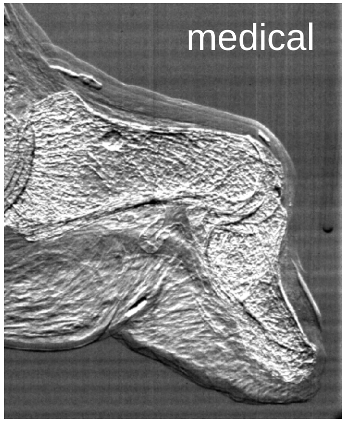
Argonne NATIONAL LABORATORY

X-ray Imaging with Current Synchrotron and Future ERL Sources

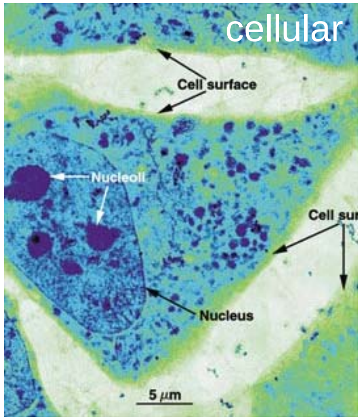
Qun Shen
X-ray Microscopy and Imaging Group
Advanced Photon Source

- *Introduction to X-ray imaging*
- *Scanning x-ray microscopy*
- *Phase-contrast & ultrafast imaging*
- *Opportunities with ERL source*
- *Summary*

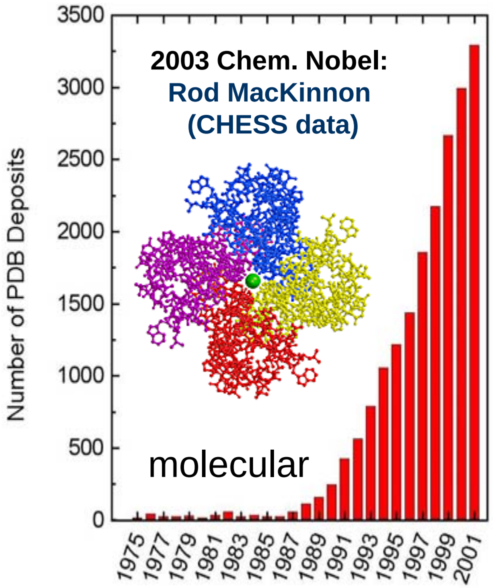
Advances in X-ray Imaging



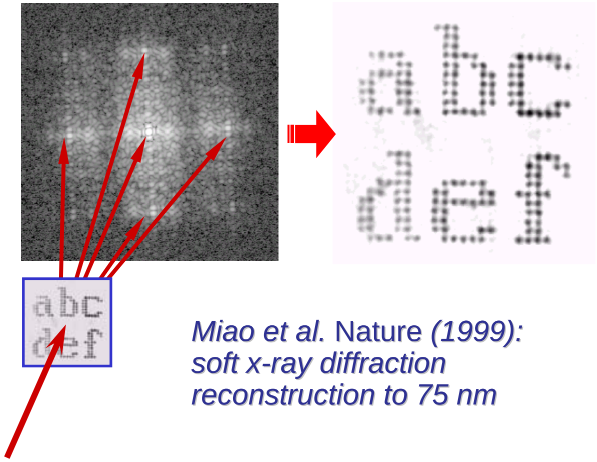
Li, Zhong, et al. (2003)



Larabell (ALS, XM-1)

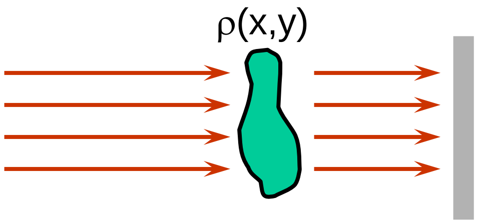


- *Old & new: emerging x-ray technologies in source & optics, advances in all 3 areas: fundamental, functional, anatomical*
- *Phase-contrast imaging: weak-absorbing features, less dose, far more clarity than traditional radiograph*
- *X-ray microscopy: could have high impact on cell biology, similar to x-ray crystallography ⇔ molecular biology*
- *Coherent diffraction imaging: new frontier on noncrystalline structures, structural molecular biology w/o need for crystals*



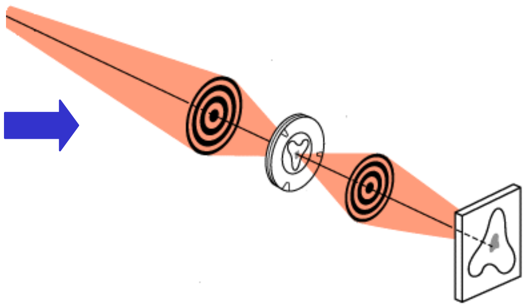
Miao et al. Nature *(1999): soft x-ray diffraction reconstruction to 75 nm*

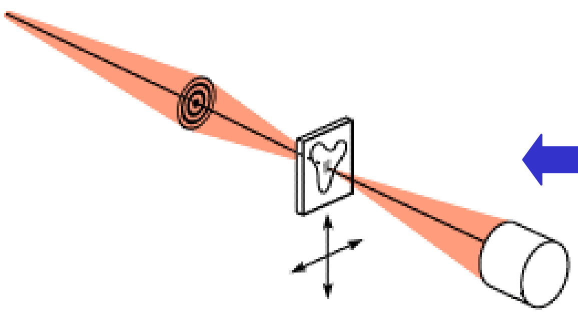
X-ray Microscopy & Imaging Basics



Direct imaging (radiography)

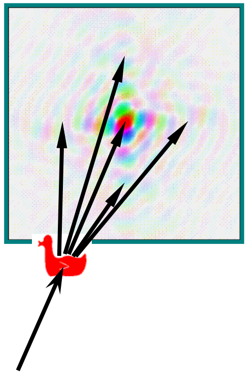
Transmission microscope (TXM)

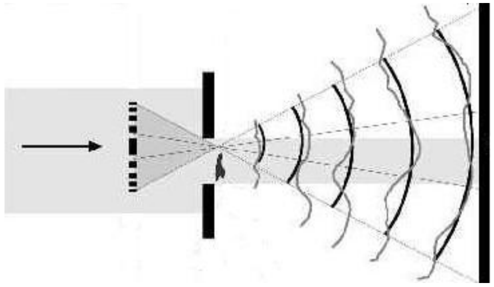




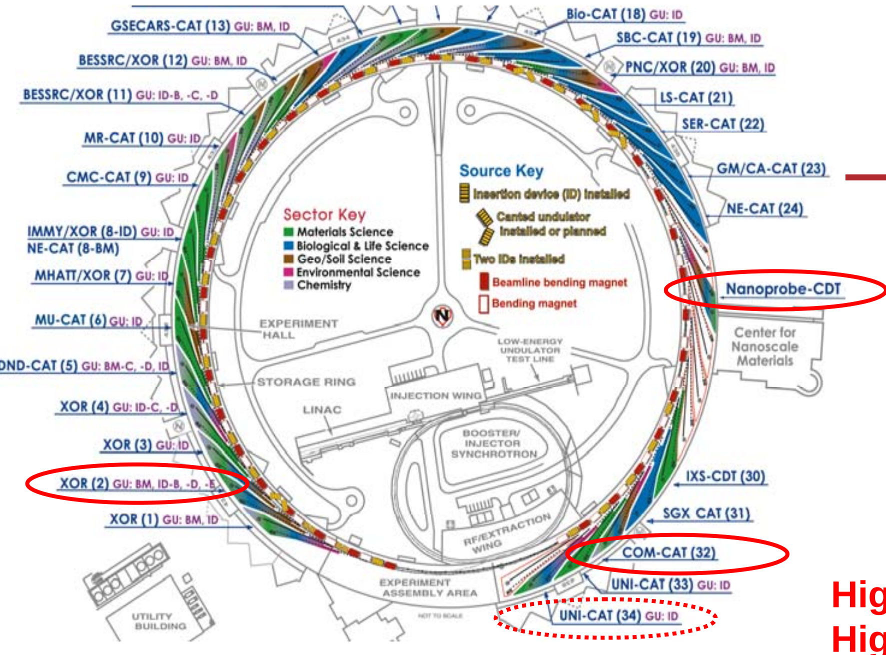
Scanning microscope (SXM)

Far-field diffraction





Holography



XMI Group at APS

X-ray Microscopy & Imaging

19 staff + 6 postdocs
3 sectors
6 beamline branches

High resolution
High sensitivity

Time-resolved
High throughput

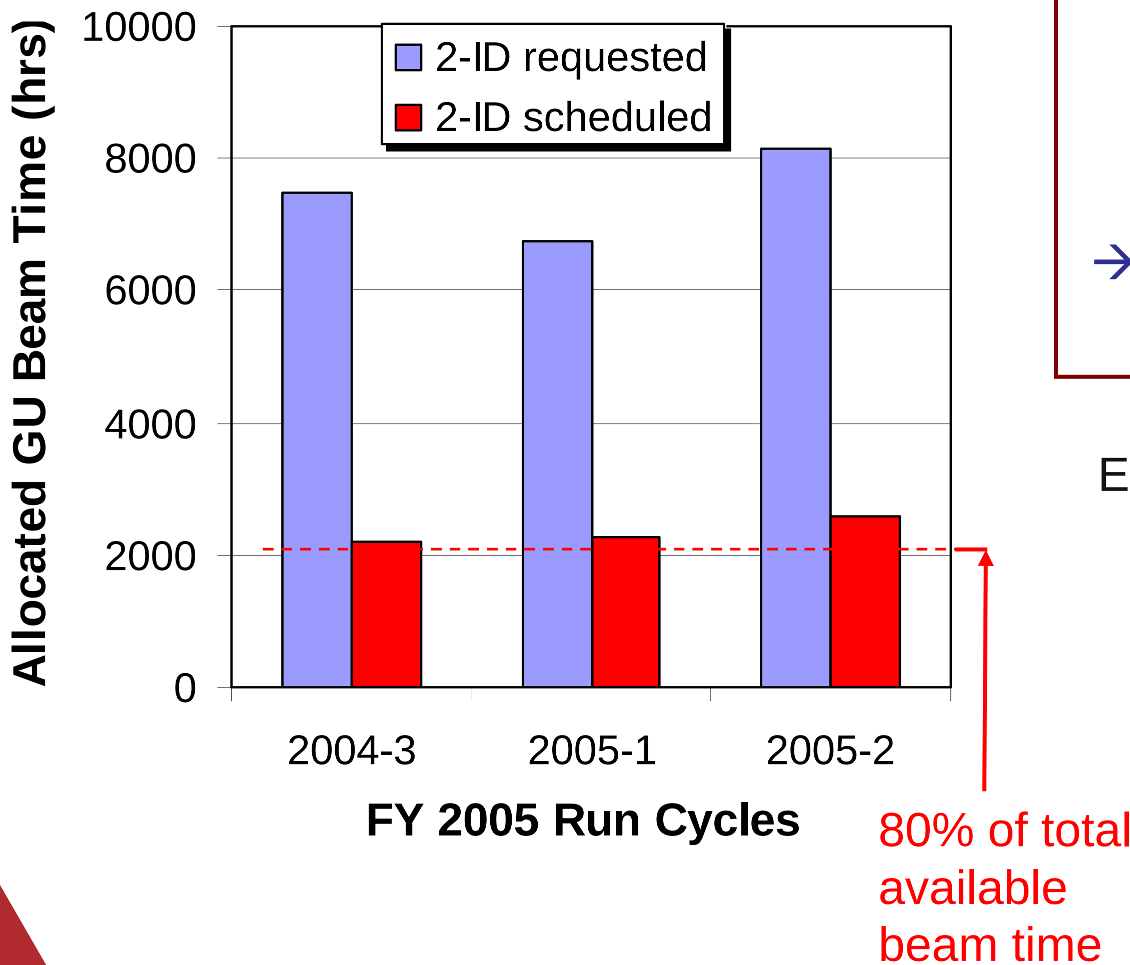
➔ **Foster R&D applications**

- Coherent Diffraction Imaging
- Scanning X-ray Microscopy
- Full-field X-ray Imaging

Support	34-ID-C	2-ID-B	2-ID-D	2-ID-E	26-ID	2-BM	32-ID
C. Roehrig		*I. McNulty*	*B. Lai*	*S. Vogt*	*J. Maser**	*F. DeCarlo*	*W-K. Lee*
J. Arko		*C. Rau*	*Z. Cai*	*D. Legnini*	*R. Winarski**	*Y. Chu*	*K. Fezzaa*
E. Wrobel	*(with former Uni-CAT)*	*L. Fan*	*Postdoc*	*Student*	*M. Holt**	*Y. Zhong*	*J. Illavsky*
B. Meurer		*M. deJonge*			*(with CNM)*	*X. Xiao*	*J. Socha*

