Argonne NATIONAL LABORATORY

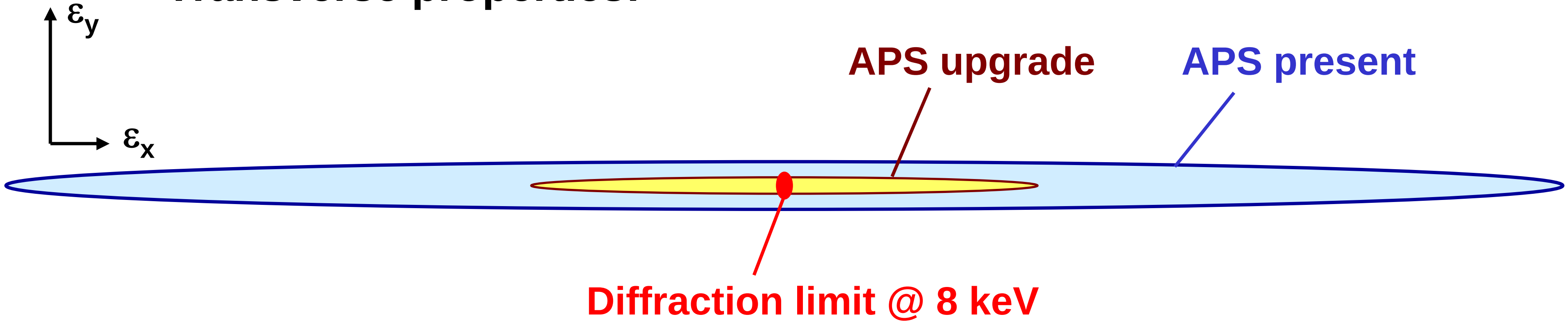
Physical Science Drivers for APS Upgrade

Qun Shen
X-ray Microscopy and Imaging Group
Advanced Photon Source

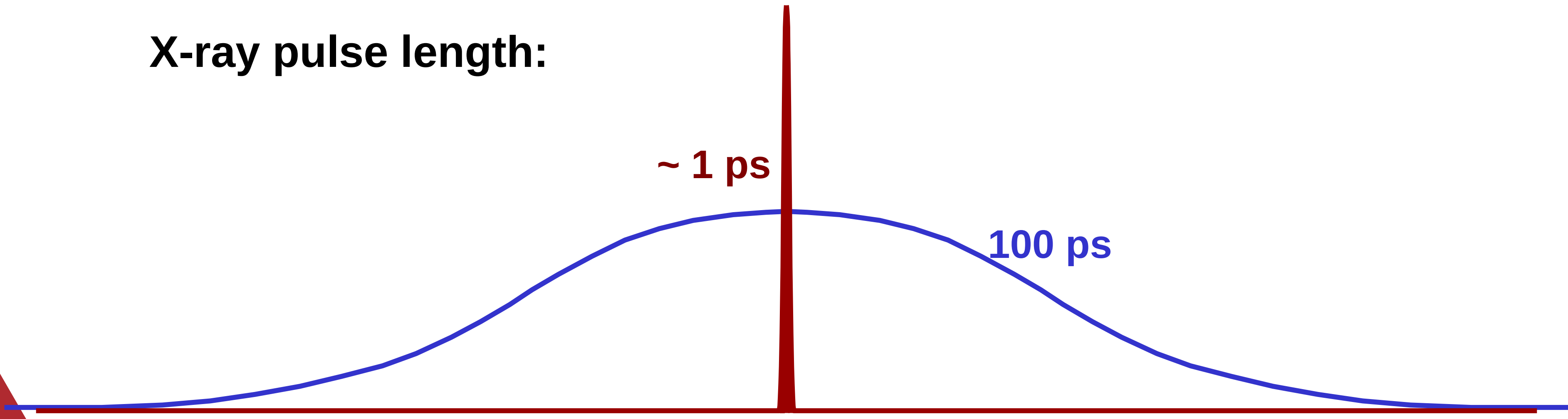
- *X-ray source properties with APS upgrade*
- *Coherent imaging*
- *Nanobeam science*
- *Short-pulse applications*
- *Implications to biology*

X-ray Source Properties with APS Upgrade

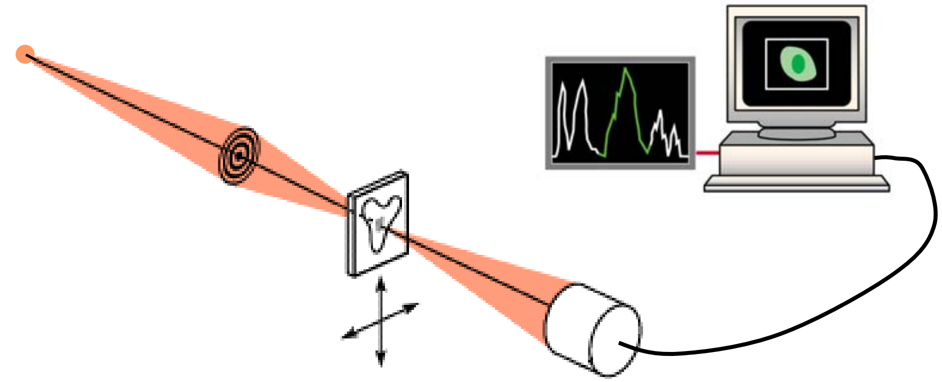
Transverse properties:



X-ray pulse length:



Advances in X-ray Science



X-ray experiment:

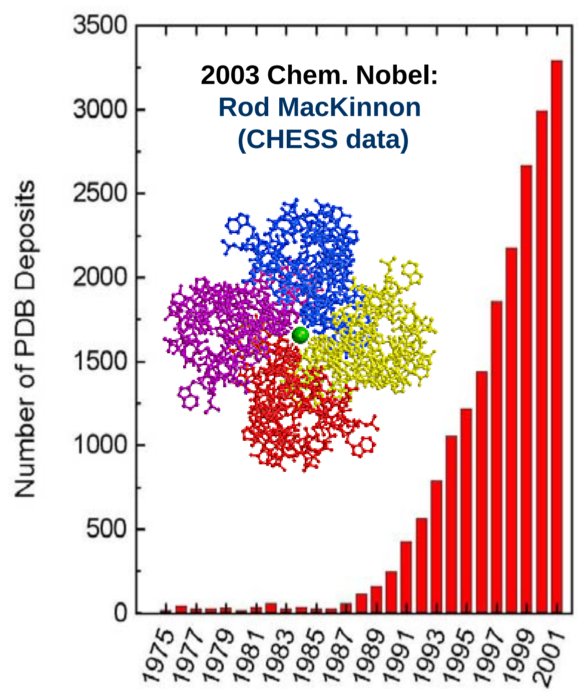
- source
- optics
- specimen
- detector
- analysis

Scientific society interests:

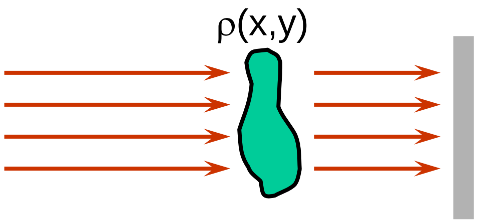
- structure → function
- crystals → noncrystalline
- homogeneous → heterogeneous
- static → dynamic / timing

Advances in X-ray capabilities:

- imaging ⇔ coherence
- nano-beams ⇔ small source
- time-resolved ⇔ short pulse

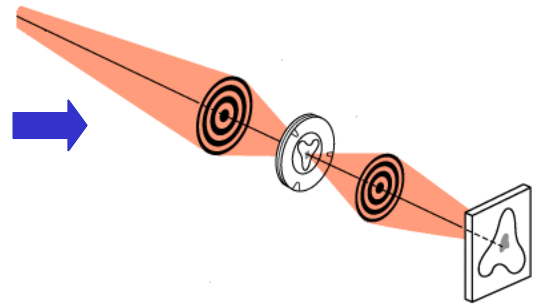


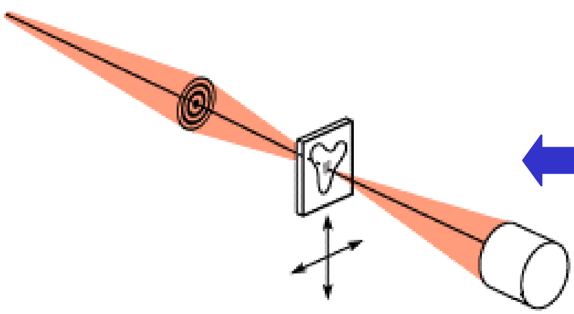
X-ray Imaging and Coherence



Direct imaging (radiography)

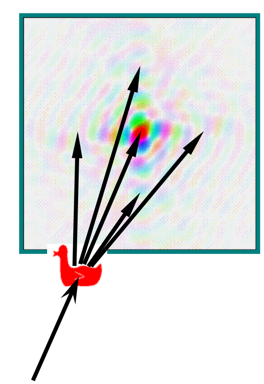
Transmission microscope (TXM)

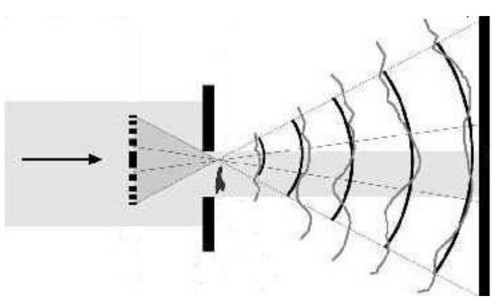




Scanning microscope (SXM)

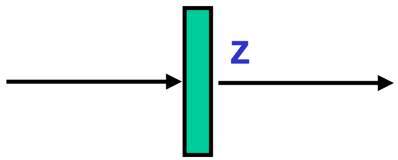
Far-field diffraction





Holography

Phase Contrast vs. Absorption Contrast Imaging

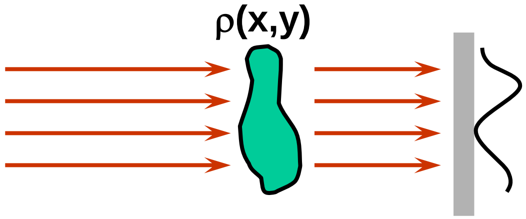


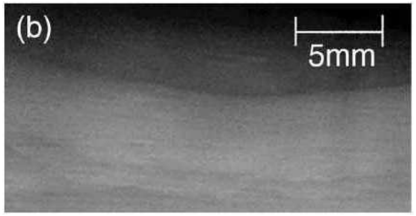
Refraction index: $n = 1 - \delta - i\beta$

$$E(z) \sim E_0\, e^{-i2\pi(-\delta - i\beta)z/\lambda} \sim E_0\, e^{i2\pi\delta z/\lambda - 2\pi\beta z/\lambda}$$

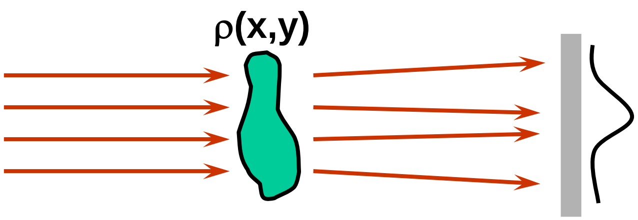
$$I(z) \sim |E(z)|^2 \sim I_0\, e^{-4\pi\beta z/\lambda}$$

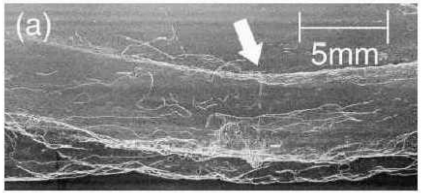
⇒ **Absorption contrast:**
$\mu z = 4\pi\beta z/\lambda \sim \lambda^3$





