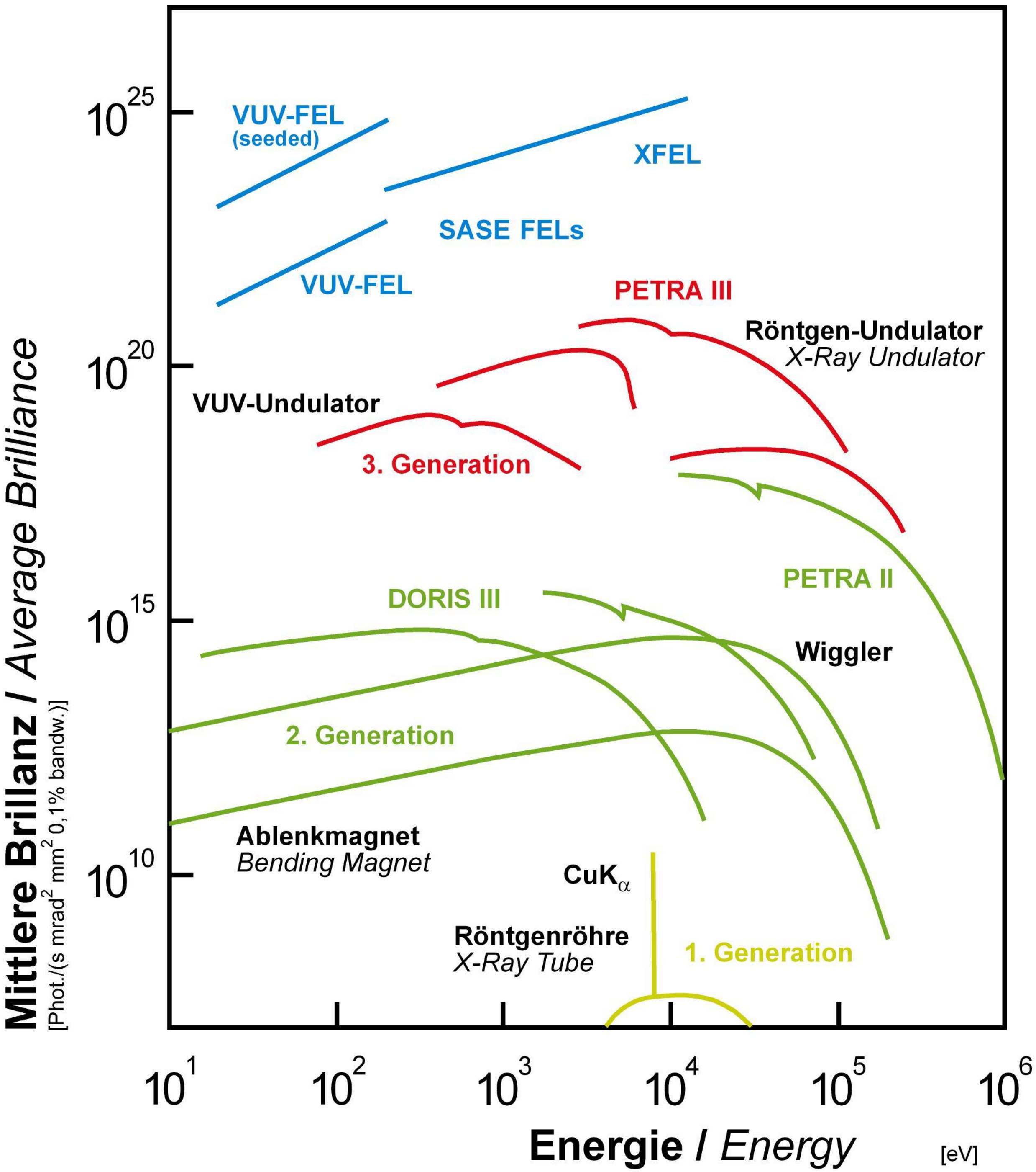
Autocorrelator hardware per pixel??

Fast read-out and software solution?? Much more flexible!