General User Beam Time Usage at 2-ID

Oversubscribed by ~3x at 2-ID

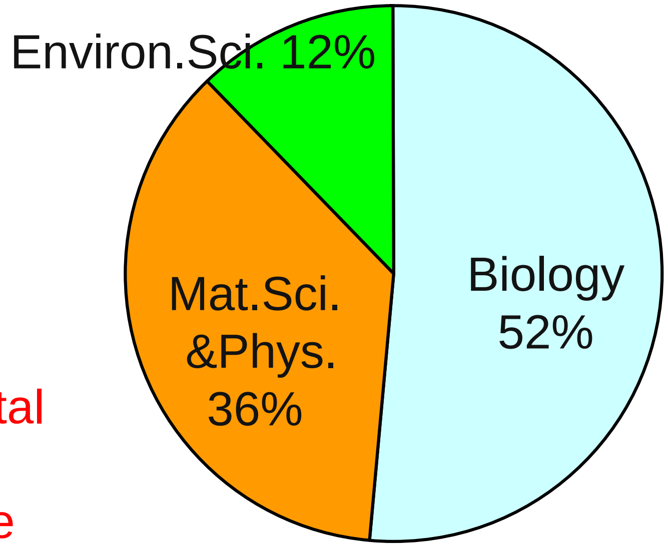


X-ray Imaging Applications

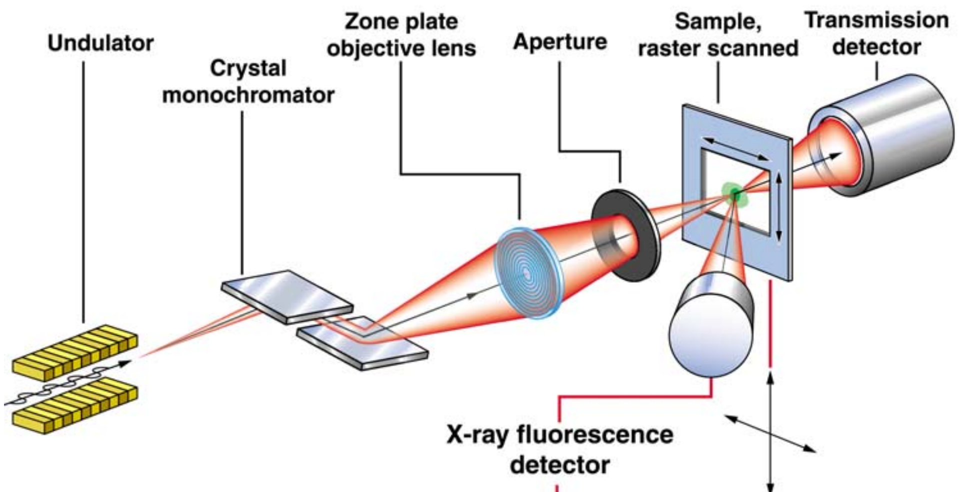
→ Studies of real-world functional materials

→ Inhomogeneous or heterogeneous

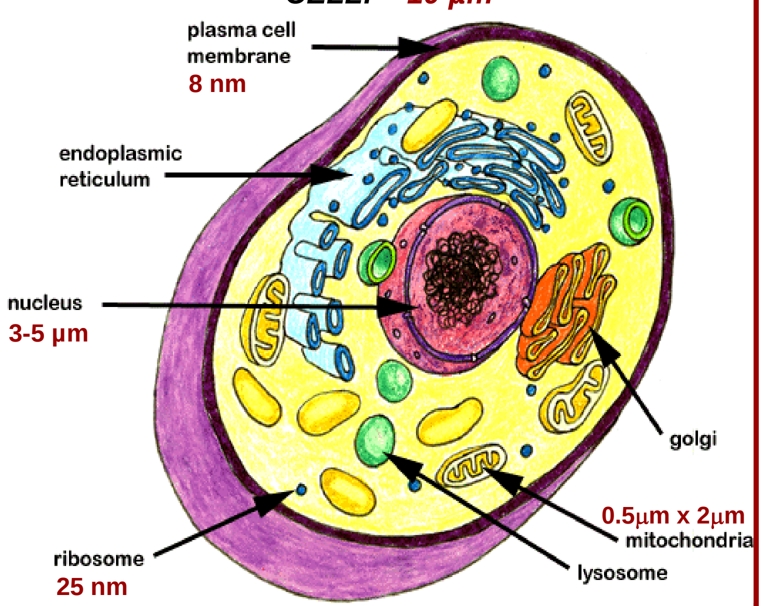
→ No identical copies of specimen => need sampling statistics



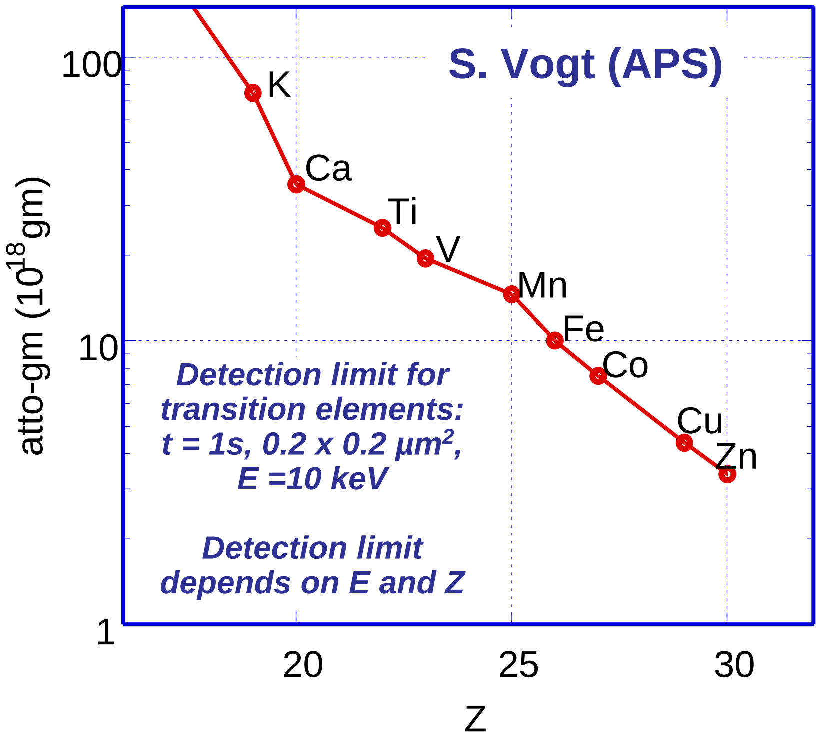
μ-XRF Studies of Trace Metals in Biological Cells



CELL: ~ 10 μm



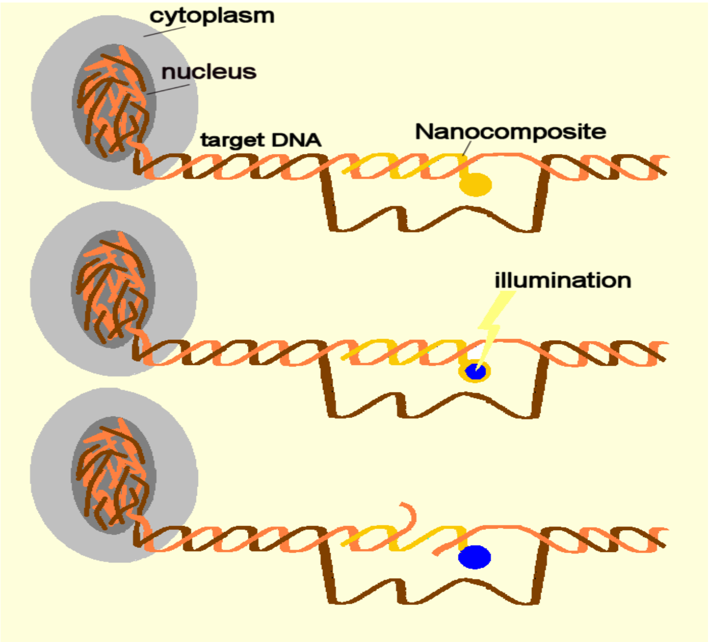
typical size of a protein: a few nanometer



- simultaneously map 15+ elements
- no dyes necessary
- very high sensitivity (<ppm)
- quantitative
- large penetration depth (> 100 μm)
- chemical state mapping & μ-XANES

TiO_2-DNA Nanocomposites as Intracellular Probes

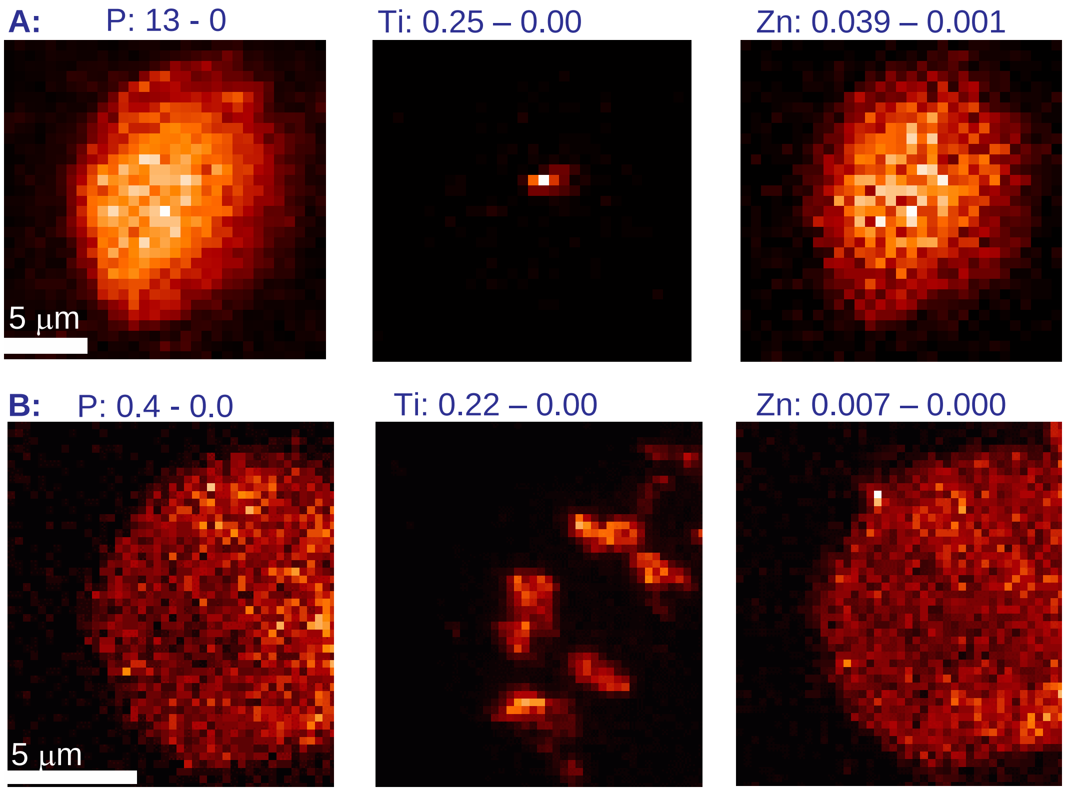
- Cell is transfected with TiO_2-DNA nanocomposites (4.3 nm Ø)
- DNA is used to target nanocomposite to specific chromosomal region
- TiO_2 allows photocleavage of targeted DNA strand upon illumination => potential to be used to for gene therapy



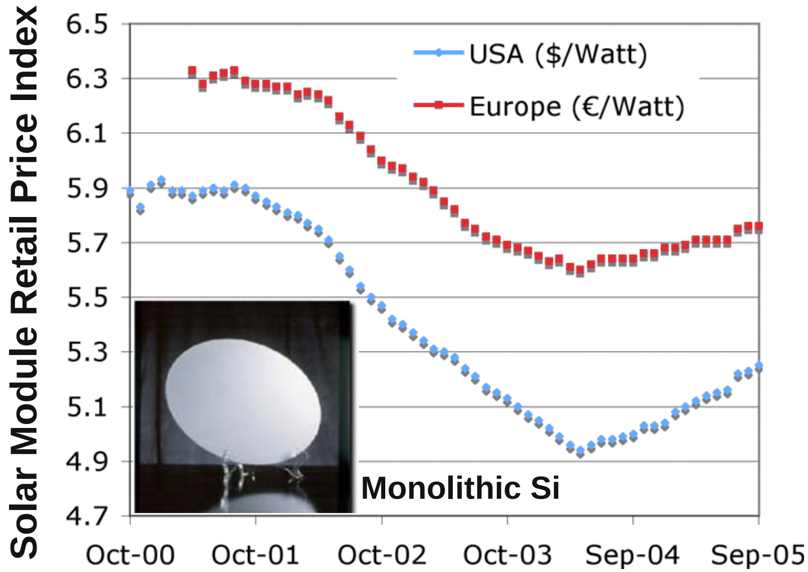
Paunesku, Vogt, *et al.*, Nature Materials 2, 343-346 (2003)

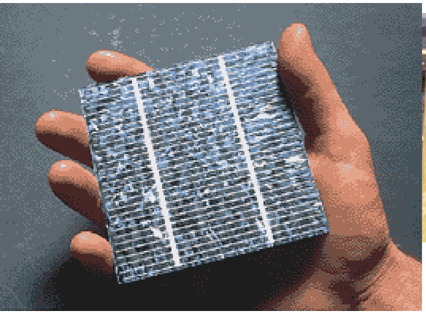
- **Map Ti distribution using X-ray fluorescence, to quantify success rate of TiO_2-DNA transfection, and visualize target**
- **A: nanocomposites targeted to nucleolus**
- **B: nanocomposites targeted to mitochondria**

Units: µg/cm^2



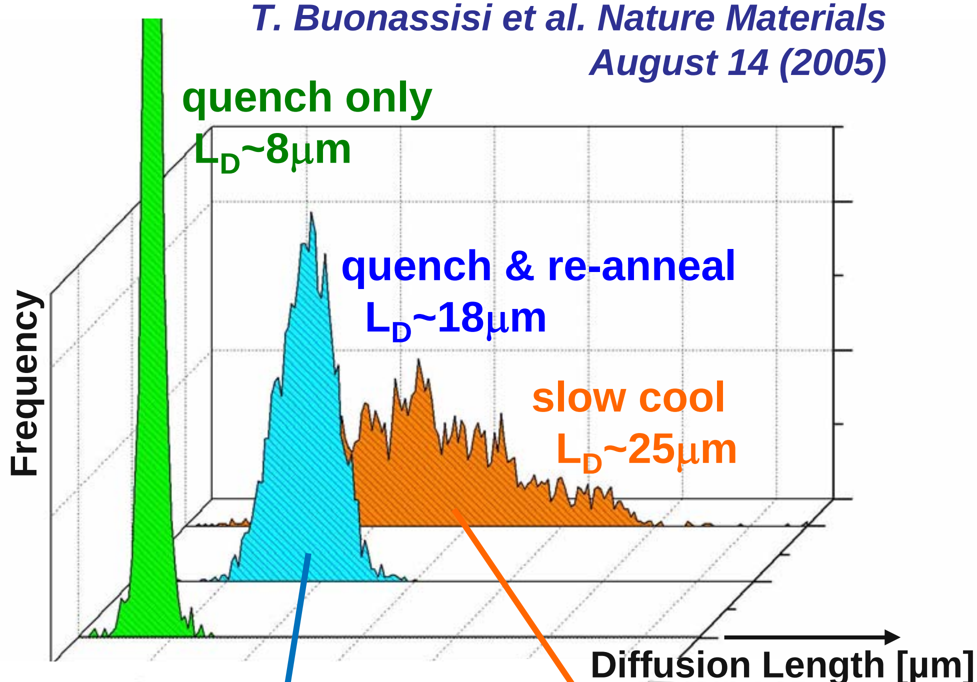
Defect Engineering for Less-Costly Solar Cells