⇒ **Phase contrast:**
$\phi(z) = 2\pi\delta z/\lambda \sim \lambda$

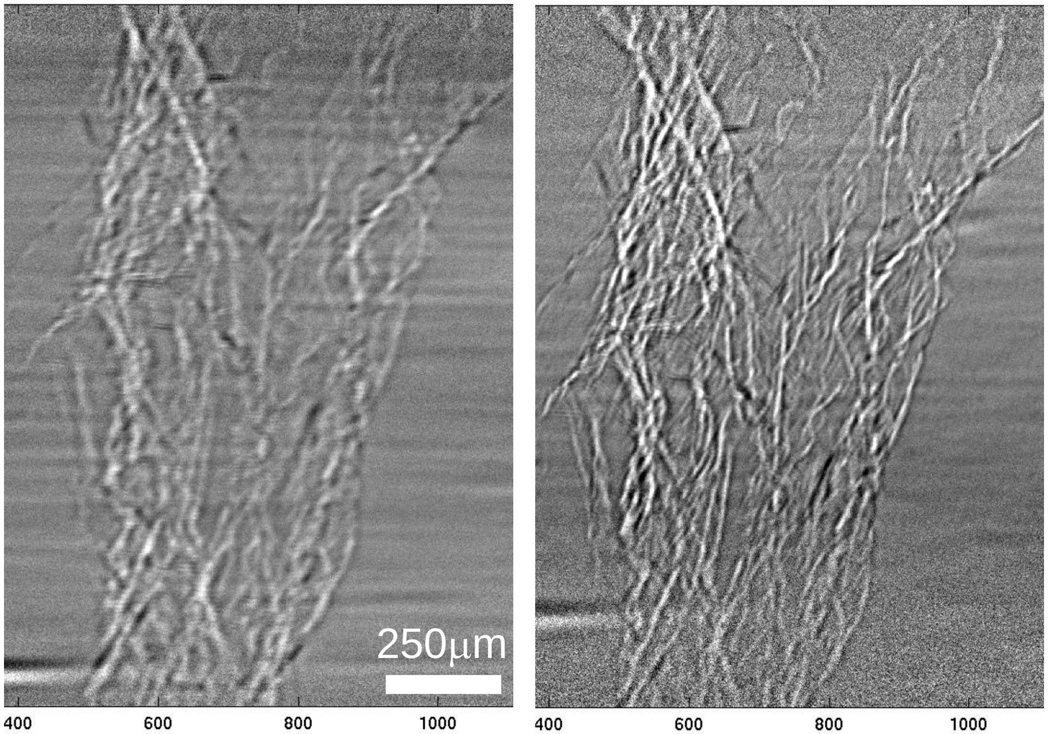


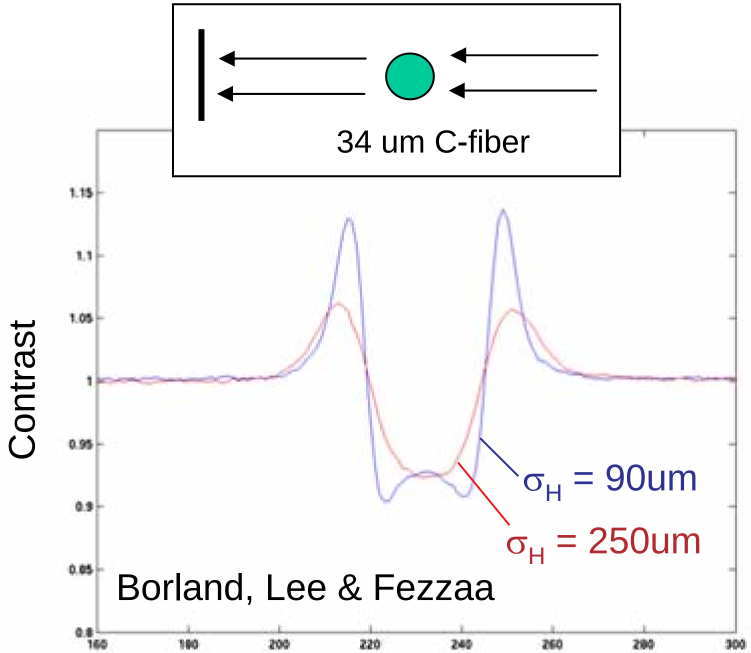


Mori et al. *(2002): broken rib with surrounding soft tissue*

Phase-Contrast Imaging Requires Local Coherence

- ***Small source size with APS upgrade would greatly enhance observable phase contrasts for weak density differences***

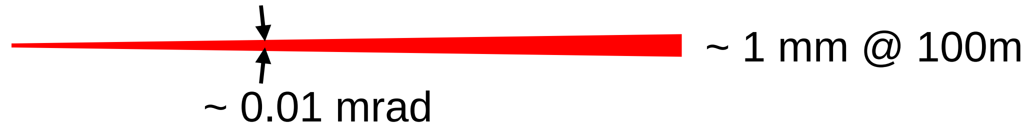




← Stress cracks in Aluminum
t = 3 mm, 30 keV, D = 1m

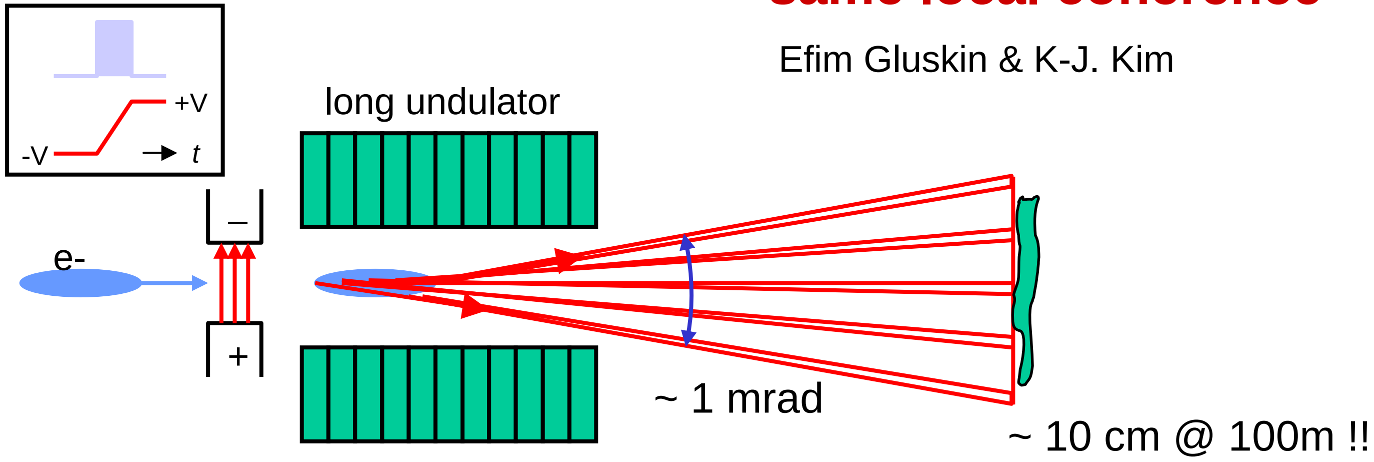
Improving Phase Contrast Imaging

- **Normal undulator beam: good coherence**
  but small field of view



- **RF-deflected undulator beam: large field-of-view**
  same local coherence

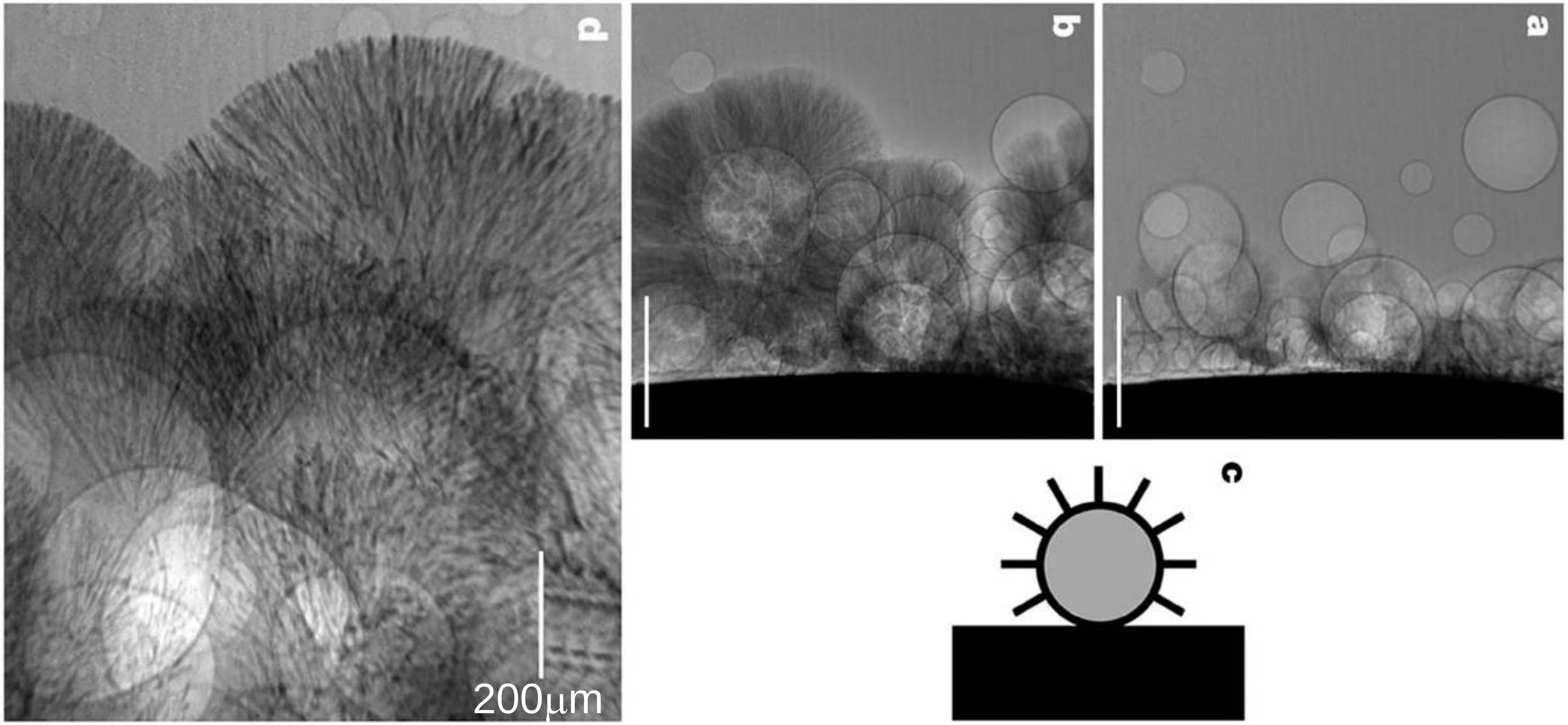
Efim Gluskin & K-J. Kim



In-situ X-ray Imaging of Electrodeposition

Tsai et al, "Building on bubbles in metal electrodeposition", Nature 417, 139 (2002)

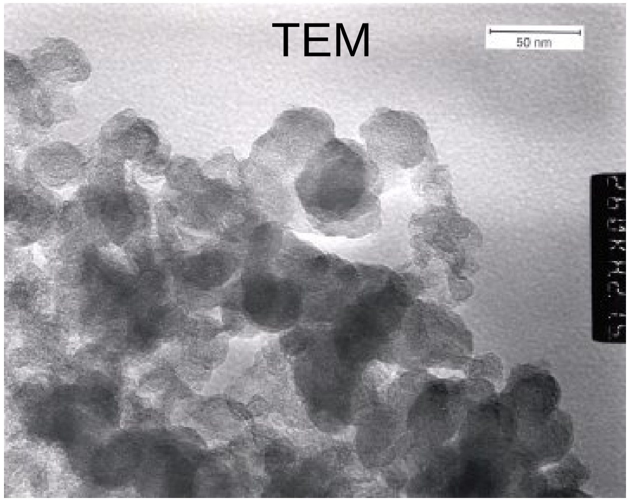
In the electrodeposition of metals, a widely used industrial technique, bubbles of gas generated near the cathode can adversely affect the quality of the metal coating. Phase-contrast imaging is used to witness directly and in real time the accumulation of zinc on hydrogen bubbles.

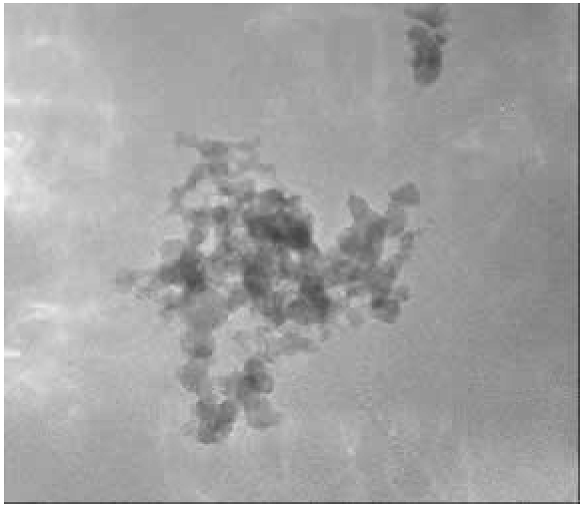


Phase Contrast Imaging with APS Upgrade

- Time-resolved imaging at unprecedented temporal resolution with few-ps single pulse capability and sub-μm microscopic details, limited only by fundamental sound velocity ~ 1 km/sec or 1 nm/ps

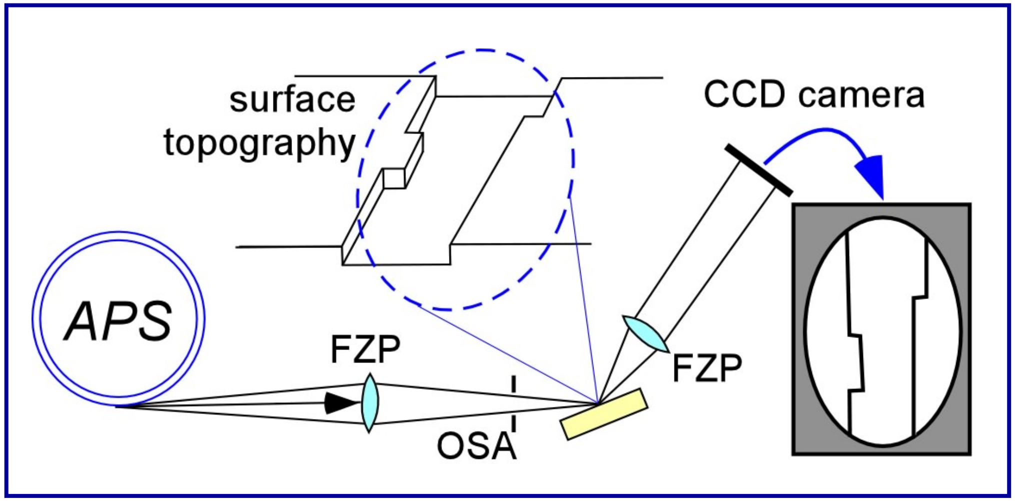
- Direct real-time imaging of low-contrast materials processing and depositions, such as formations of carbon particulates in engines, polymer aggregates and polymer thin-film coatings