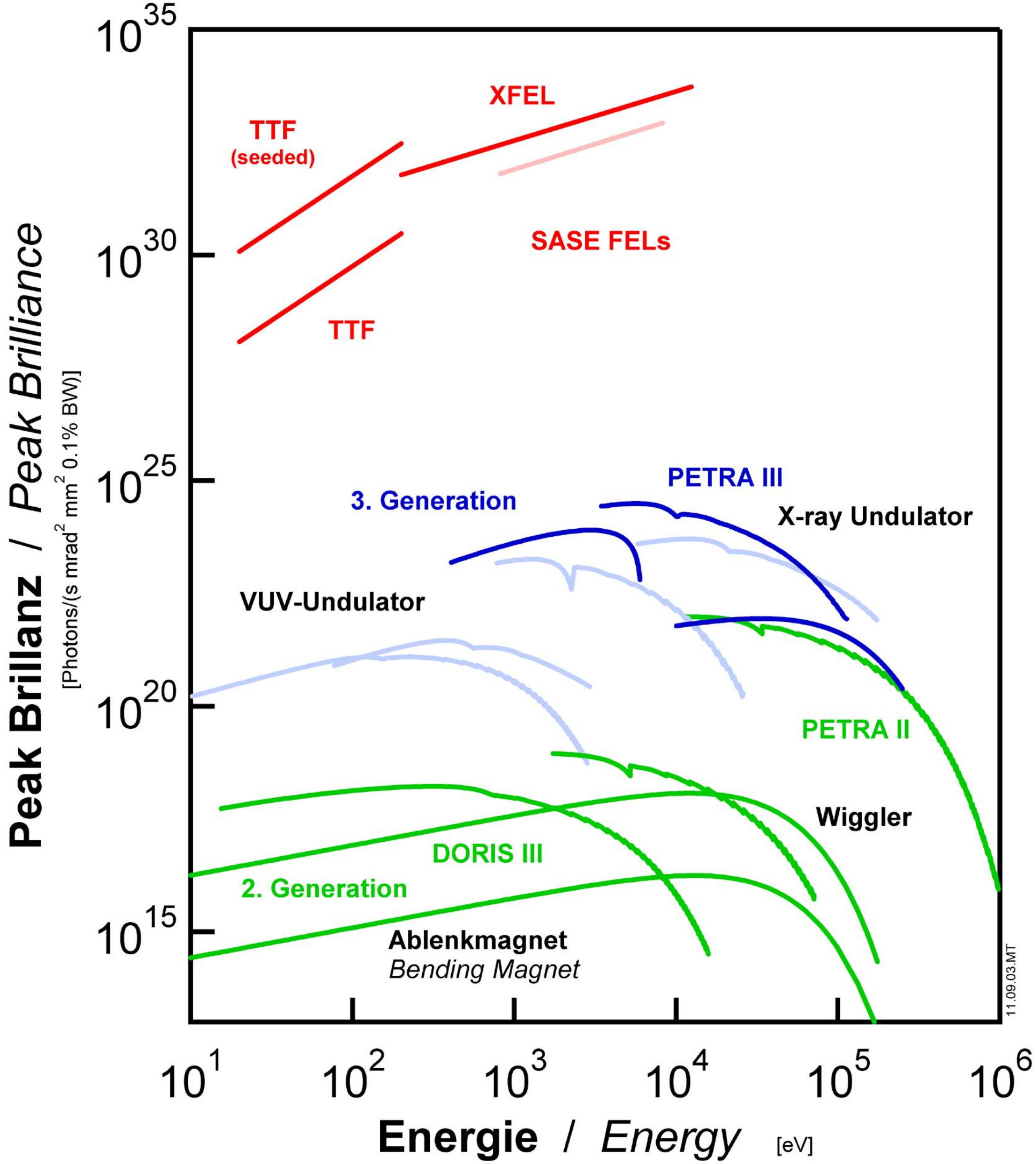
Single photon device or integrating device?

„simple“ register/counter or ADC

Future sources, especially X-FEL, many photons per bunch!



VUV-FEL
(seeded)
XFEL
SASE FELs
VUV-FEL
PETRA III
Röntgen-Undulator
X-Ray Undulator
VUV-Undulator
3. Generation
PETRA II
DORIS III
Wiggler
2. Generation
Ablenkmagnet
Bending Magnet
CuKα
Röntgenröhre
X-Ray Tube
1. Generation
Mittlere Brillanz / Average Brilliance
[Phot./(s mrad² mm² 0,1% bandw.)]
10^25
10^20
10^15
10^10
10^1
10^2
10^3
10^4
10^5
10^6
Energie / Energy
[eV]



Peak Brillanz / Peak Brilliance
[Photons/(s mrad² mm² 0.1% BW)]
10^35
10^30
10^25
10^20
10^15
10^1
10^2
10^3
10^4
10^5
10^6
Energie / Energy [eV]
XFEL
TTF
(seeded)
TTF
SASE FELs
PETRA III
3. Generation
X-ray Undulator
VUV-Undulator
PETRA II
Wiggler
DORIS III
2. Generation
Ablenkmagnet
Bending Magnet
11.09.03.MT

# Conclusions

- XPCS is a well established technique for slow dynamics
- Coherent Imaging and reconstruction (including magnetic imaging) increasing
- Detectors are bottleneck; there is a need for fast 2D-detector systems

# Acknowledgements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- A. Robert, ESRF Grenoble
- M. Tolan, Univ. Dortmund
- A. Baron, Spring-8
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