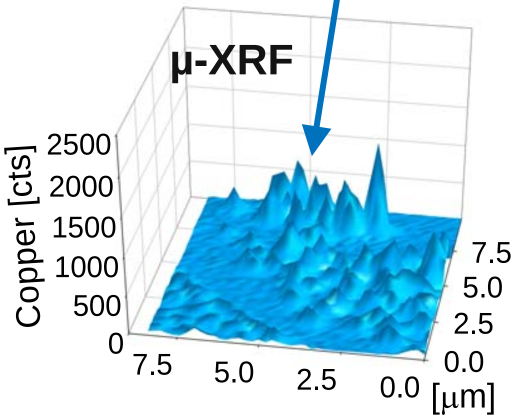


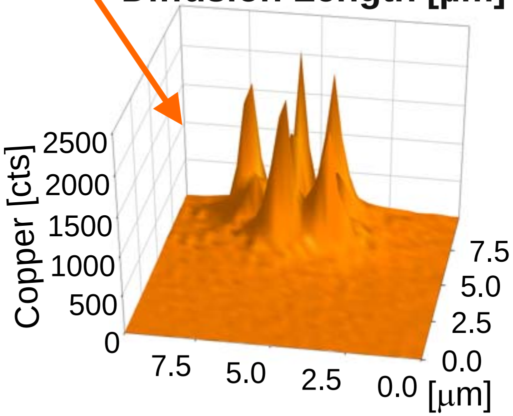


Multicrystalline mc-Si

T. Buonassisi et al. Nature Materials August 14 (2005)







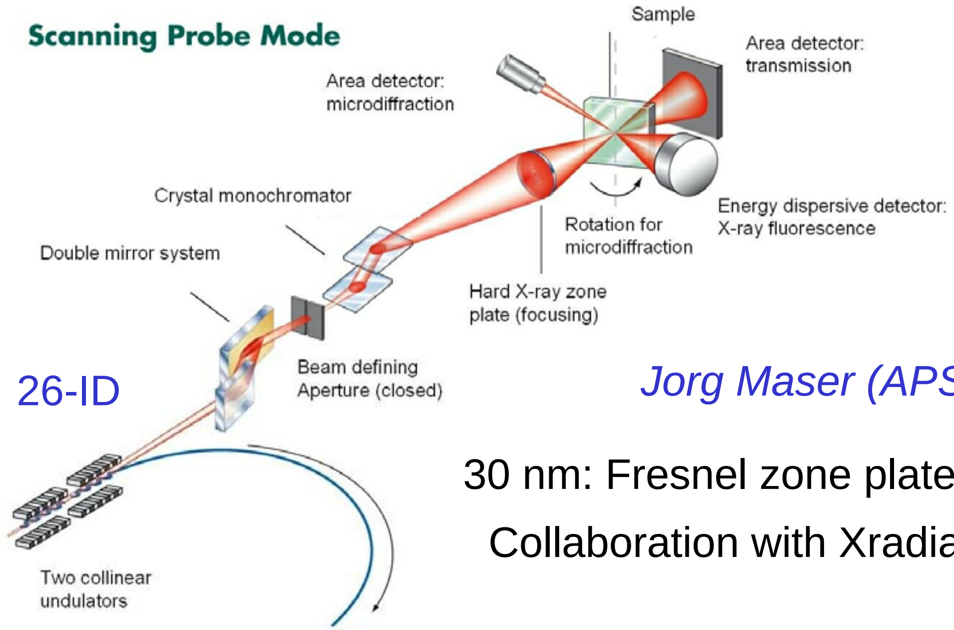
- **Metal impurities in mc-Si**

➔ Device performance?

➔ Defect engineering?

New & Proposed Nanoprobes at APS

Nanoprobe: joint effort CNM and APS



26-ID

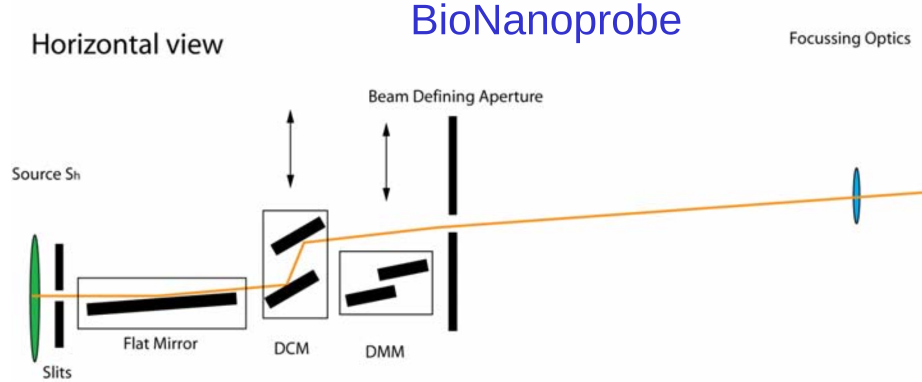
Jorg Maser (APS)

30 nm: Fresnel zone plate

Collaboration with Xradia

Bio-nanoprobe: new ID, 30 nm
Woloschak (Northwestern)
Vogt (APS)

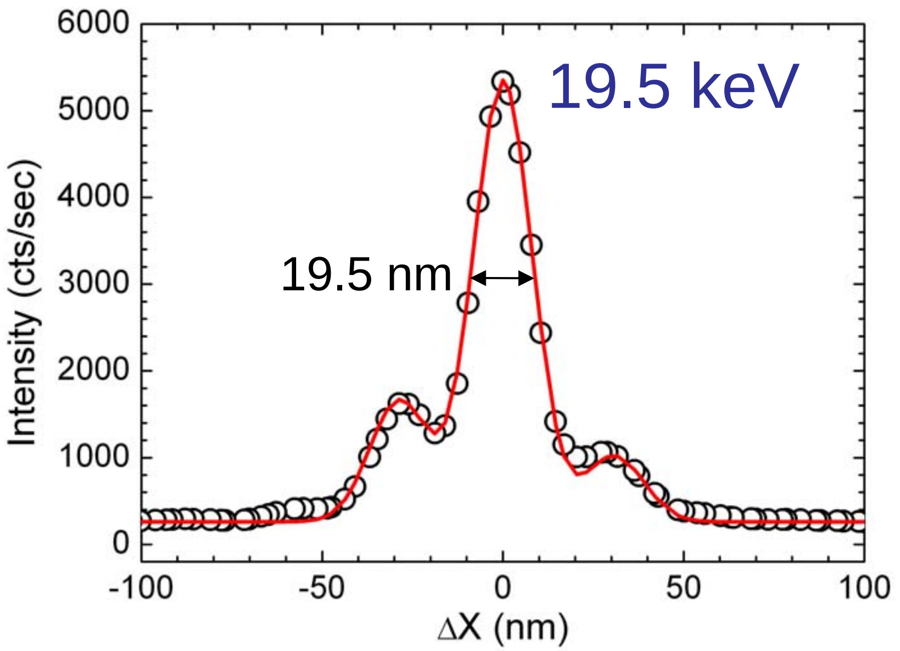
BioNanoprobe

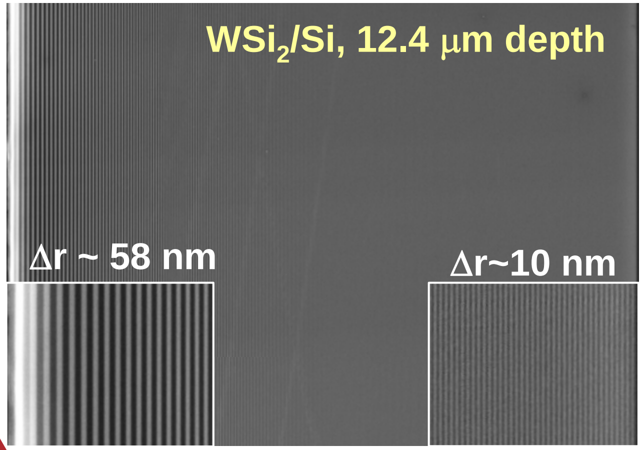


High throughput: BM, medium resolution
Vogt (APS)
biological tissue arrays
XRF from whole cells

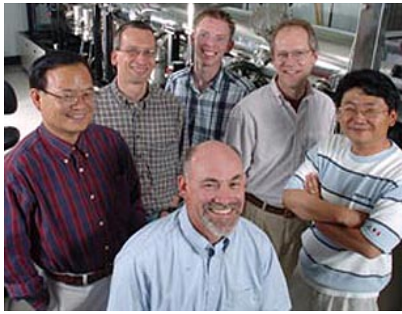
Advanced Focusing Optics: Pathway for 5 nm Focusing of Hard X-rays

Multilayer Laue Lens



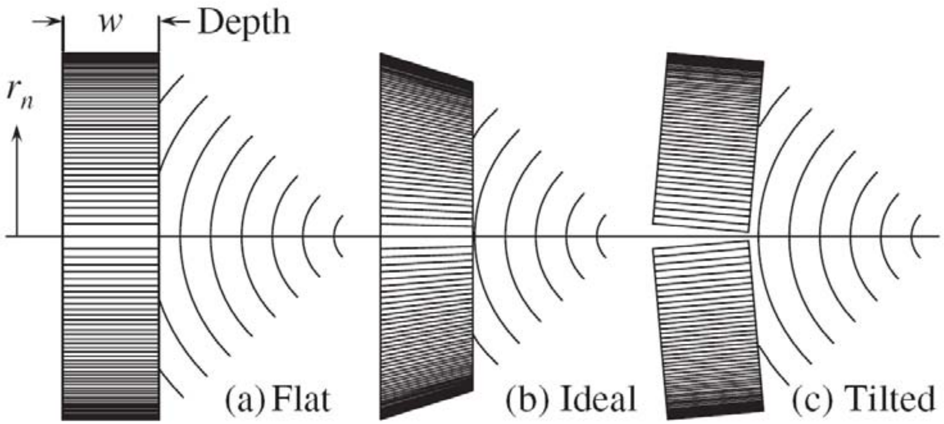


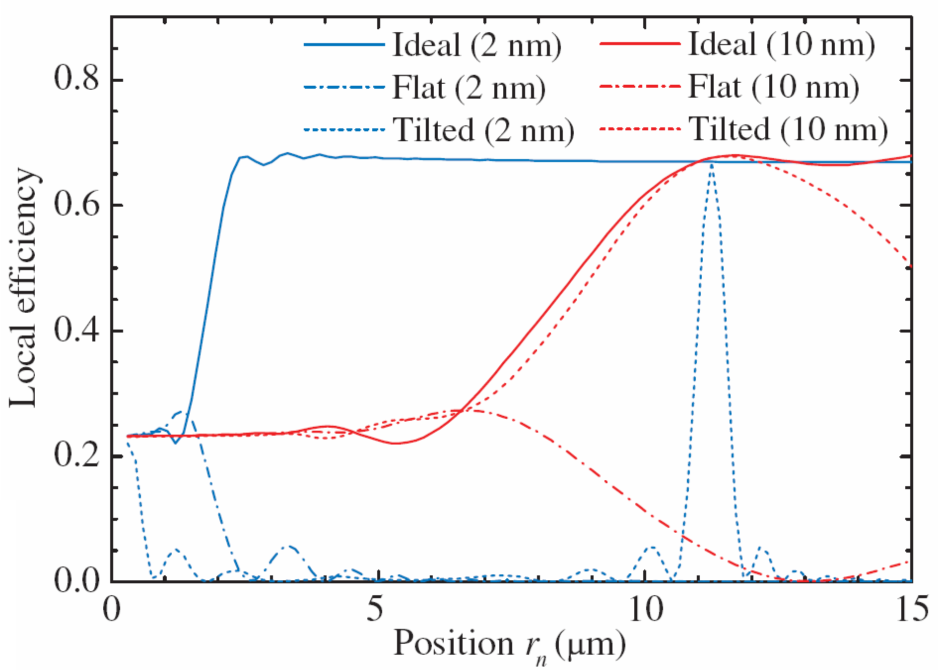
H.C. Kang, J. Maser, G.B. Stephenson, C. Liu, R. Conley, A.T. Macrander, S. Vogt
Phys. Rev. Lett. 96, 127401 (2006)



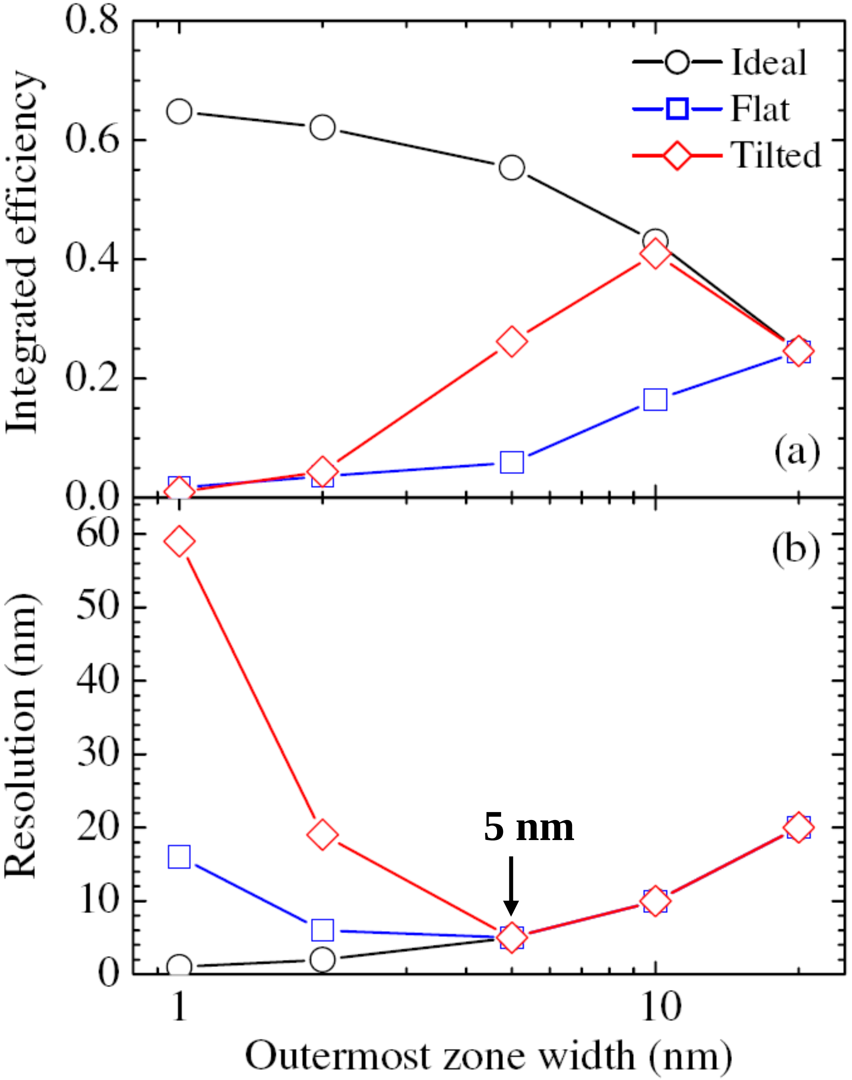
➔ Opens up new capabilities in SXM !