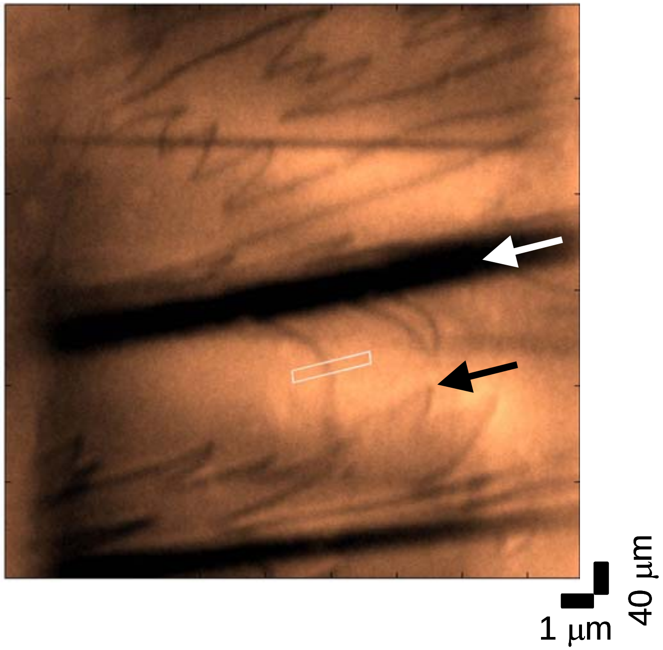


Phase-Contrast X-ray Diffraction Microscopy

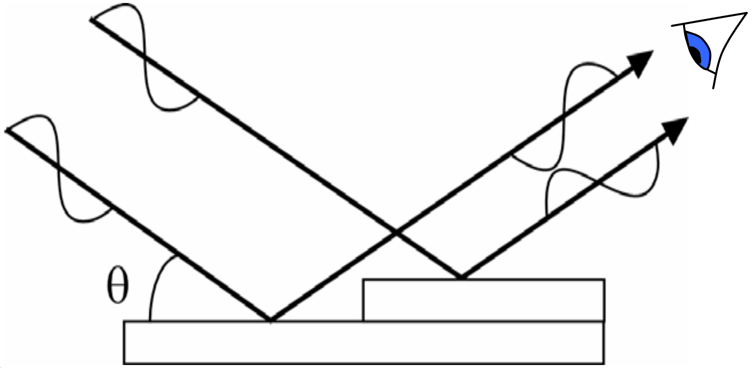
*P. Fenter, C. Park, Z. Zhang, and S. Wang, submitted (2006)



Step distributions on $KAlSi_3O_8$ (001)

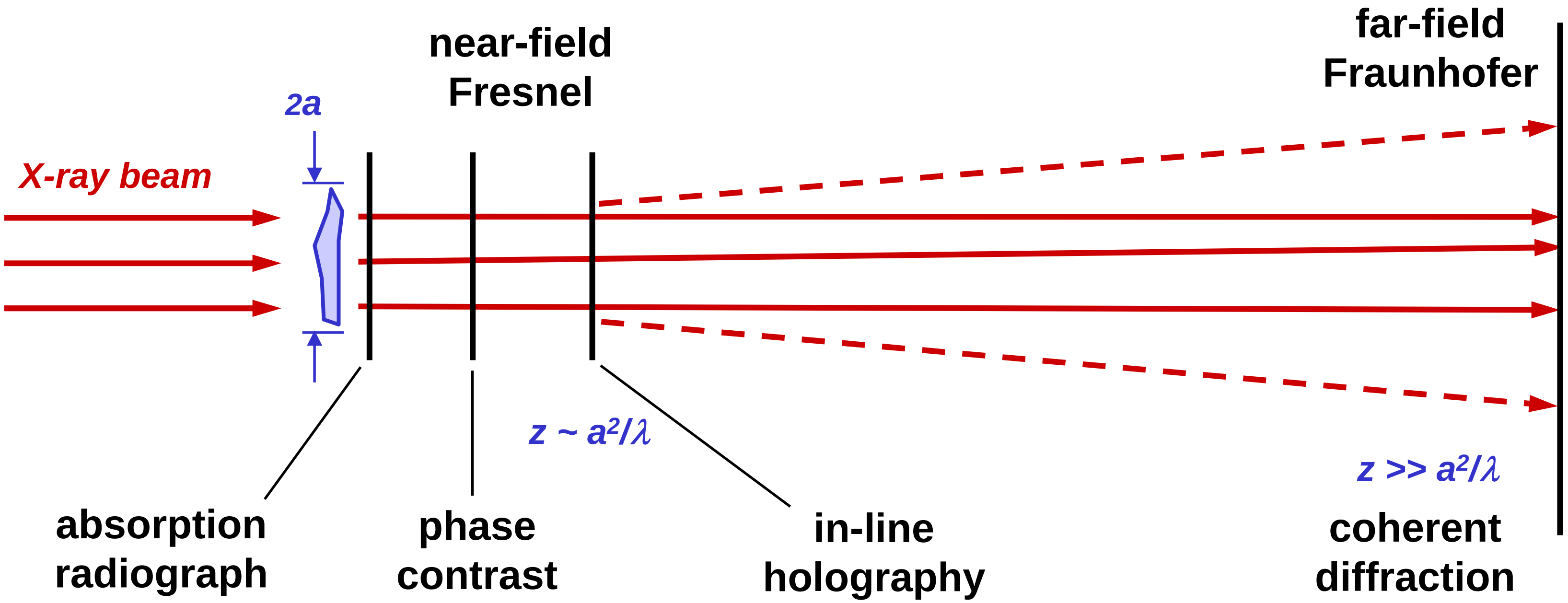


X-ray Reflection Interface Microscopy

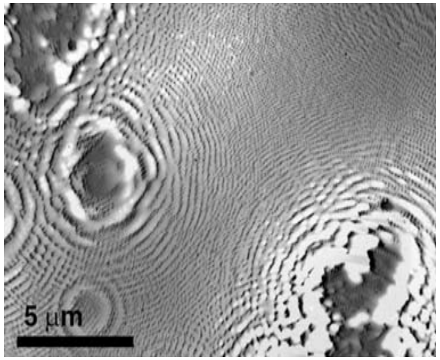


Non-invasive structural tool (no probe tip):
- reactions in aggressive chemical conditions
- extreme pH, corrosive gases
- elevated temperature
- buried interfaces
- in-situ, real-time observations of interfacial reactions

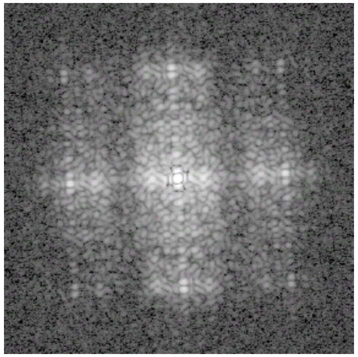
Different Regimes of X-ray Imaging



Kagoshima et al.
JJAP (1999).

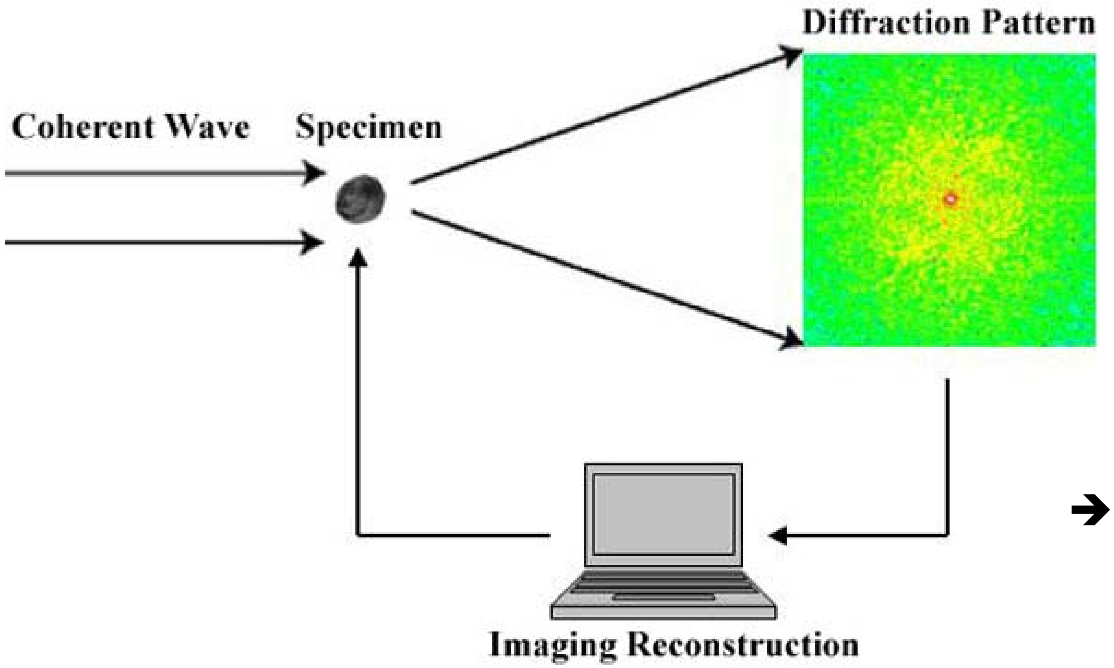


Jacobsen (2003).



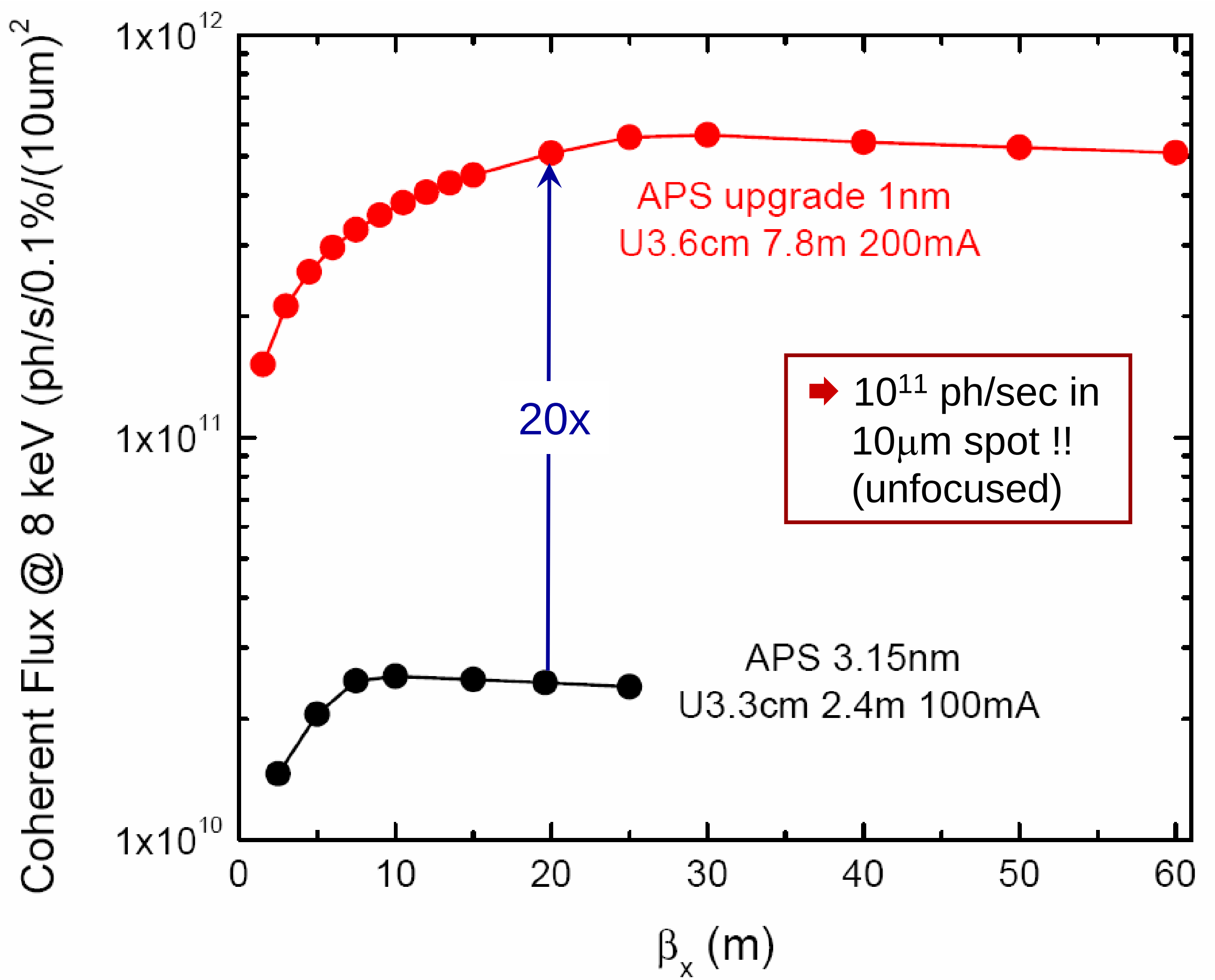
Miao et al.
Nature (1999).