Towards nm Focusing of Hard X-rays





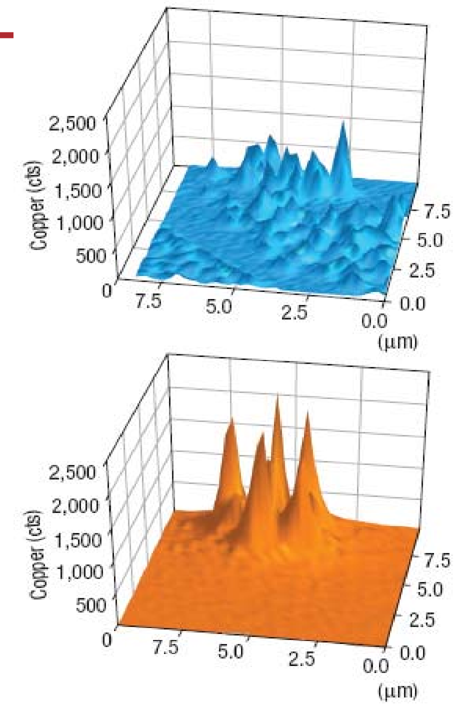
Kang et al, *PRL 96, 127401 (2006)*

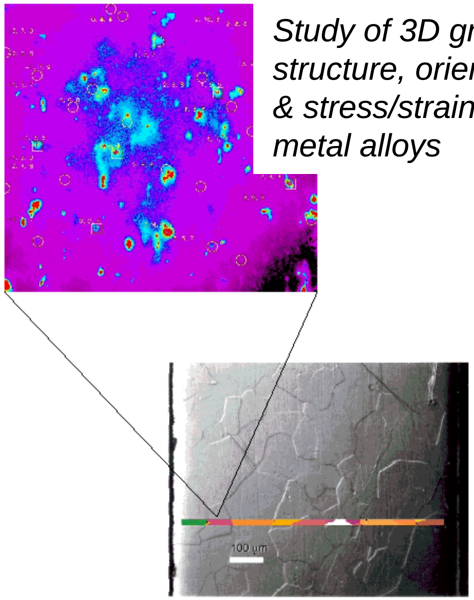


ERL: Imaging Functional Units in Materials Science & Biology at nm-scale

- ERL would allow the most efficient usage of nm-focusing x-ray optics such as MLL, and advance state-of-the-art scanning x-ray microscopy to < 5 nm spatial resolution, with working distance
- 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 organelle level, and develop new approaches to diagnose and treat diseases
- For materials science, it will offer a non-destructive penetrating probe for impurity/defects, grain boundaries, and nano-domain engineering of functional electronic and engineered materials such as solar cells and metal alloys

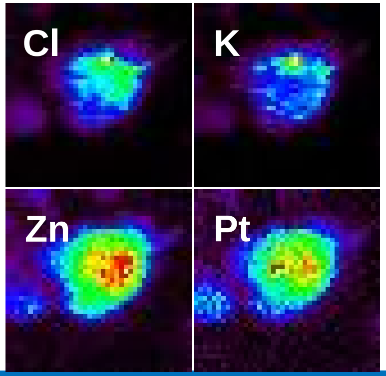
Metal impurities in solar cells



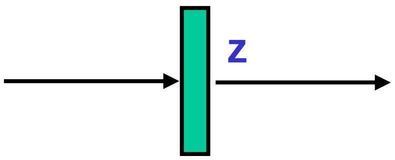


Study of 3D grain structure, orientation & stress/strain in metal alloys

Elemental distribution in cisplatin-treated cancer cell



Phase Contrast vs. Absorption Contrast Imaging

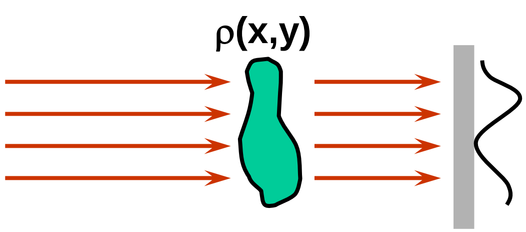


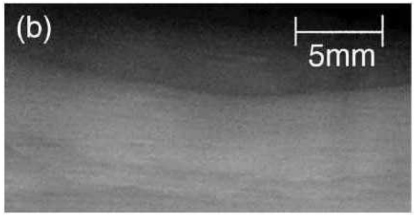
Refraction index: $n = 1 - \delta - i\beta$

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

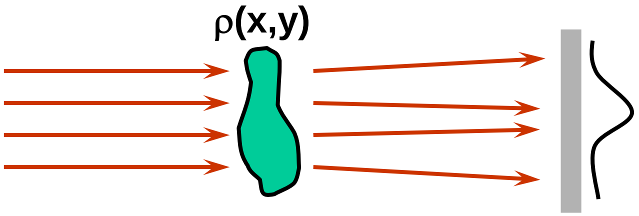
$$I(z) \sim |E(z)|^2 \sim I_0\, e^{-4\pi\beta z/\lambda}$$

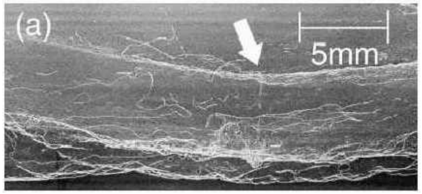
⇒ **Absorption contrast:**
$\mu z = 4\pi\beta z/\lambda \sim \lambda^3$





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





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

Imaging Biomechanics and Animal Physiology

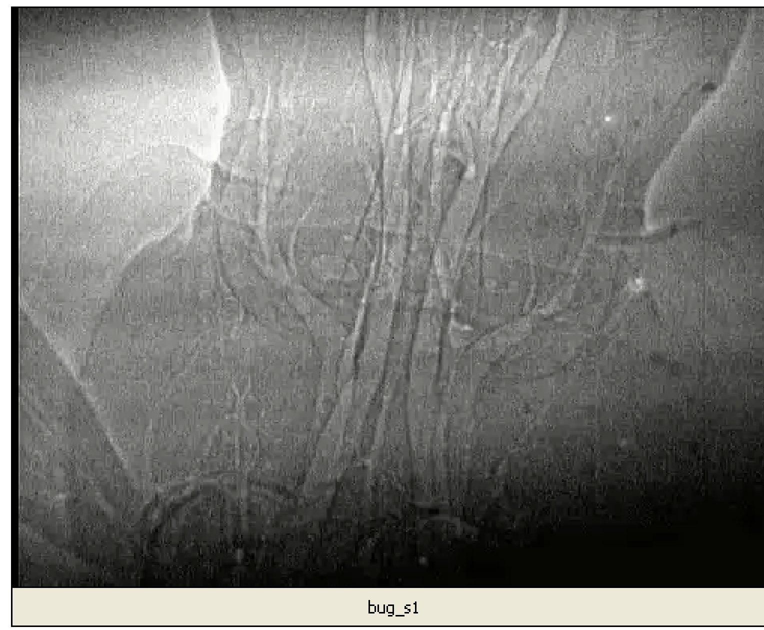
Tracheal Respiration in Insects Visualized with Synchrotron X-ray Imaging

Mark W. Westneat,*[1] Oliver Betz,[1,2] Richard W. Blob,[1,3]
Kamel Fezzaa,[4] W. James Cooper,[1,5] Wah-Keat Lee[4]
Field museum of Chicago & APS, Argonne National Lab.

wood beetle

Science (2003) 299, 598-599.

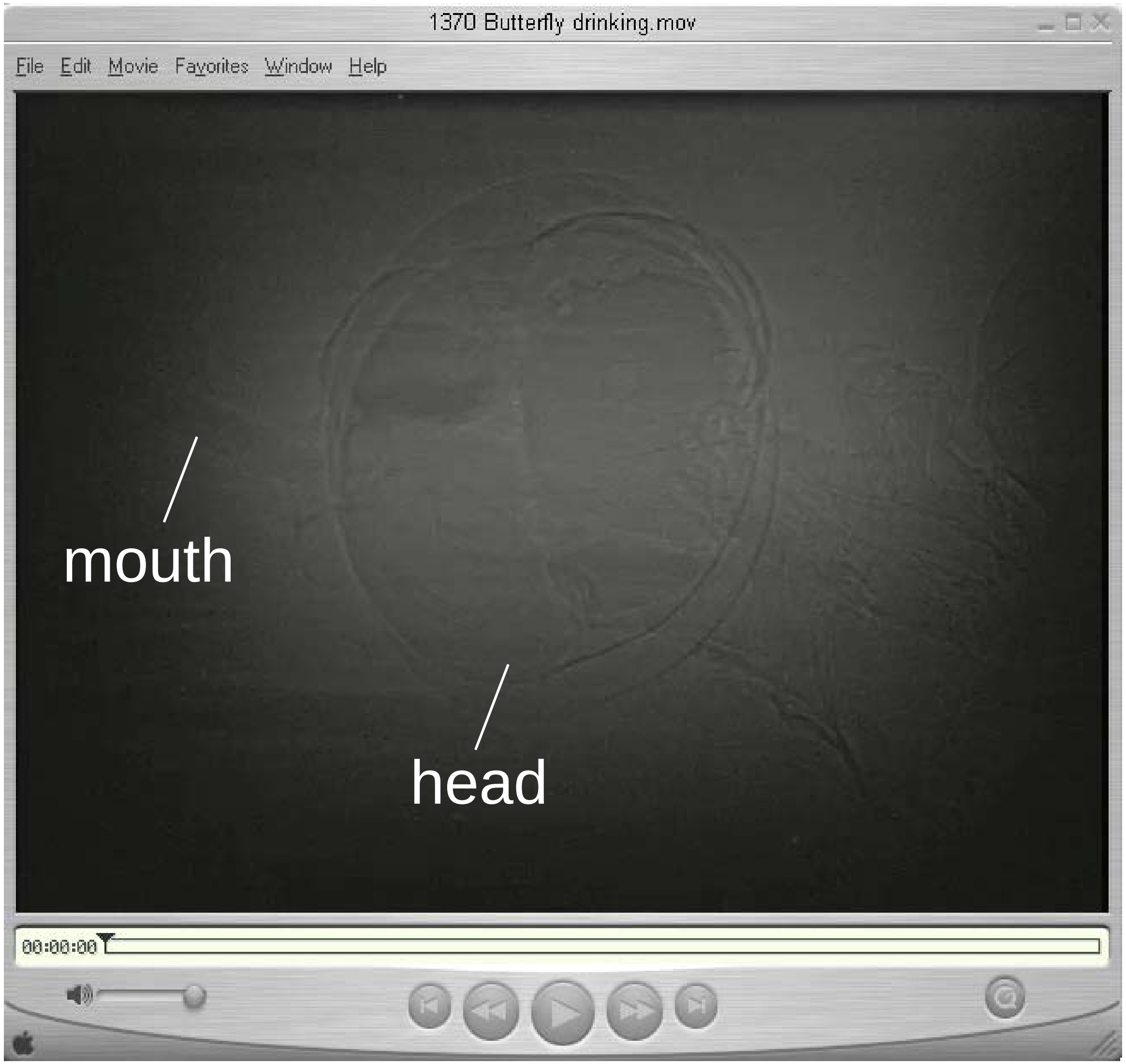
- **Animal functions**
- **Biomechanics**
- **Internal movements**
- **New findings not known before**



Comparative Biology:
understanding of evolutionary transitions underlying diversity of life

Imaging Biomechanics and Animal Physiology

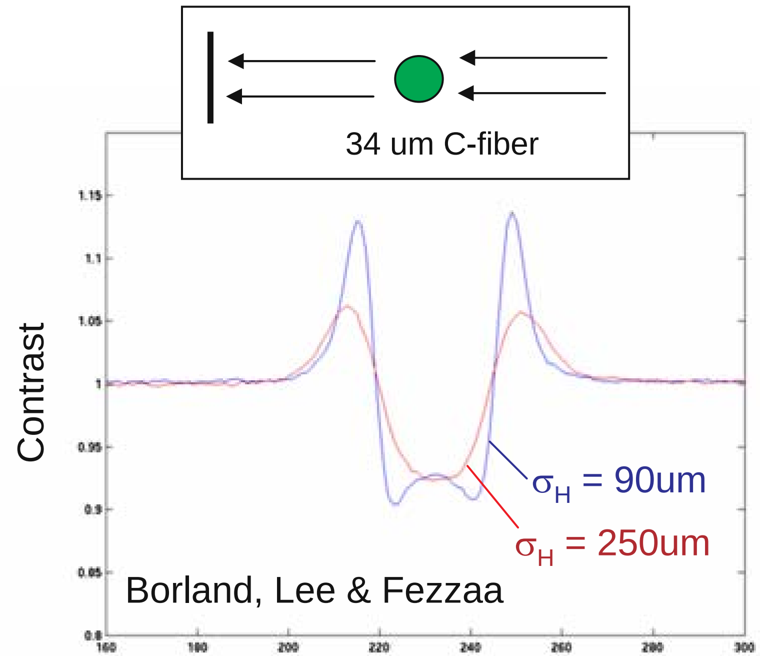
Butterfly drinking an iodine-laced honey solution.
Field of view is about 2mm x 3mm