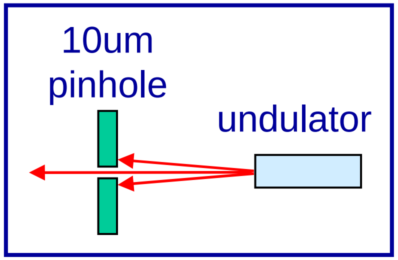
Coherent X-ray Diffraction Imaging



- Coherent diffraction imaging is much like crystallography but applied to noncrystalline materials
- First proposed by David Sayre in 1980, and first experimental demonstration by John Miao et al in 1999 using soft x-rays
- Requires a fully coherent x-ray beam

Effects of APS Upgrade on Coherent Diffraction





$$\sigma_x = \sqrt{\varepsilon_x \beta_x}$$

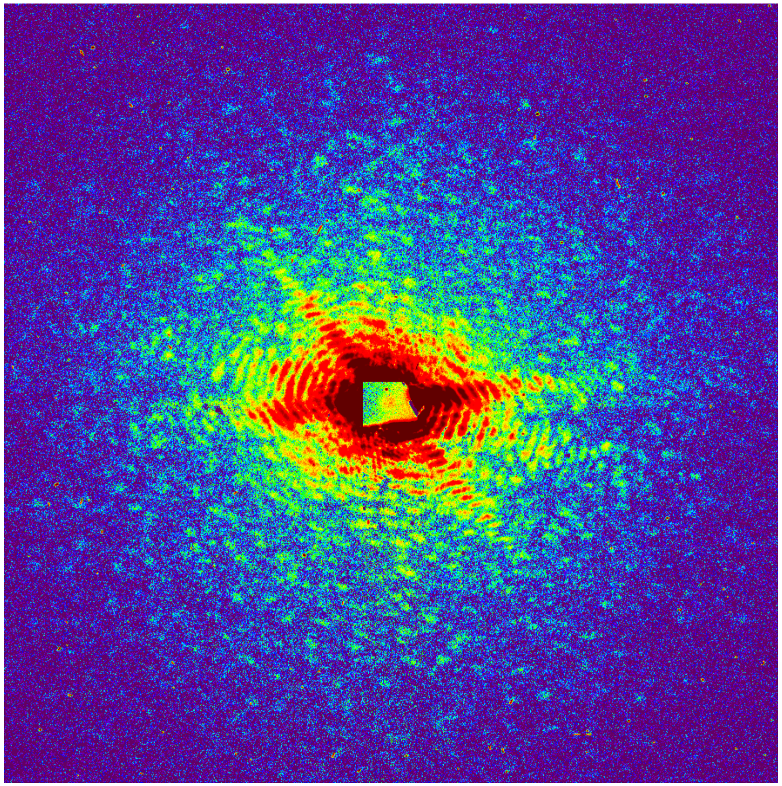
$$\sigma_{x'} = \sqrt{\varepsilon_x / \beta_x}$$

In addition:

- Detectors: faster readout, high sensitivity & dynamic range
- Improve S/N by reducing background

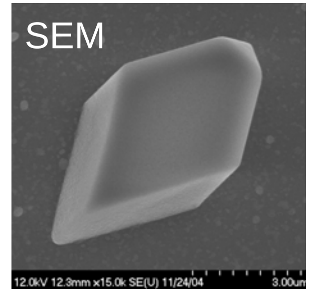
Improving S/N with Crystal Guard Aperture

Xiao, de Jonge, Chu & Shen, submitted (2006)



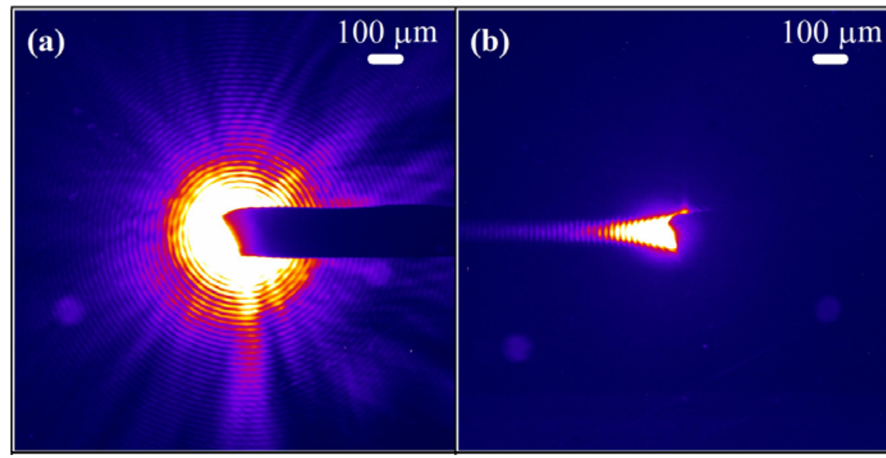
X-ray energy: 8.16 keV

Highest angle signal ⇔ ~45nm resolution

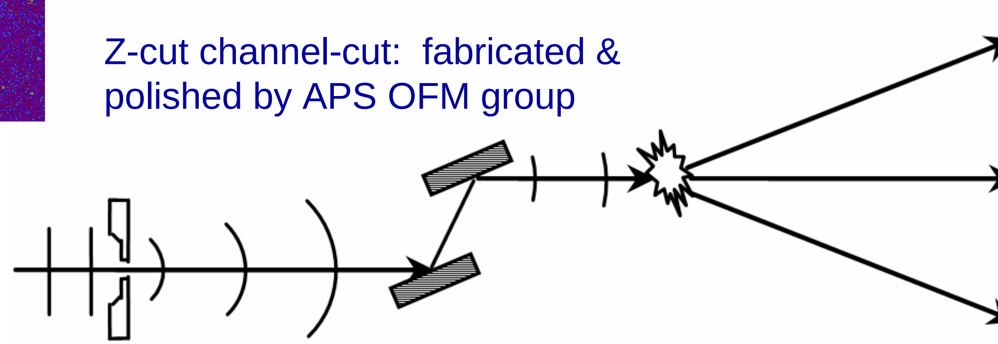


Sample: Y. Xiao

Pb microparticle electrodeposited on Si-N window

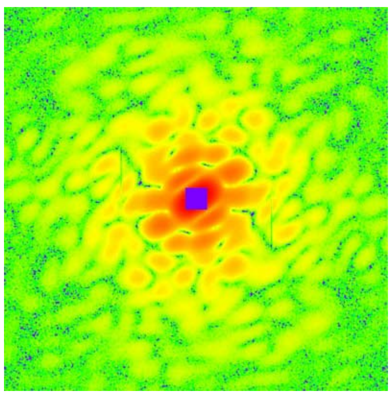


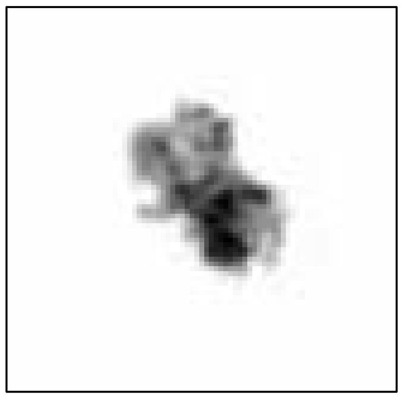
Z-cut channel-cut: fabricated & polished by APS OFM group



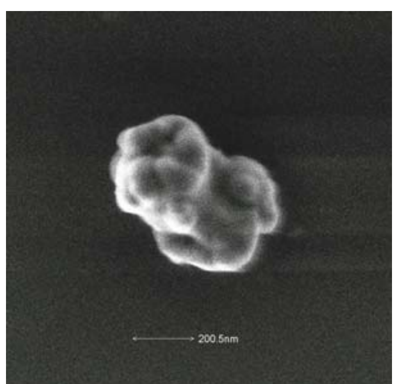
Internal Structure and Strain of Nanoparticles

Miao et al, "Quantitative Image Reconstruction of GaN Quantum Dots from Oversampled Diffraction Intensities Alone", ***Phys. Rev. Lett.*** **(2005)**

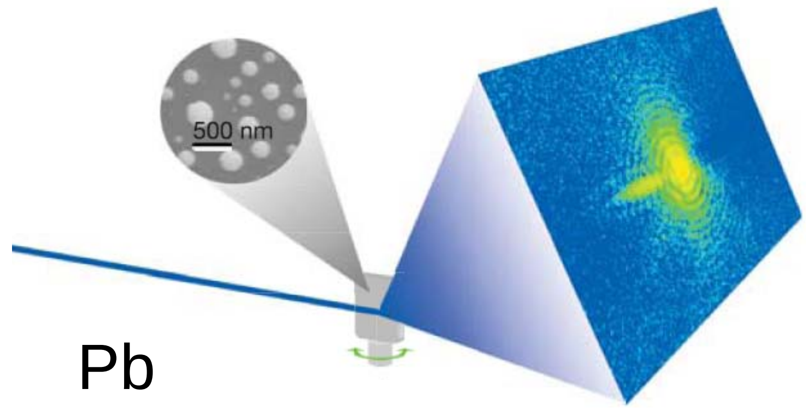




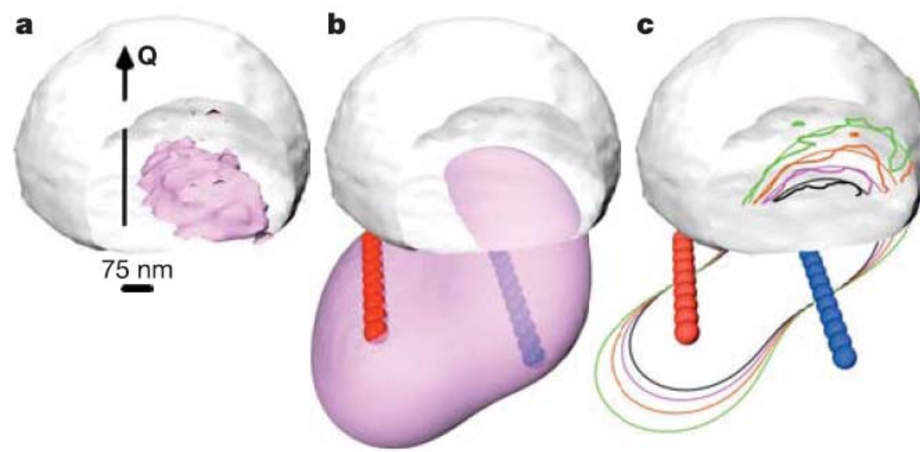
GaN



M.A. Pferfer et al., "Three-dimensional mapping of a deformation field inside a nanocrystal", Nature 442, 63 (2006)

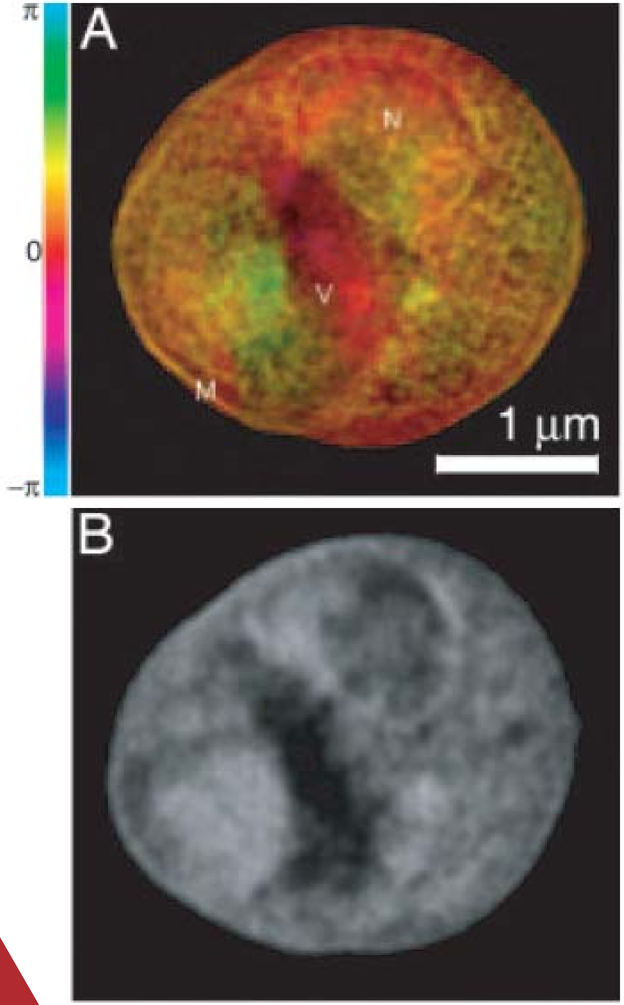


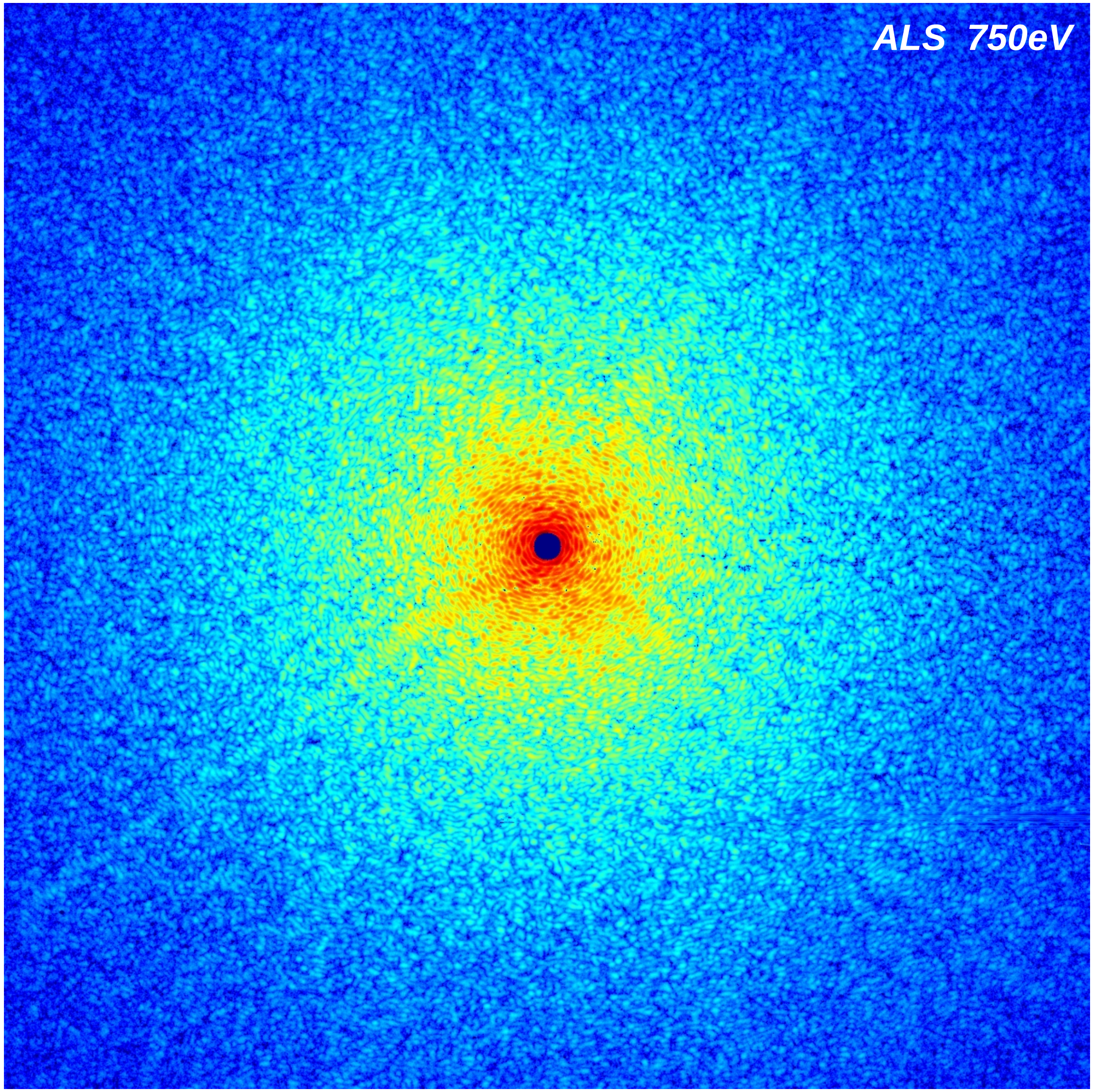
Pb



Coherent Diffraction Imaging of Freeze-dried Yeast Cell

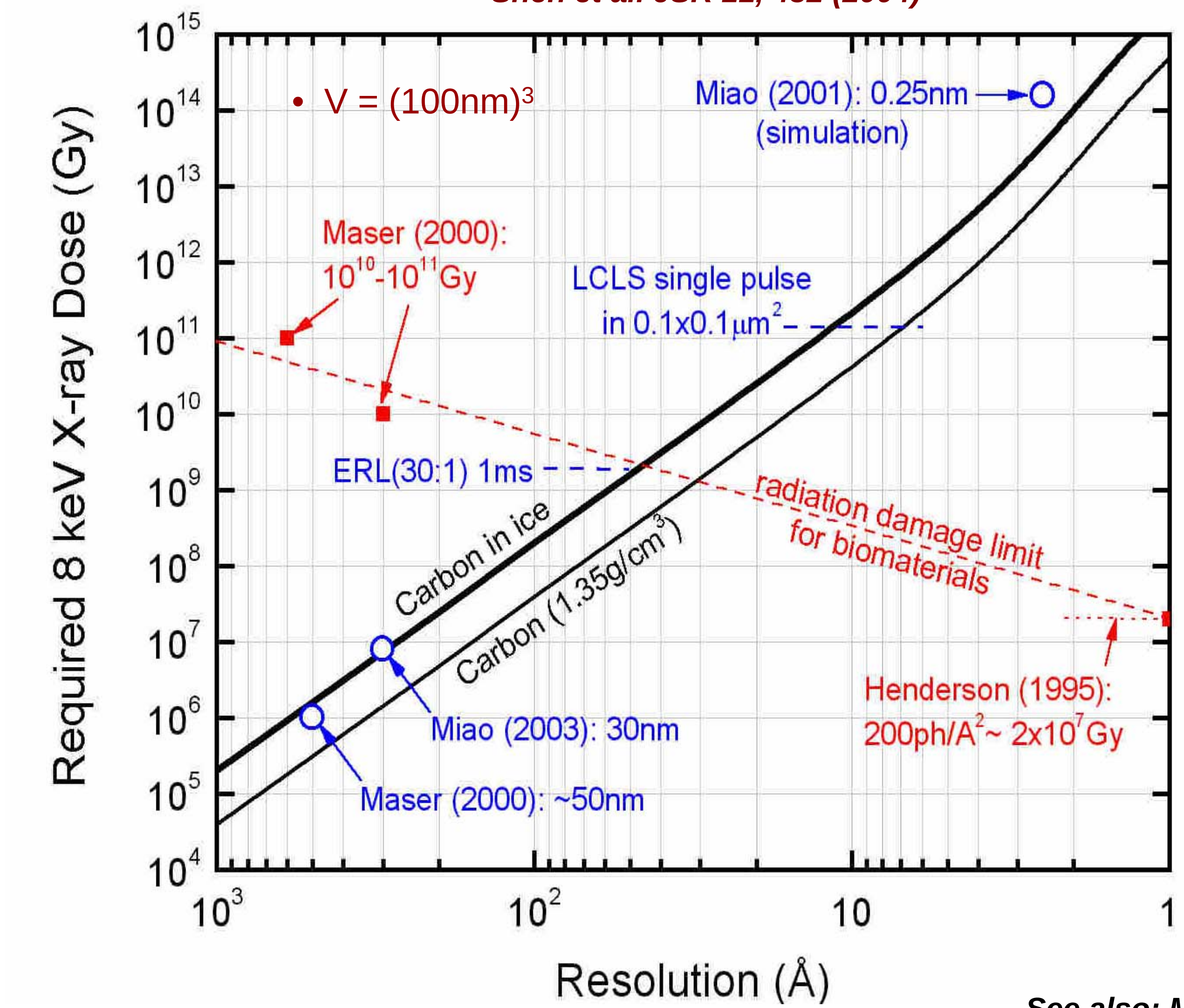
Shapiro et al. PNAS 102, 15343 (2005)





Dose vs. Resolution & Radiation Damage (biomaterials)

Shen et al. JSR 11, 432 (2004)



$$Dose = \frac{(I_0 \Delta t)\mu \cdot E}{\rho}$$

To go beyond damage limit:

→ Femtosecond diffraction with XFEL pulses [Hajdu 2000]

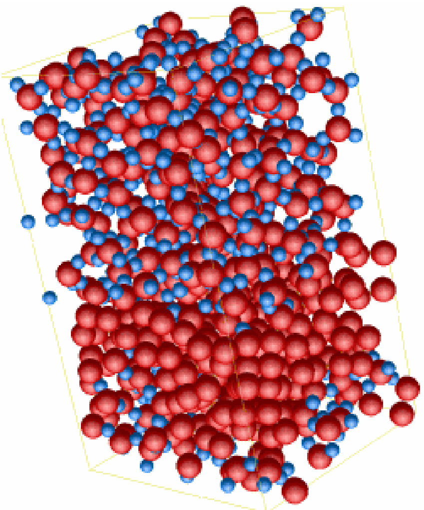
→ Self-assembled macromolecule array

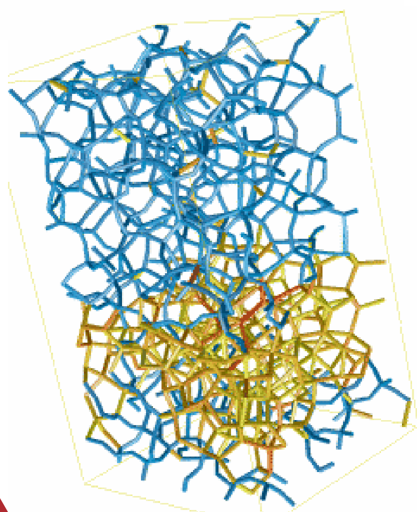
→ Continuous stream of molecules oriented by laser [Spence 2004]

See also: Marchesini et al. Opt. Express (2003)

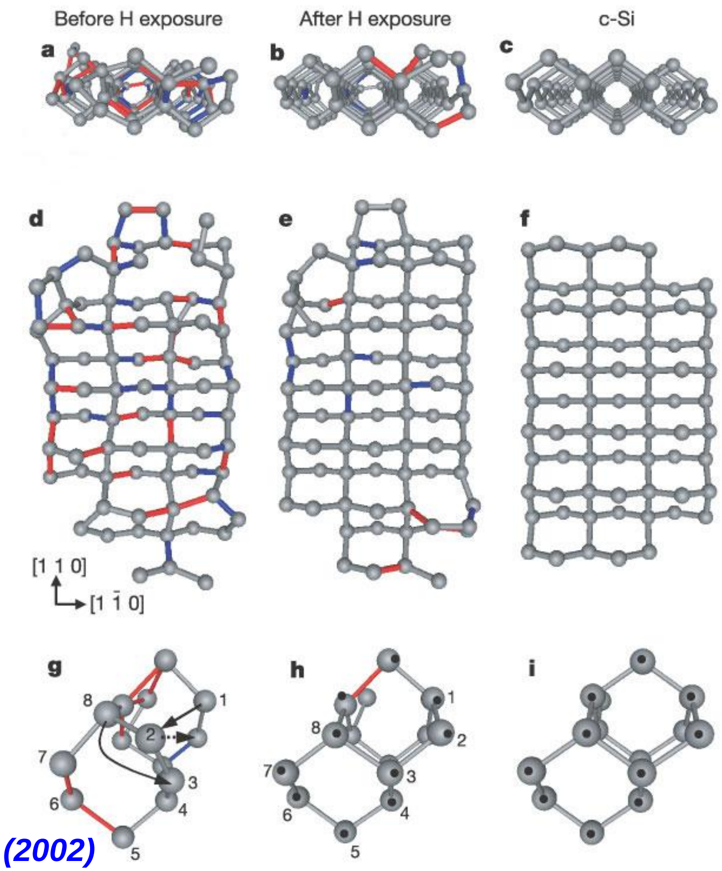
New Opportunities with Coherent Diffraction Imaging

Veit Elser (Cornell)

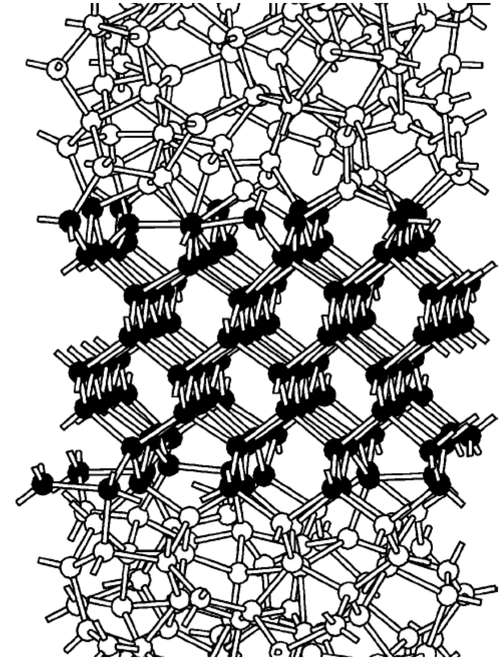




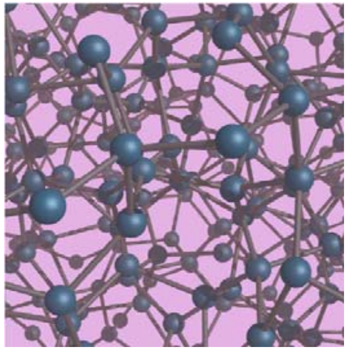
Amorphous Silicon (α-Si):
- atomic resolution structure data still lacking
- one of grand challenges in solid state physics