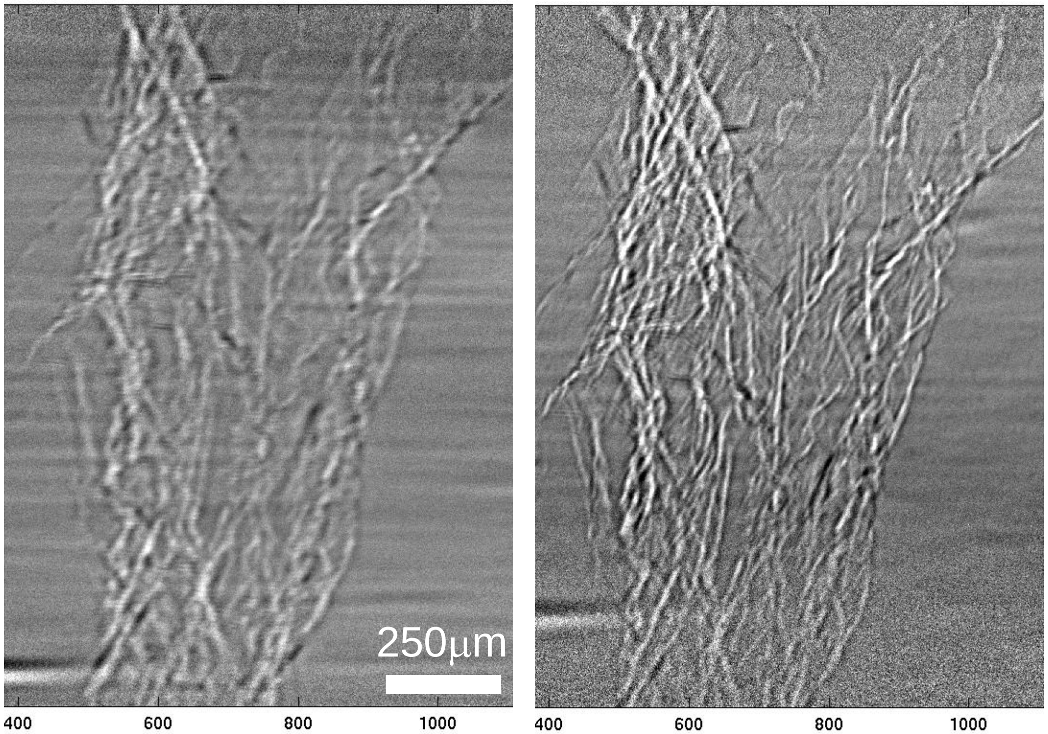


Socha, Lee, et al. (2006) unpublished

Phase-Contrast Imaging with ERL

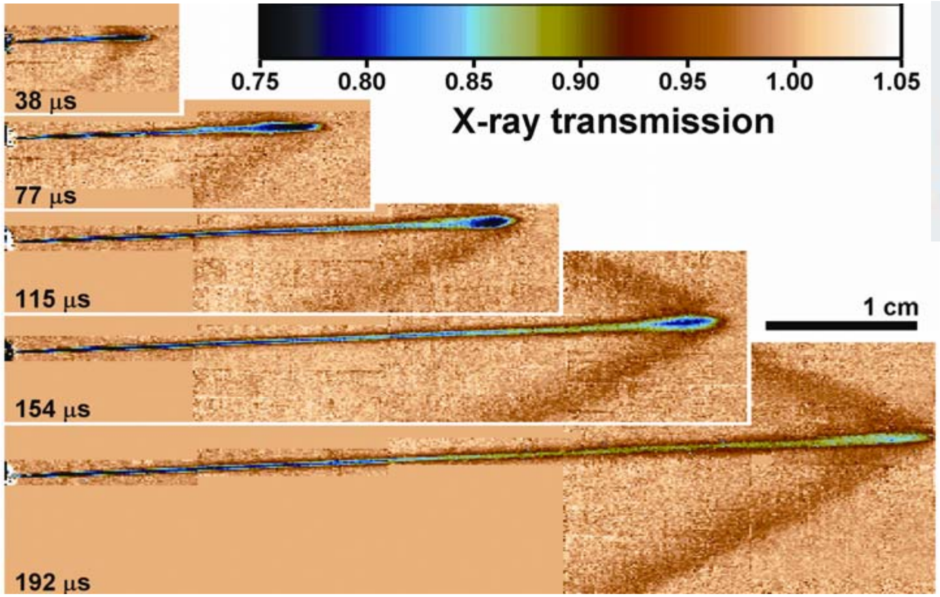
- **Small round source size of ERL would greatly enhance observable phase contrasts for weak density differences**





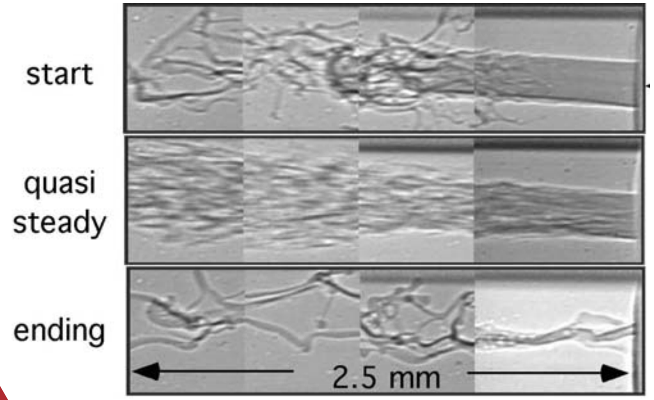
← Stress cracks in Aluminum
t = 3 mm, 30 keV, D = 1m

Ultrafast Imaging of Fuel Spray in Gasoline Engines

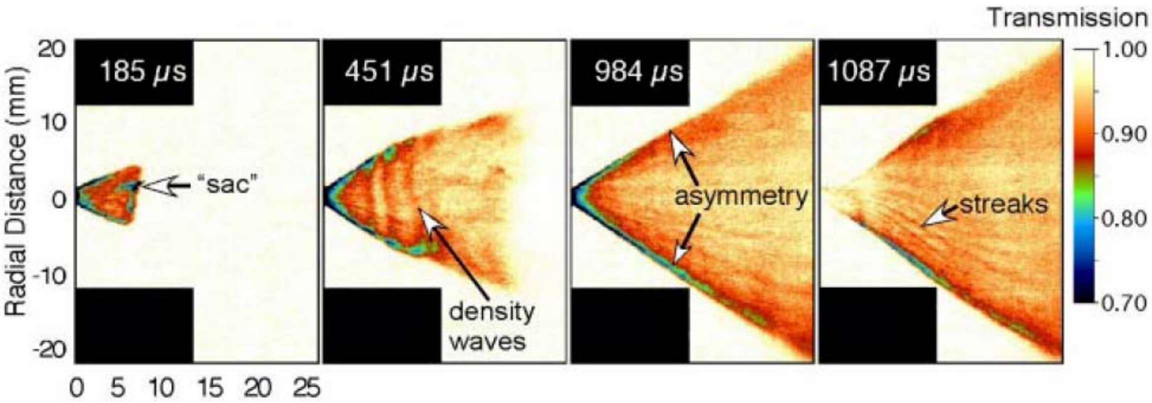


AG MacPhee, MW Tate, CF Powell, et al., Science, 295, 1261 (2002).

- ✓ Supersonic liquid jet can generate shock waves
- ✓ X-radiographs yield characteristics of the shock waves
- ✓ The shock waves can be quantitatively simulated

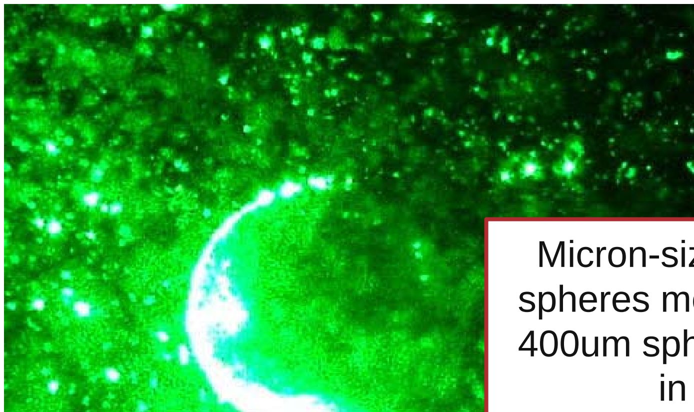


X-ray flash imaging at 300ns !!



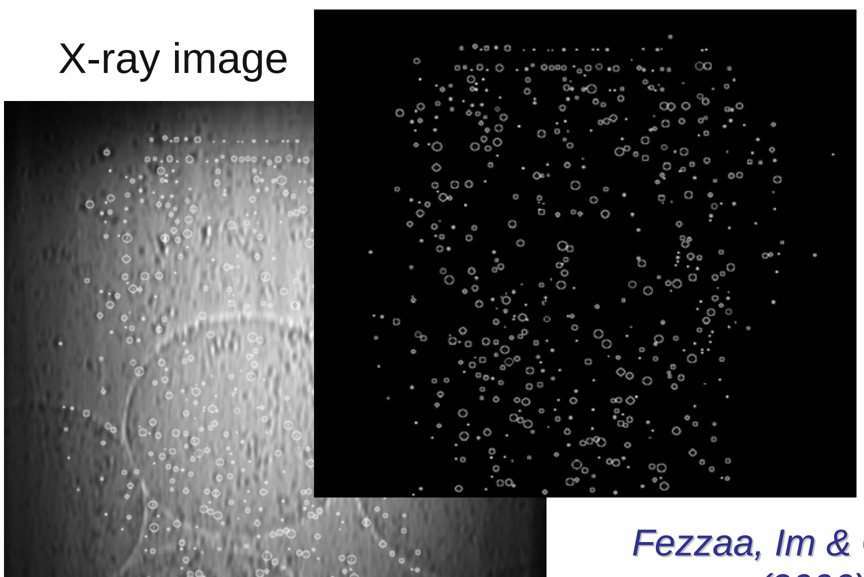
Particle Imaging Velocimetry (PIV)

Visible light image

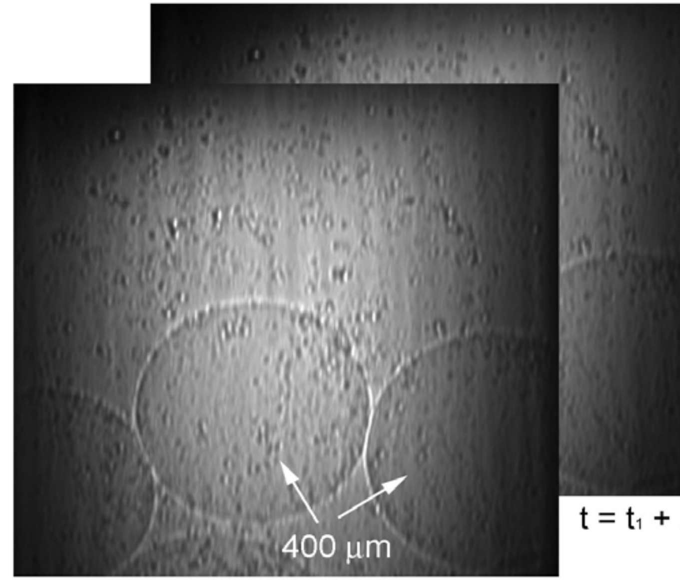


Micron-sized polystyrene spheres moving around big 400um spheres (obstacles) in glycerin

X-ray image



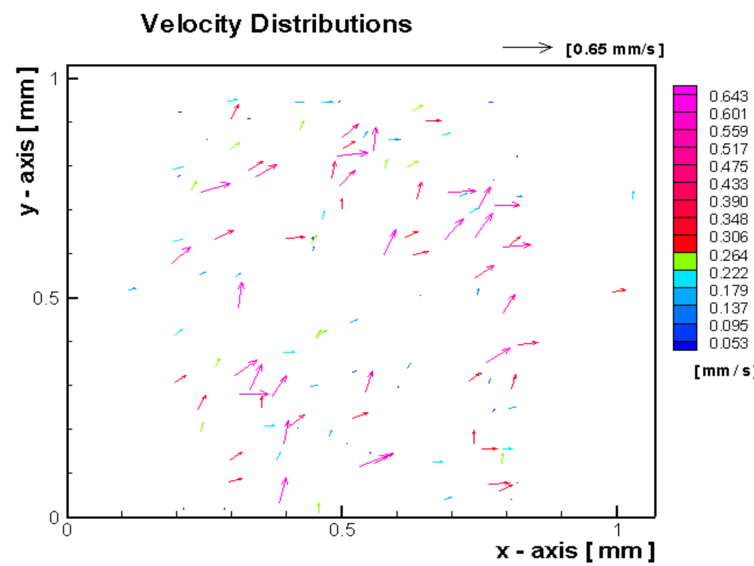
Fezzaa, Im & Cheong (2006)