Nature 418, 62 (2002)



PRB 58, 4579 (1998)

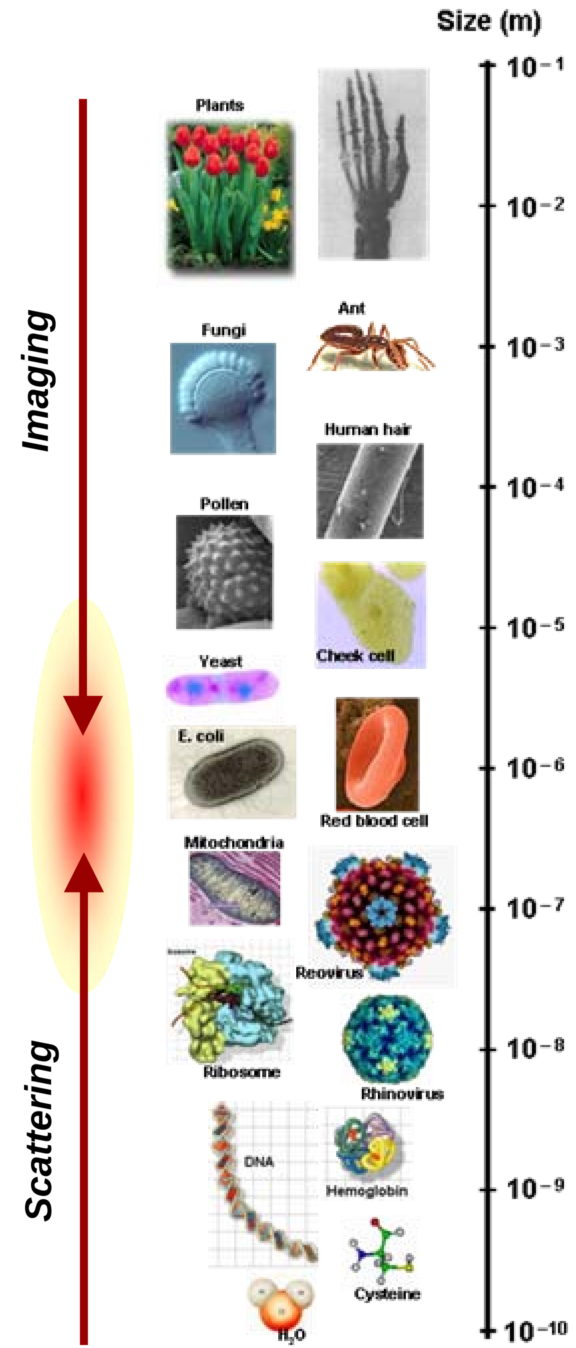


Thoughts on APS Upgrade & CDI

- ***Coherent X-ray Diffraction Imaging** is an exciting research field with many potential applications, but requires further R&D and optimizations of both experiments and theory.*

- ***Upgraded APS** is expected to deliver ~20x more coherent hard x-rays in typical CDI experiments. Along with R&D in optimized experimental conditions, APS offers excellent opportunities to play a leading role in realizing the full potentials of emerging CDI applications.*

- ***Emerging Applications**:*
  -- structure and strain in nanoparticles
  -- atomic structure of amorphous materials
  -- 2D crystallography for e.g. membrane proteins
  -- few unit-cell crystals
  -- organelle structures in biological cells
  -- laser-oriented macromolecules
  -- bio-organic-inorganic hybrid structures



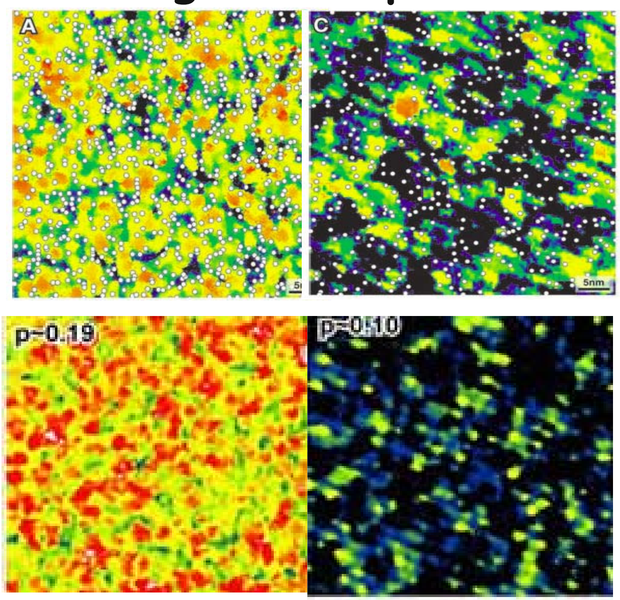
Shen et al, Phys Today (March 2006)

Domains and domain wall fluctuations

Oleg Shpyrko & Eric Isaacs

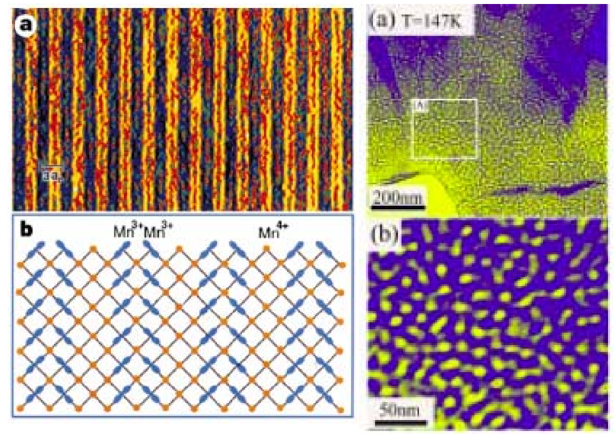
Strongly competing spin, charge, orbital, lattice degrees of freedom: inhomogeneities

High-Tc cuprates:



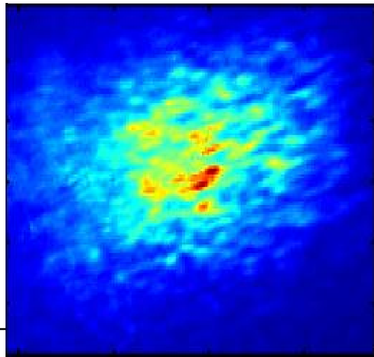
K. McElroy, S. Davis et al., *Science* (2004)

CMR manganites:



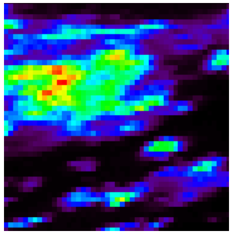
S. Mori et al., *Nature* (1998)
M. Uehara et al., *Nature* (1999)

XPCS: Coherent x-ray speckle image

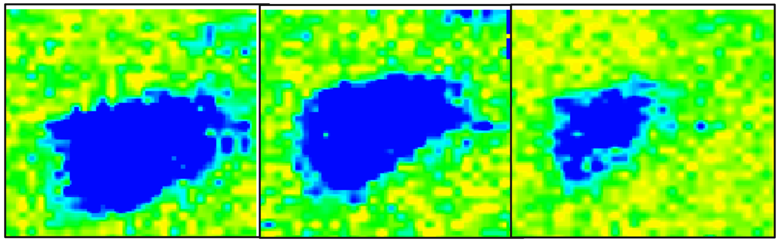


AFM Chromium:

charge-:



and spin-density wave domains:



P. G. Evans, E. D. Isaacs, et al., *Science* (2002)

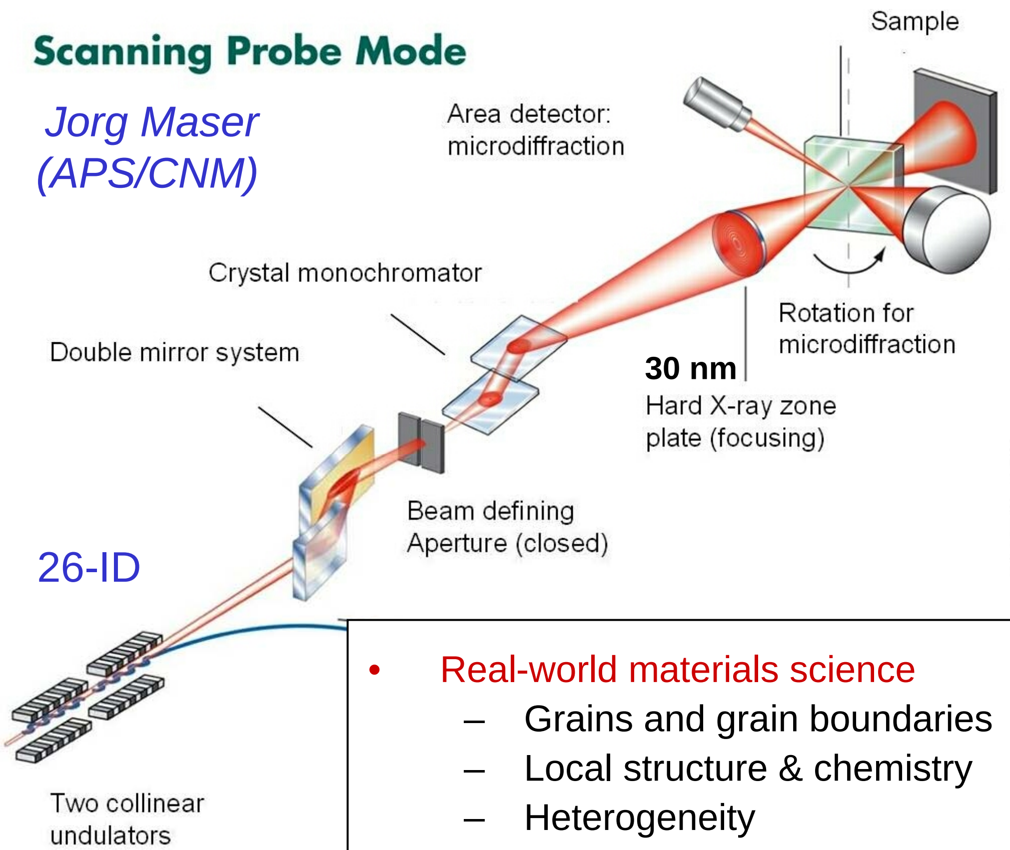
Science with Nano-focused X-rays

APS/CNM Nanoprobe

Scanning Probe Mode

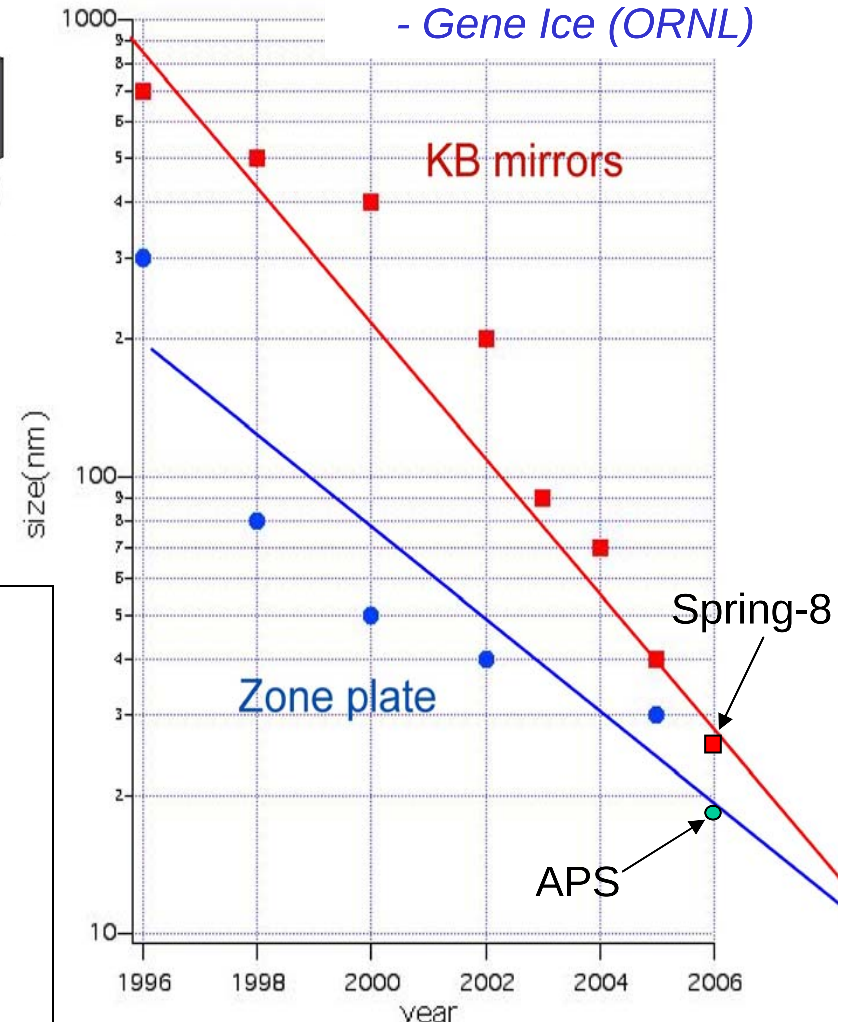
Jorg Maser (APS/CNM)

26-ID



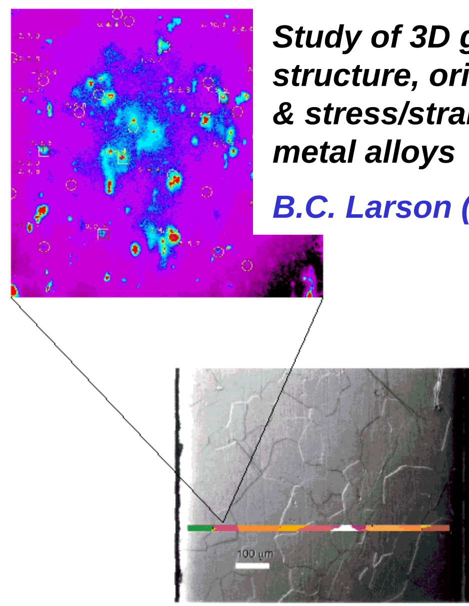
- Real-world materials science
  - Grains and grain boundaries
  - Local structure & chemistry
  - Heterogeneity
  - Defects/ correlations
- Trace elements in biology
  - Biological cells
  - Soft tissues
  - Bionanotechnology

Small beams change everything!