$t = t_1 + \Delta t$

$t = t_1$

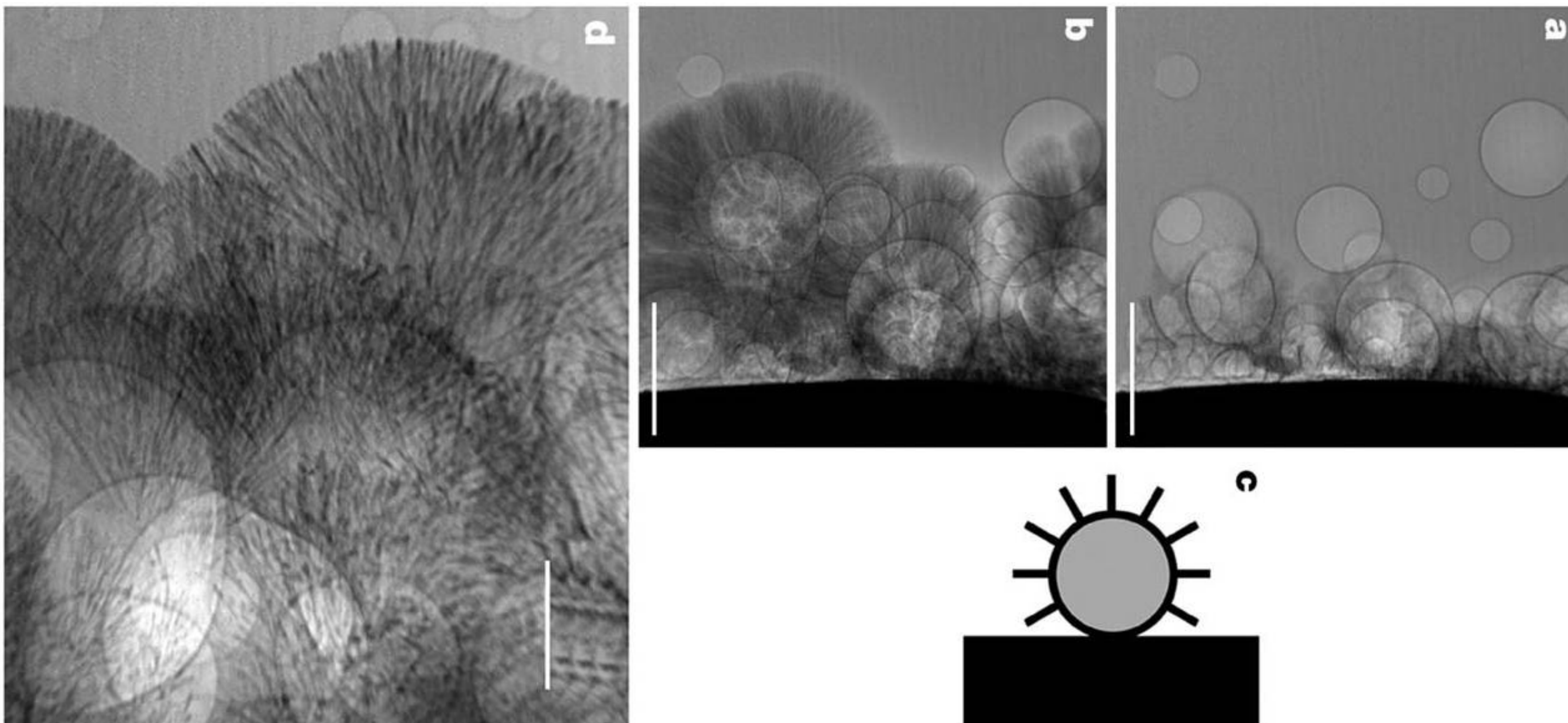
Two consecutive frames in x-ray phase contrast mode, of the polystyrene spheres in motion.



In-situ X-ray Imaging of Electrodeposition

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

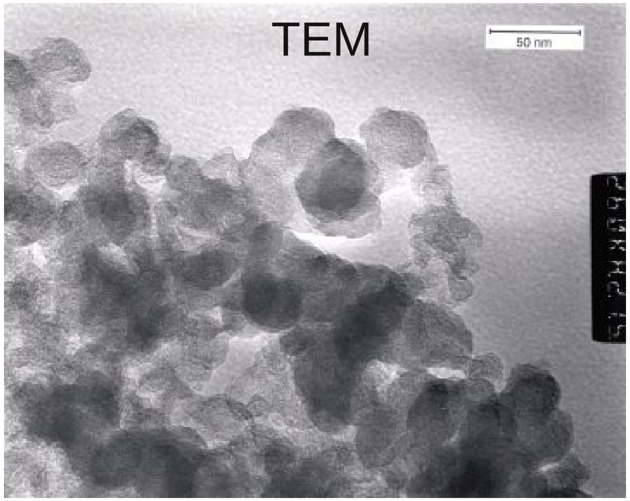
In the electrodeposition of metals, a widely used industrial technique, bubbles of gas generated near the cathode can adversely affect the quality of the metal coating. Phase-contrast imaging is used to witness directly and in real time the accumulation of zinc on hydrogen bubbles.

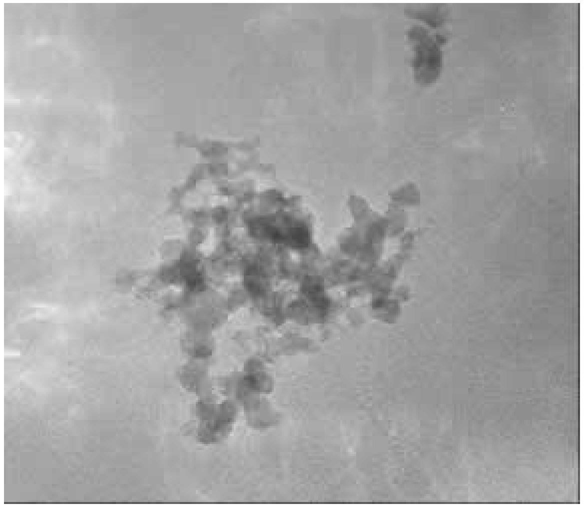


Ultrafast Imaging with ERL

- ERL would allow ultrafast 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

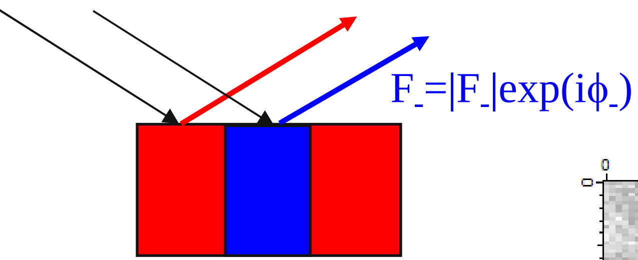
- ERL would allow direct real-time imaging of low-contrast materials processing and depositions, such as formations of carbon particulates in engines, polymer aggregates and polymer thin-film coatings





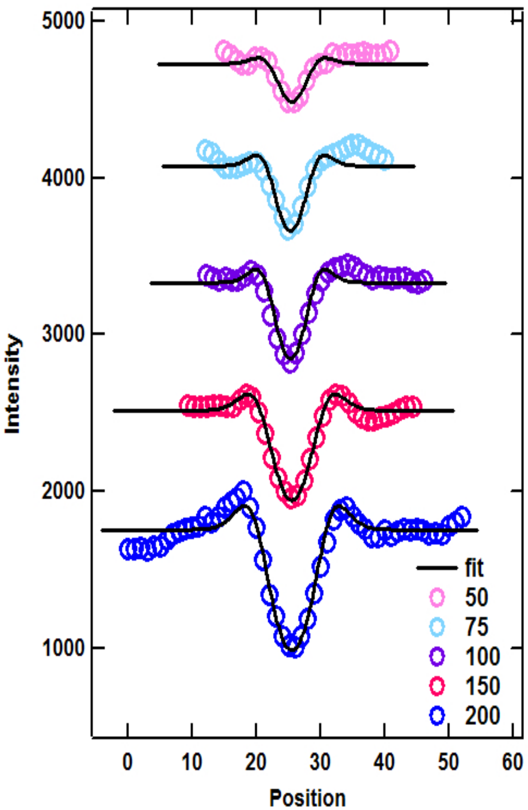
Phase-contrast Imaging in X-ray Topography

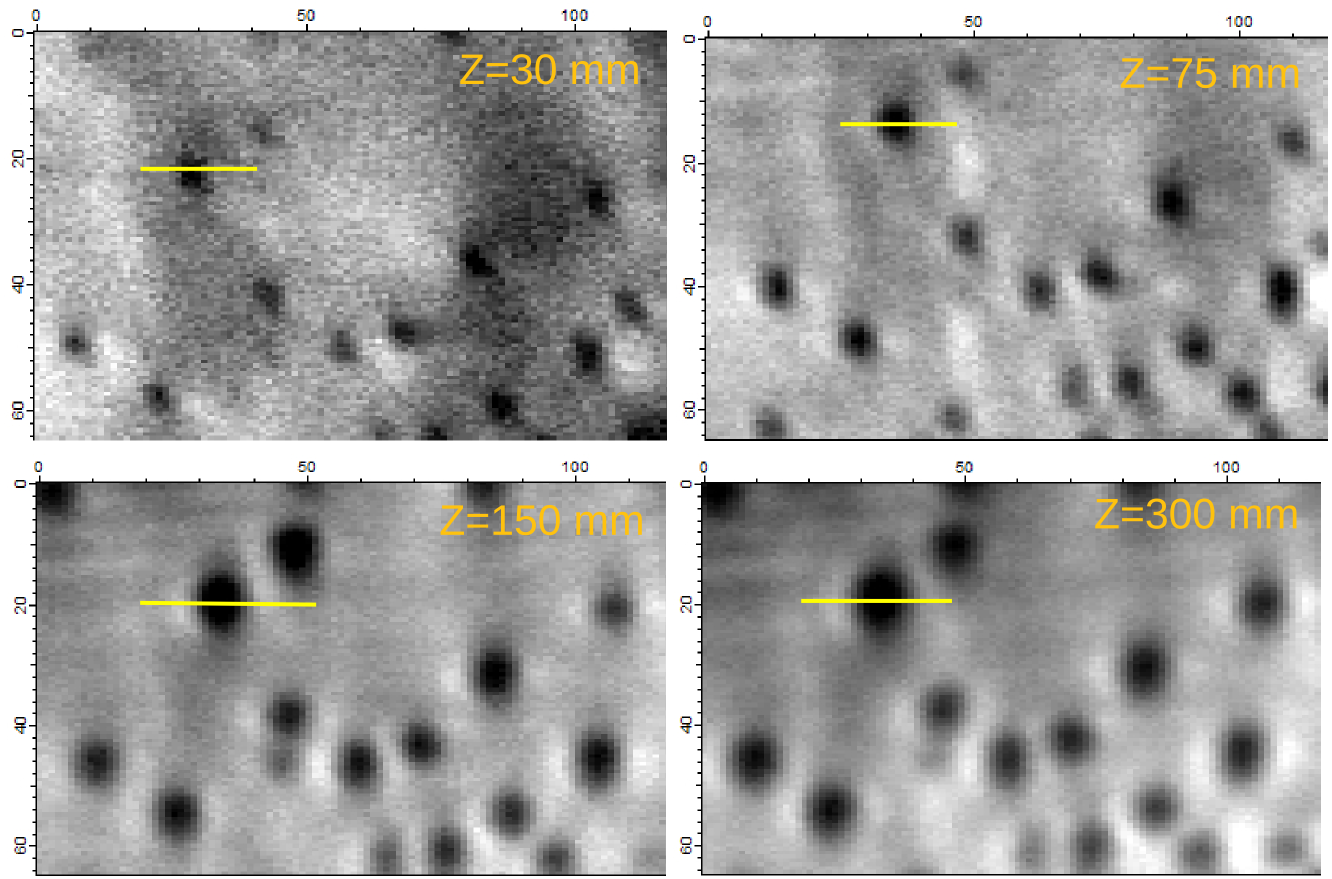
Chu & Zhong (APS)



ferroelectric domains

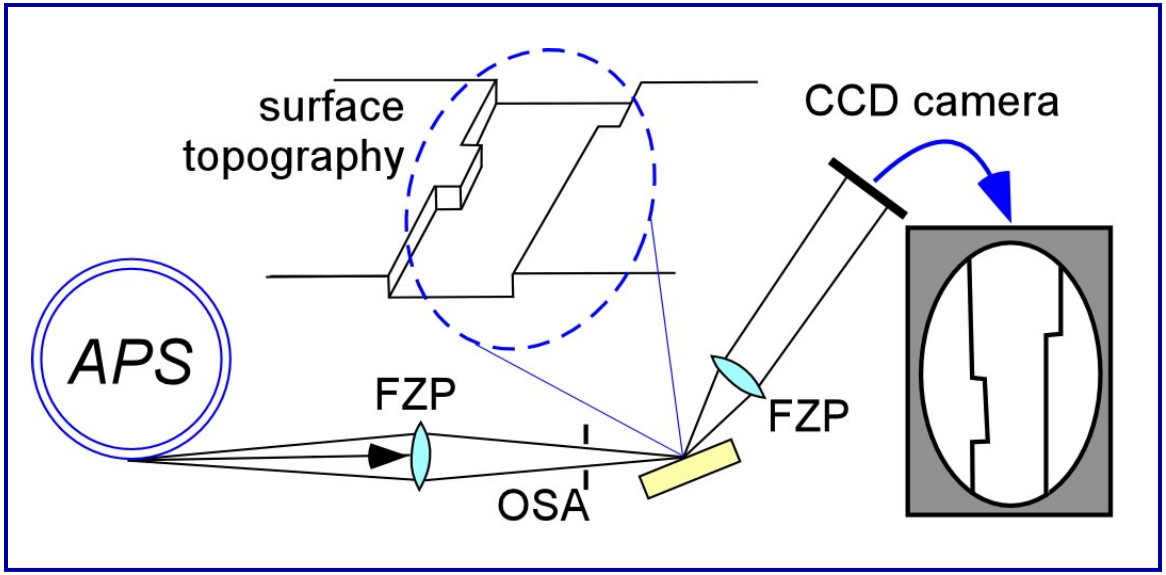
- Fresnel wave propagation → enhanced phase-contrasts
- Physical domain size can be deduced by analysis