X-ray Imaging of Functional Units in Materials Science and Biology

- Nano-focus beamline will be capable of focusing 10-20 keV x-rays down to 5 nm in size, using latest x-ray optics developed at the APS and CNM
- Nanometer beam and improved detectors will provide unprecedented elemental sensitivity to sub-zepto ($<10^{-21}$) grams for trace metals (e.g. Zn, Fe, Mn) in biological cells, with potential to locate single metal atoms at 5-nm resolution
- Enable molecular imaging of metal-containing proteins, functional contrast agents, and novel therapeutic drugs at the organelle level
- Single-atom detectability for materials science will offer a non-destructive penetrating probe for impurity/defect studies and nano-domain engineering of functional electronic materials such as solar cells

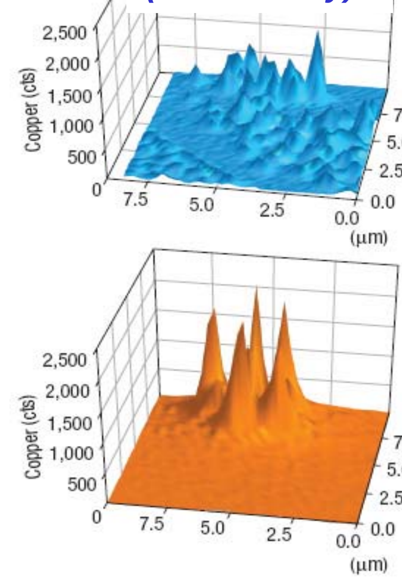


Study of 3D grain structure, orientation & stress/strain in metal alloys

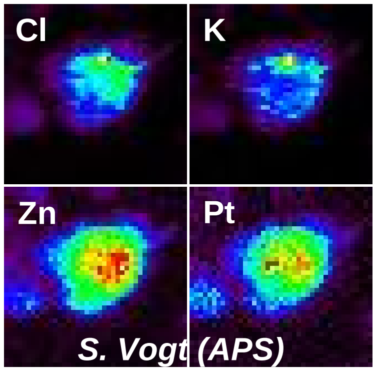
B.C. Larson (ORNL)

Metal impurities in solar cells

T. Buonassisi (UC Berkeley)



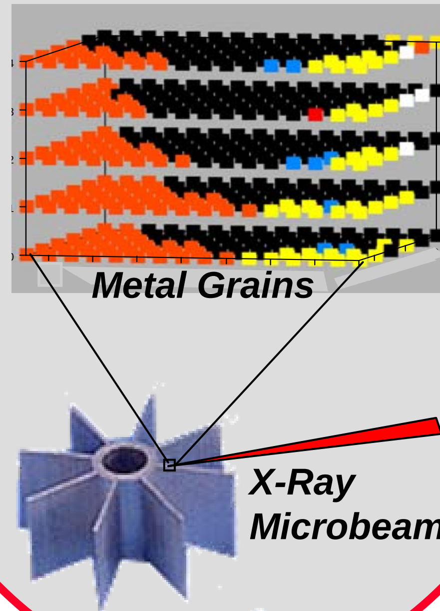
Elemental distribution in a cisplatin-treated cancer cell



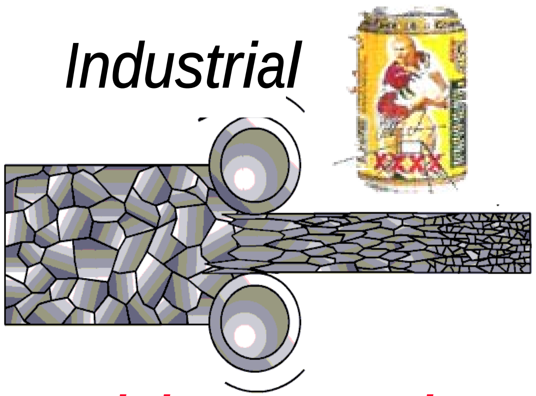
Three-Dimensional X-Ray Structural Microscopy

Submicron Resolution
Nondestructive
General

3-D X-Ray Structural Microscopy

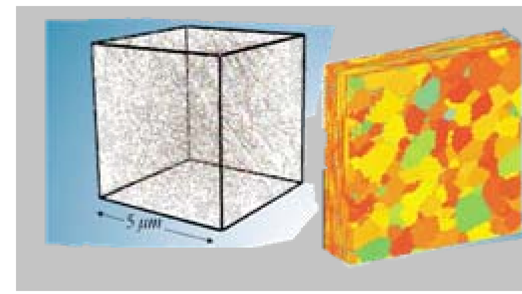


Industrial



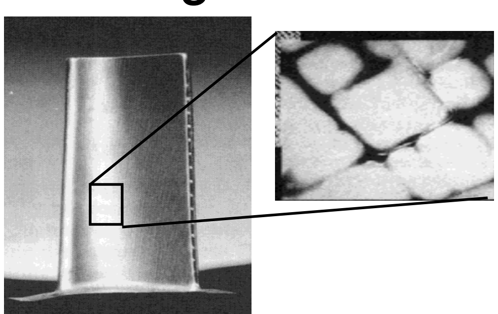
Materials Processing

Fundamental



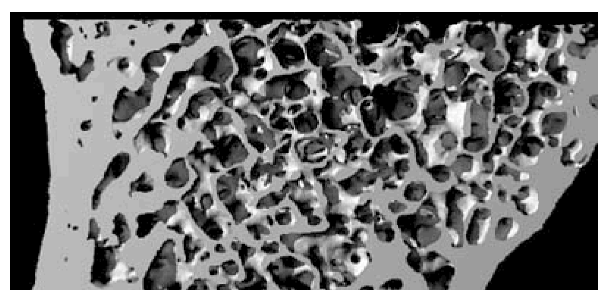
Multi-Scale Modeling

Technological

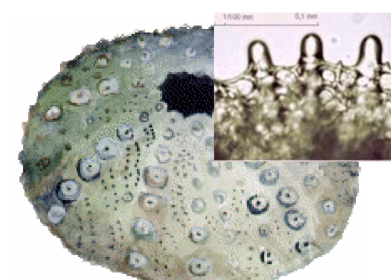


Turbine Blades

Biological



Bone Structure



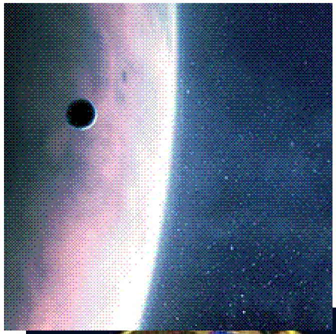
Marine Structures

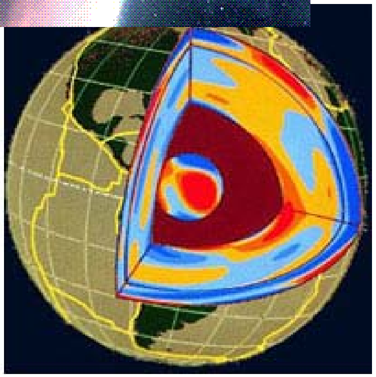
New High-Pressure Frontiers with Nanofocus

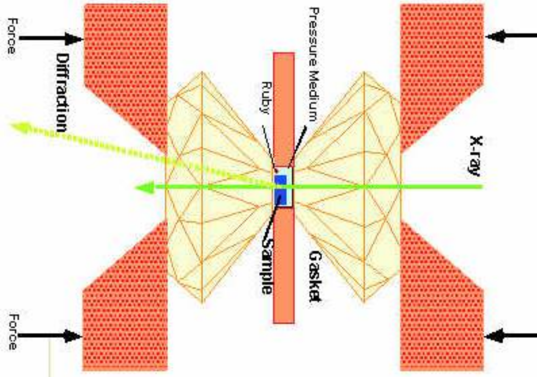
David Mao (Carnegie Institute of Washington)

- Terapascal pressure with nanoscale probes – With tens of micrometer anvil tip and optical-x-ray probe, major breakthroughs were achieved in the 1970-80's to advance sustainable pressures from 50 to 350 GPa. With the new prospects of nanofabrication of diamond anvil tips and new facilities to probe high-pressure samples with tens of nanometer resolution, we can expect the next breakthroughs of reaching TPa pressures and opening the vast new areas for discovery of myriad novel materials and phenomena at nanoscale.

- Warm condensed matter at high pressures -- Up to 6,000 K temperatures have been achieved with laser-heating at high-pressure as a result of coordinated development of 5 micrometer x-ray probe on tens of micrometer focused laser-heating area. With the prospect of tens of nanometer x-ray probe, the heating spot can be reduced to the diffraction limit of the heating laser (<1 um) and the maximum temperature could be greatly extended above one electron volt (12,400 K), thus enabling static high-pressure research at warm condensed matter region including applications to astrophysics and high energy-density materials.