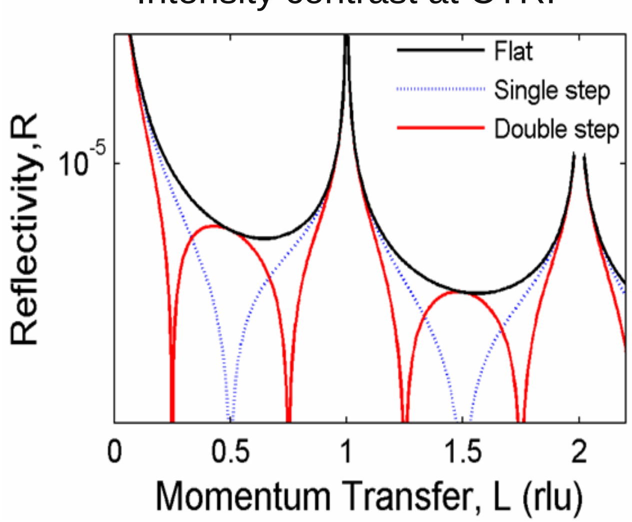


Phase-Contrast X-ray Diffraction Microscopy

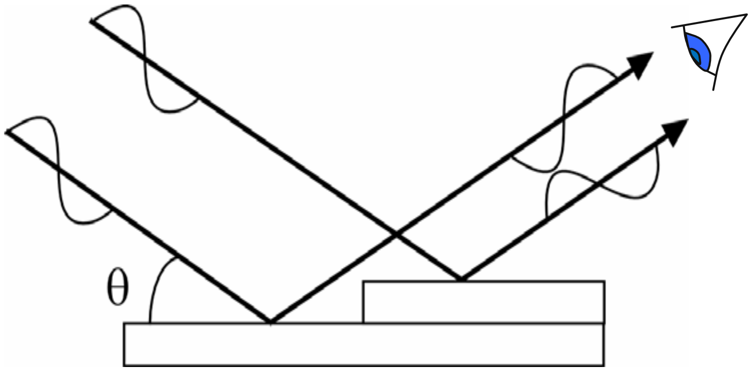
*P. Fenter, C. Park, Z. Zhang, and S. Wang, in review (2006)



Intensity contrast at CTR:



Phase contrast mechanism:



X-ray Reflection Interface Microscopy

Characteristics:

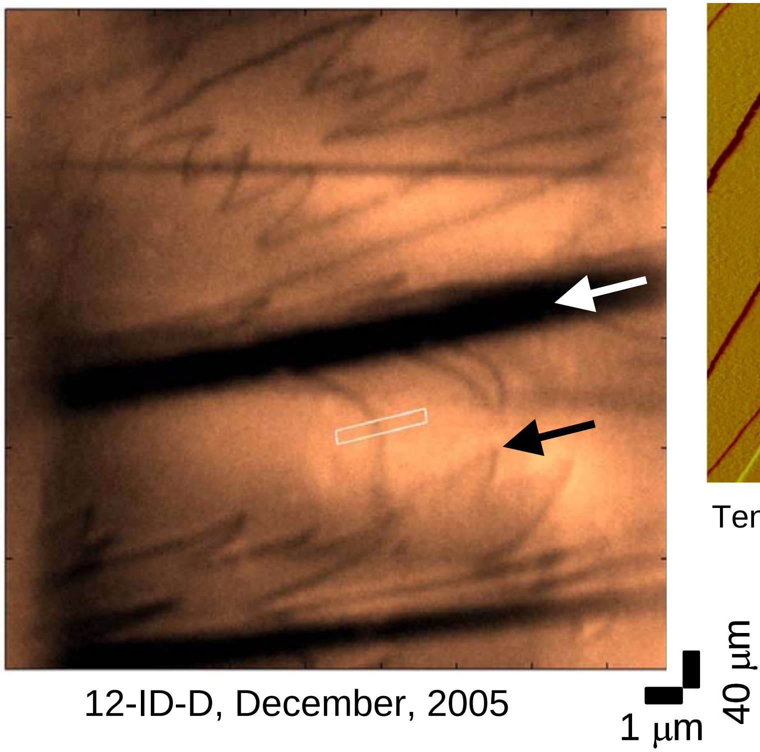
→ Strong contrast at defects (~100%), but weak reflected beam intensity ($R < 10^{-5}$)

→ Sub-nm vertical sensitivity, but modest lateral resolution (~100 nm, set by FZP),

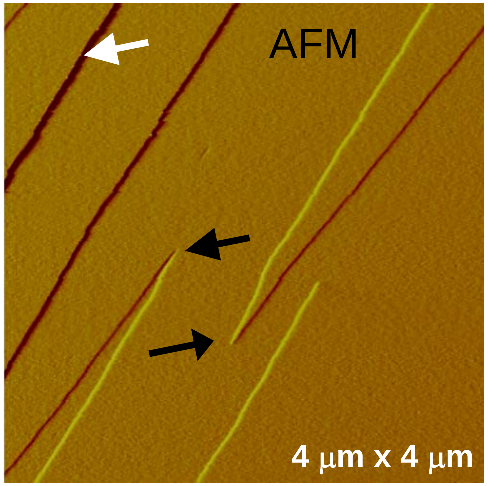
Observation of Surface Step Distributions with XRIM

P. Fenter, C. Park, Z. Zhang, and S. Wang, in review (2006)

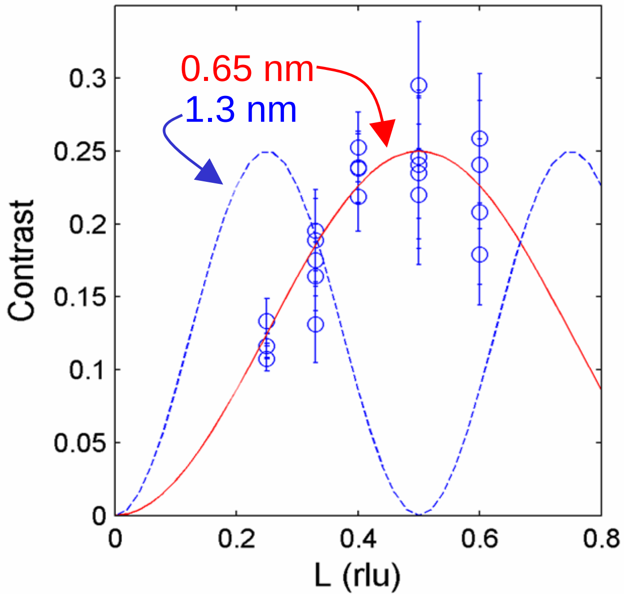
Step distributions on $KAlSi_3O_8$ (001)



12-ID-D, December, 2005



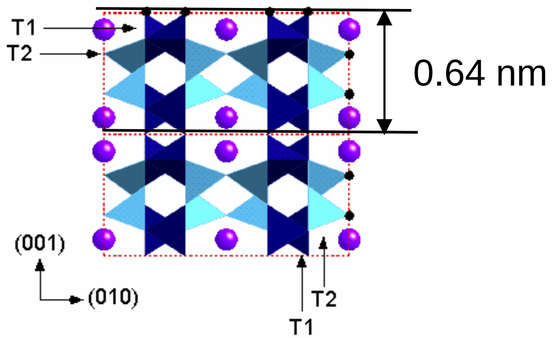
Teng et al., GCA **65,** 3459 (2001)



Imaging Conditions:

- $\theta = 1.4°$
- E = 10 keV
- L = 0.25 rlu (Q = 0.24 $Å^{-1}$)
- Sample held in air

Elementary step structure:



New Opportunities with XRIM

P. Fenter (ANL)

A new capability combining:

- exquisite structural sensitivity derived from interfacial X-ray scattering
- high spatial resolution derived from X-ray microscopy

A *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:

- geochemical reactions at solid-liquid interfaces
  - dissolution
  - heterogeneous growth
  - nucleation site distribution (terrace vs. step)
  - phase determination (e.g., calcite vs. aragonite for $CaCO_3$)
  - nano-particle hetero-epitaxy
- materials growth (MOCVD, MBE, oxides)
- corrosion and oxidation
- ferroelectric domain switching
- magnetic domain structures

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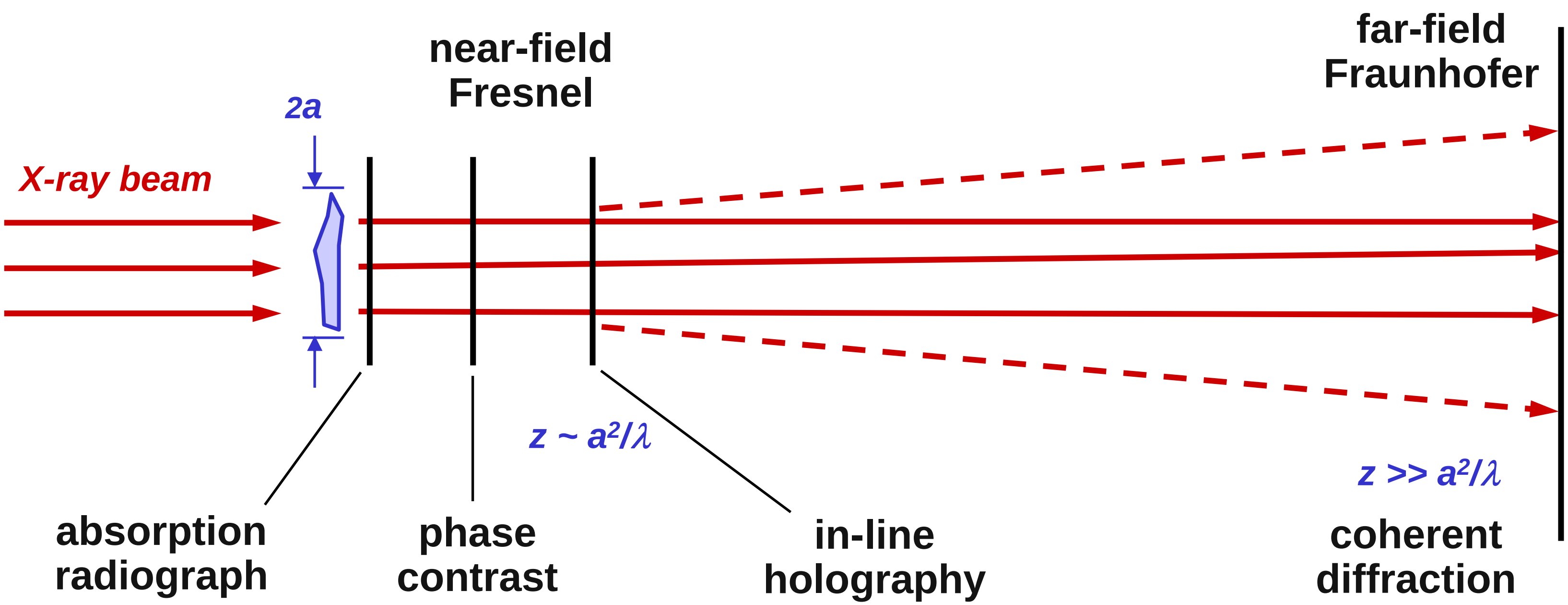
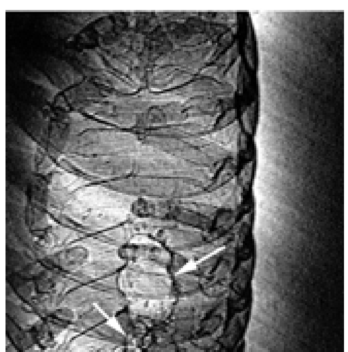
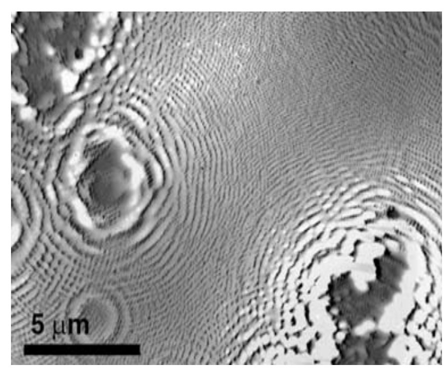
Different Regimes of X-ray Imaging

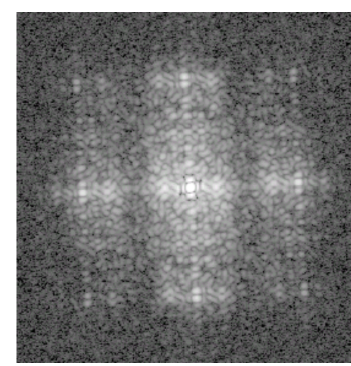




Kagoshima et al.
JJAP (1999).

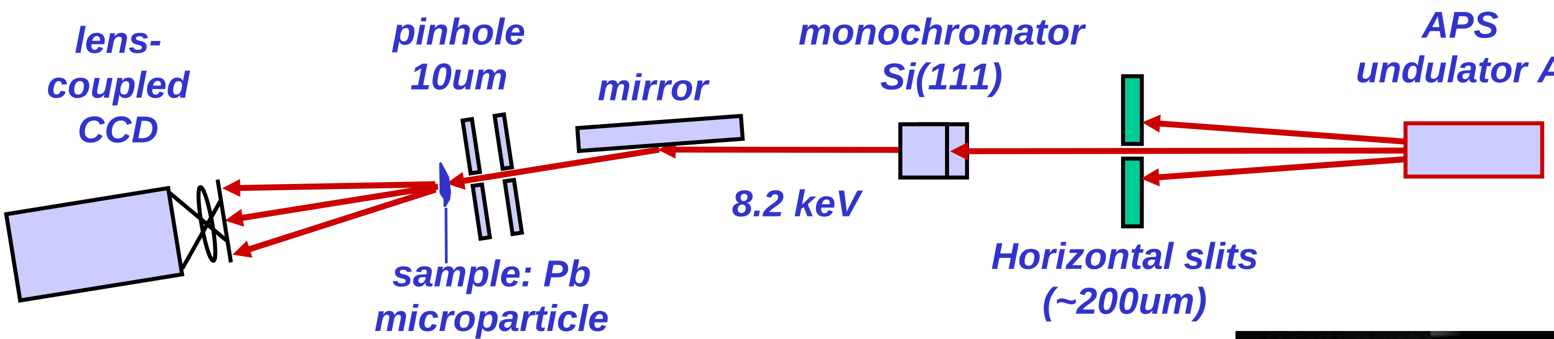


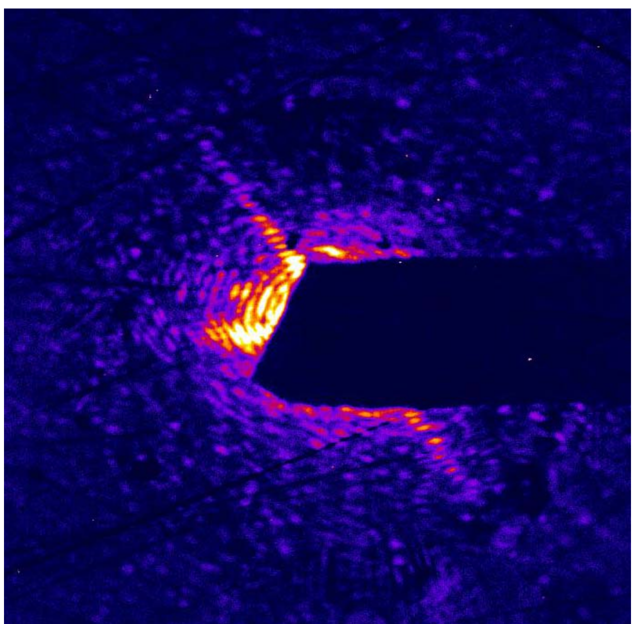
Jacobsen (2003).

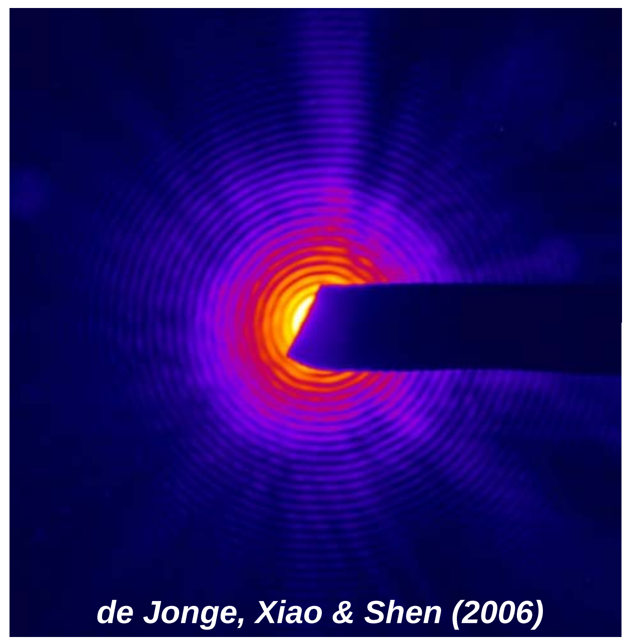


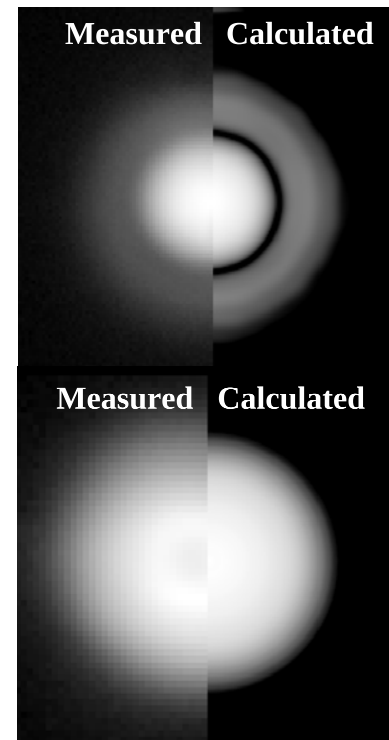
Miao et al.
Nature (1999).

Coherent Diffraction Imaging

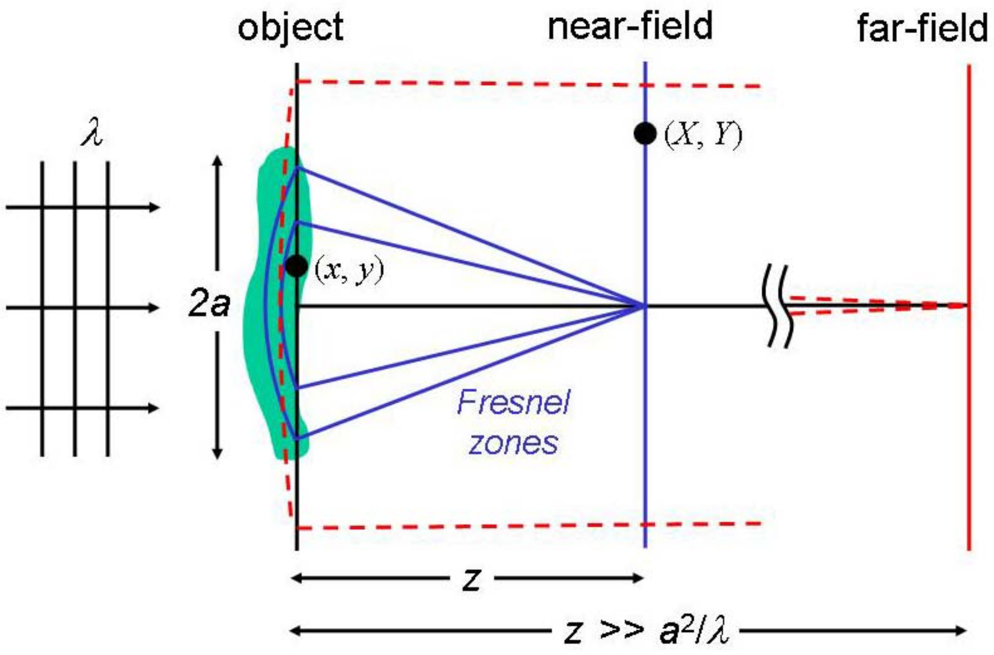






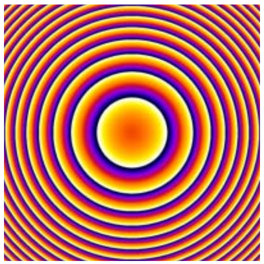


Distorted Object Approach for Wave Propagation & Phasing



Phase-chirped distorted object:

$$\bar{u}(x,y) \equiv u(x,y)e^{-\frac{i\pi}{\lambda z}(x^2+y^2)}$$



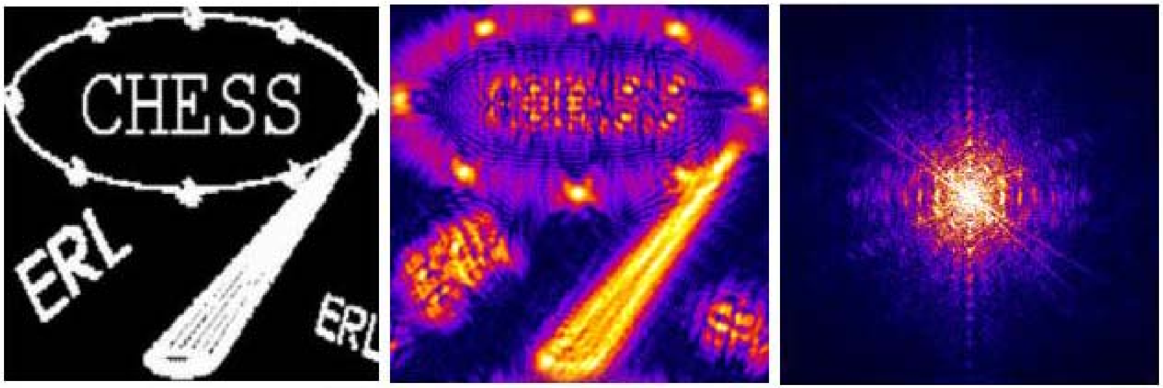
$$F(X,Y) = \frac{i\,e^{-ikR}}{\lambda R}\iint \bar{u}(x,y)\,e^{-\frac{ik}{z}(Xx+Yy)}dxdy$$

- Momentum transfer: $(Q_x, Q_y) = (kX/z, kY/z)$
- Number of Fresnel zones: $N_z = a^2/(\lambda z)$

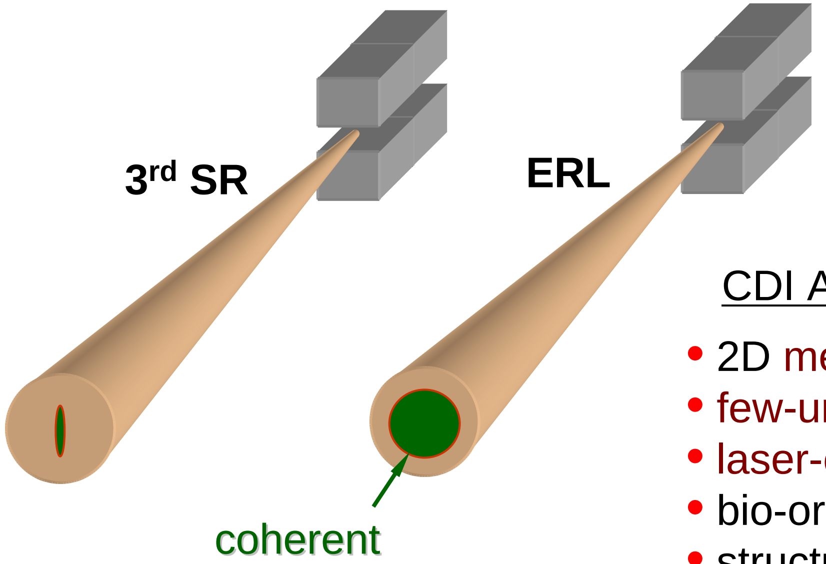
⇒ Unified wave propagation method by Fourier transform

⇒ Unified iterative phasing algorithm development

Xiao & Shen, PRB 72, 033103 (2005)

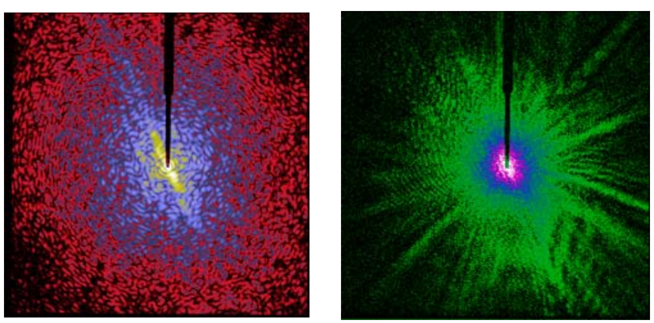


ERL: Ideal Source for Coherent Diffraction Imaging !

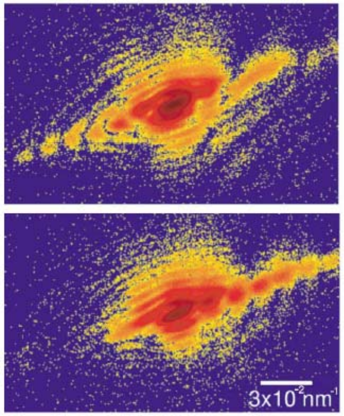


CDI Applications:

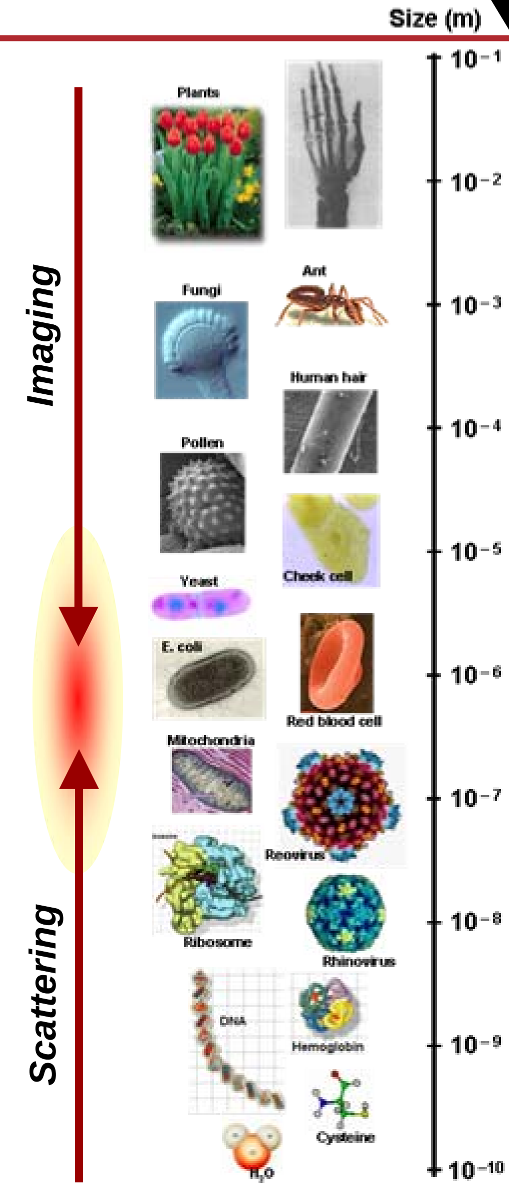
- 2D membrane protein crystals
- few-unit-cell crystals
- laser-oriented macromolecules
- bio-organic-inorganic hybrids
- structure & strain in nanoparticles
- biological cells



Miao – actin filaments, 2-ID-B



Robinson – Au particles 34-ID-C



Shen, Hao & Gruner, Physics Today (March 2006)

Summary

❖ ***X-ray Microscopy & Imaging*** ***is an exciting research field with many on-going and potential applications, in both scanning probe and full field imaging modes.***

❖ ***ERL X-ray Source*** ***will open up novel x-ray imaging opportunities, especially in scanning x-ray micrsocopy, time-resolved phase imaging and coherent diffraction imaging areas, because of its round diffraction-limited source, its high degree of coherence, and its short-pulse capabilities.***

❖ ***Novel materials research*** ***almost impossible to do today may be possible with the ERL source, such as nano-domain engineering in solar-cell materials, imaging electrochemical deposition at high temporal and spatial resolution, imaging particulates and polymer aggregates formations, and real-time imaging of surface and interfaces during chemical reactions, etc.***

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Thank You !