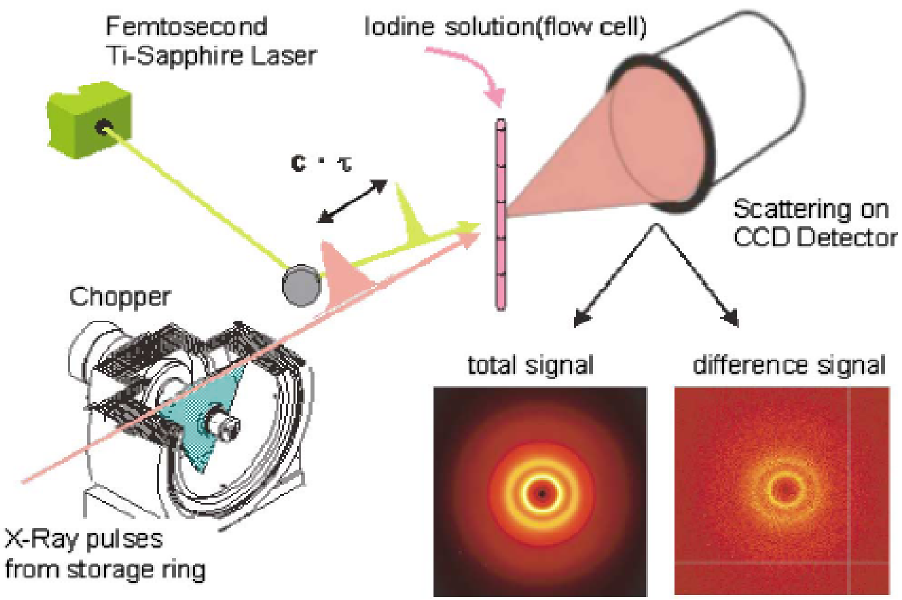


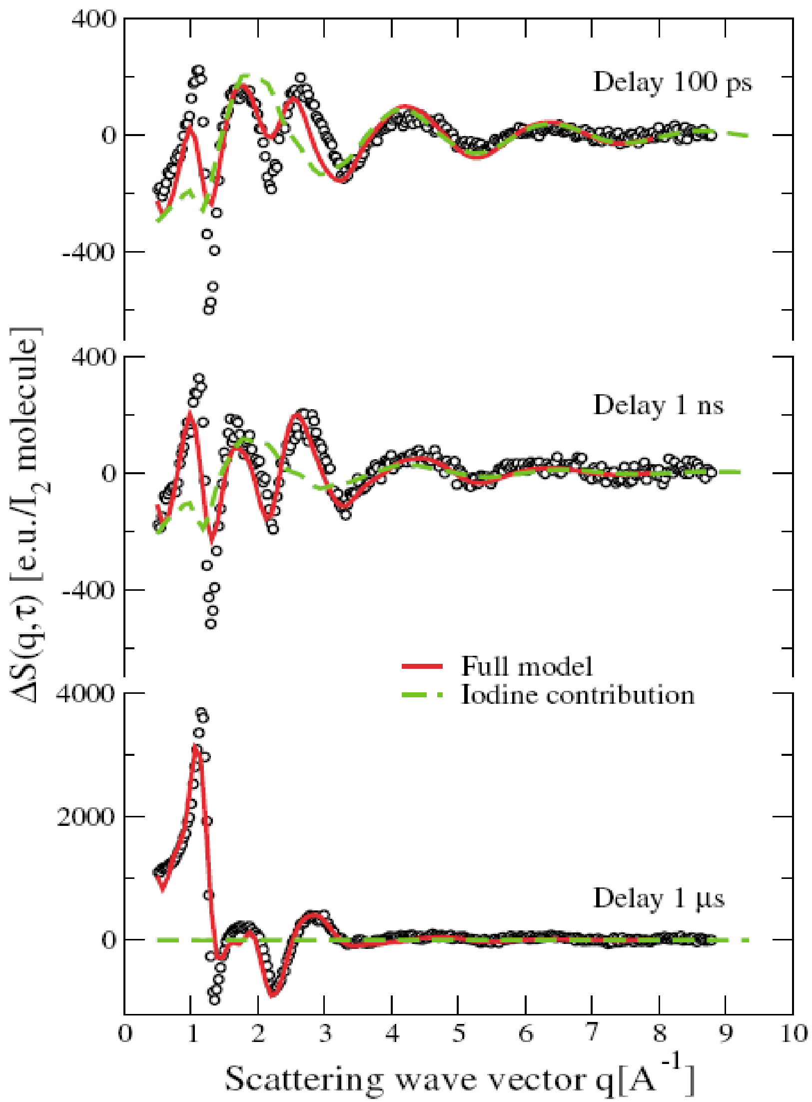


Science with Short X-ray Pulses

Plech, Wulff, et al. PRL (2004)

Visualizing Chemical Reactions in Solution by Picosecond X-Ray Diffraction



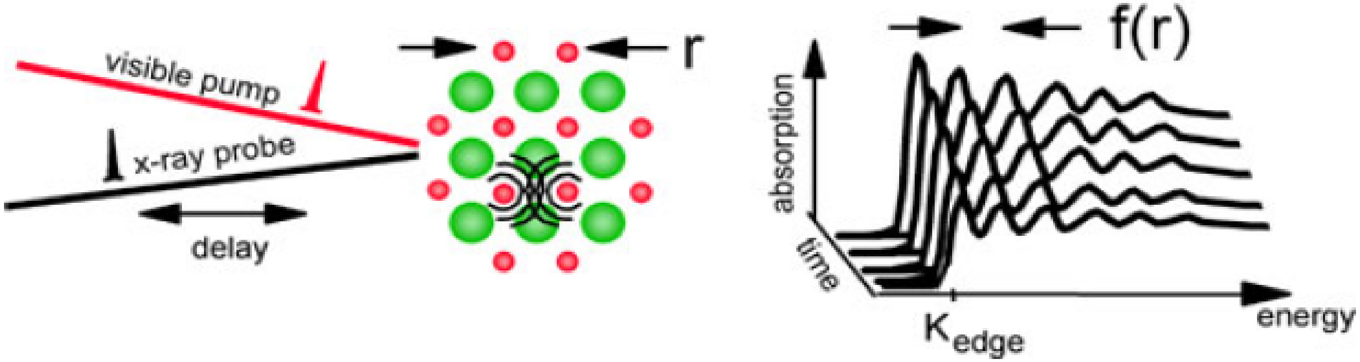


ESRF

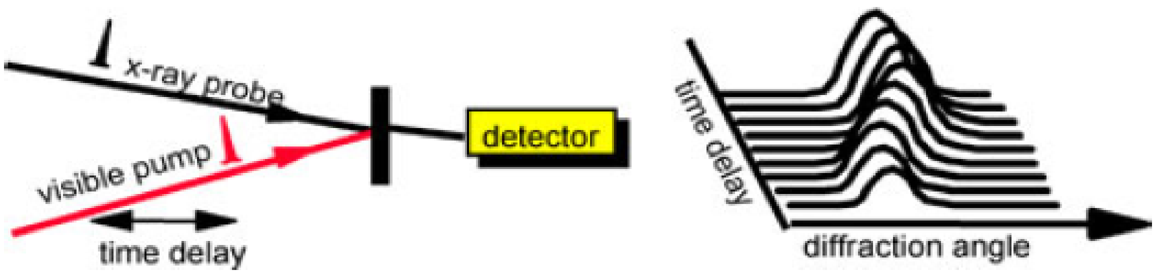
Laser-pump, X-ray probe Experiments

D. Reis (Michigan)

Time-resolved EXAFS, NEXAFS, surface EXAFS



Time-resolved x-ray diffraction

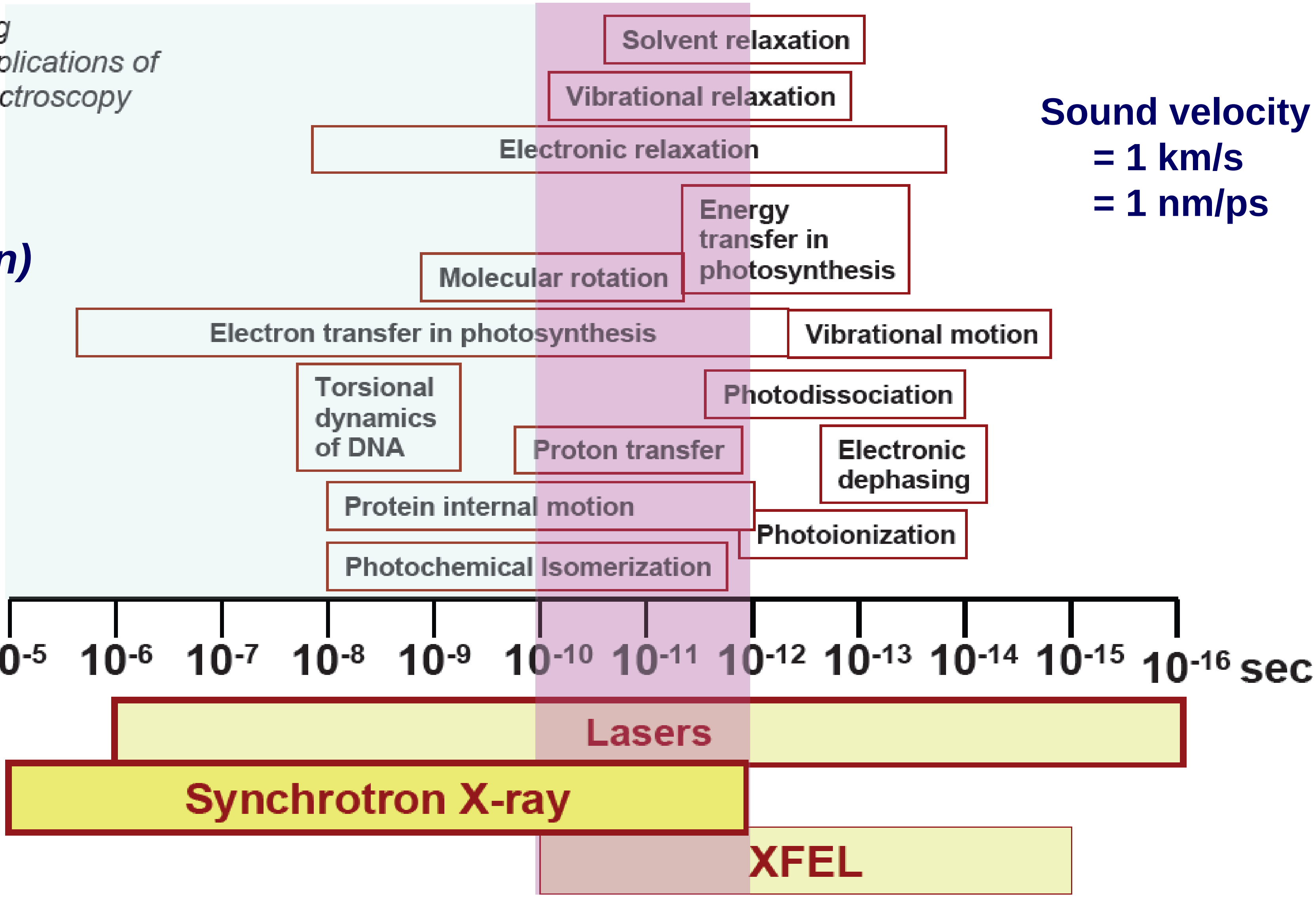


Multiple Temporal Scales in Chemical Sciences

G. R. Fleming
Chemical Applications of Ultrafast Spectroscopy
1986

D. Reis (Michigan)

Sound velocity = 1 km/s = 1 nm/ps

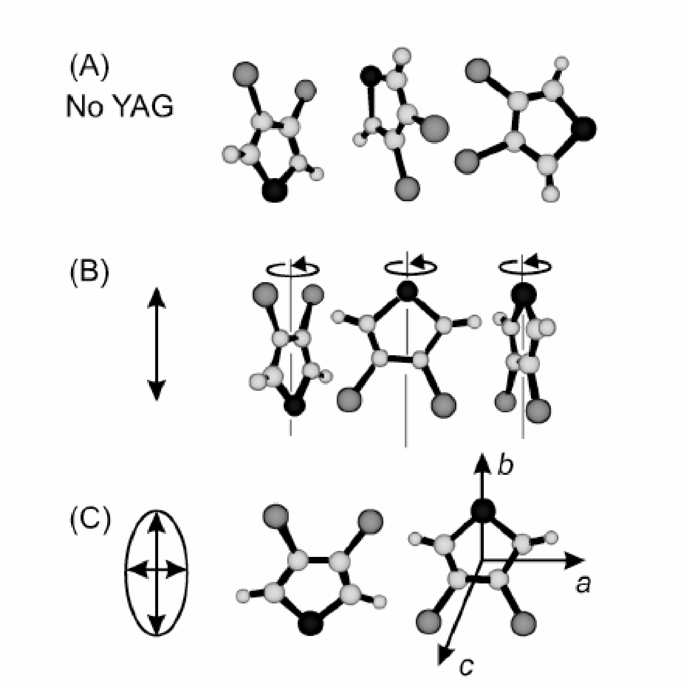


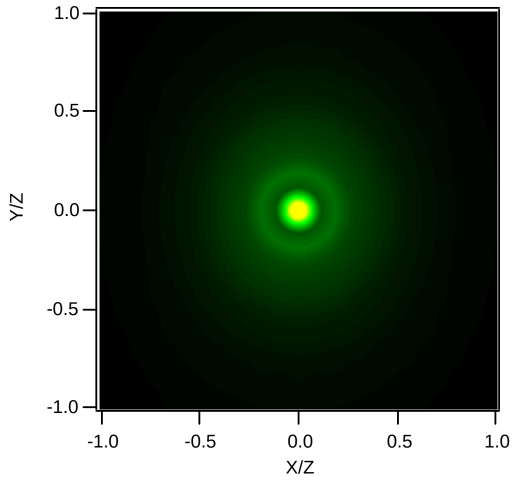
Probing Transient Molecular Structures in Photochemical Processes Using Pulsed X-rays

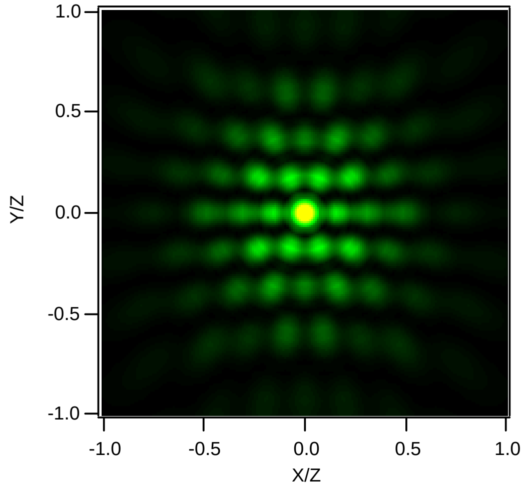
Laser Alignment of Complex Molecules

Linda Young (ANL) - Cornell ERL Workshop (2006)

3-D alignment w/elliptically polz'd fields
3,4 dibromothiophene







J.J. Larsen et al., PRL 85, 2470 (2000)

Summary

❖ ***APS Upgrade*** ***is an exciting opportunity to expand beyond the current capabilities (coherence, small source, short pulse) to satisfy growing interests in physical science community***

❖ ***These interests*** ***include:***

in-situ imaging of chemical reactions and materials processing;
structural studies of non-crystalline or nanocrystalline materials;
3D mapping of local structures & defects at nm-scale;
high-pressure frontiers beyond planetary astrophysics;
dream to follow chemical reactions at ps to sub-ps levels;
……

❖ ***Many of the potential advances*** ***will have direct implications to biology and life sciences***

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Thank You !