Nanoparticle Structure Using Coherent X-ray Diffraction

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Morris Fine Lecture
Northwestern University
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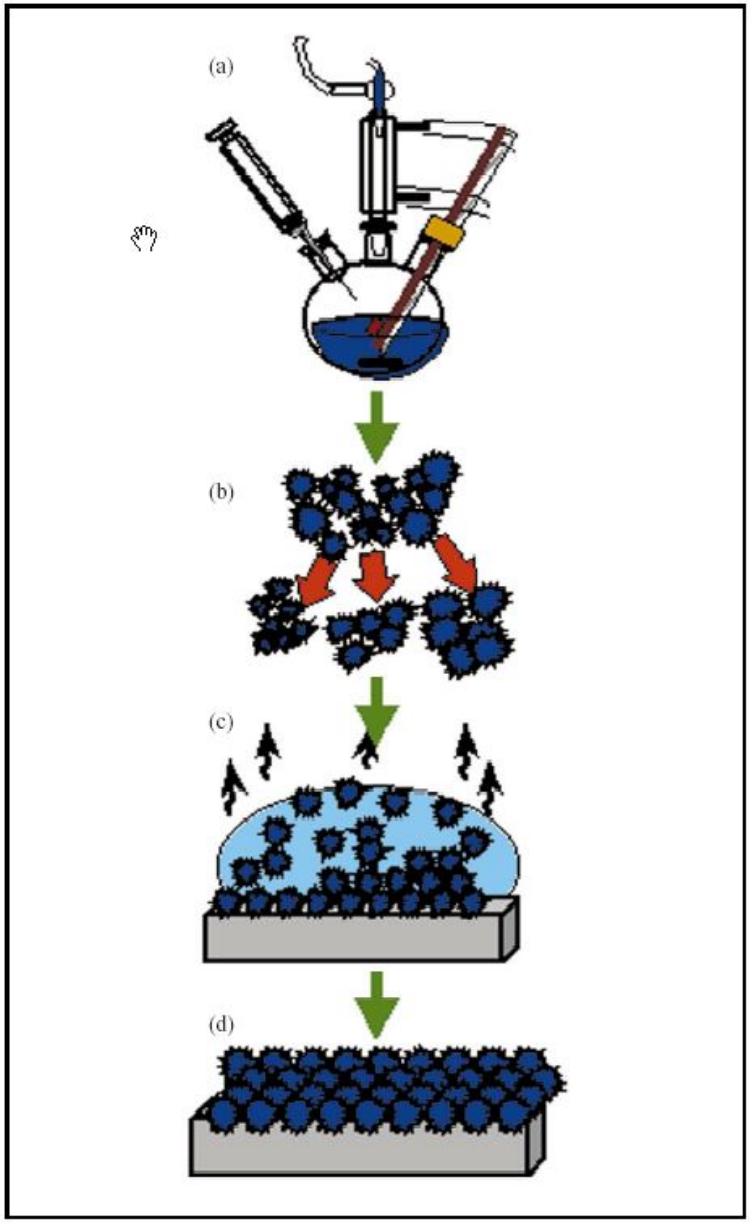
Outline

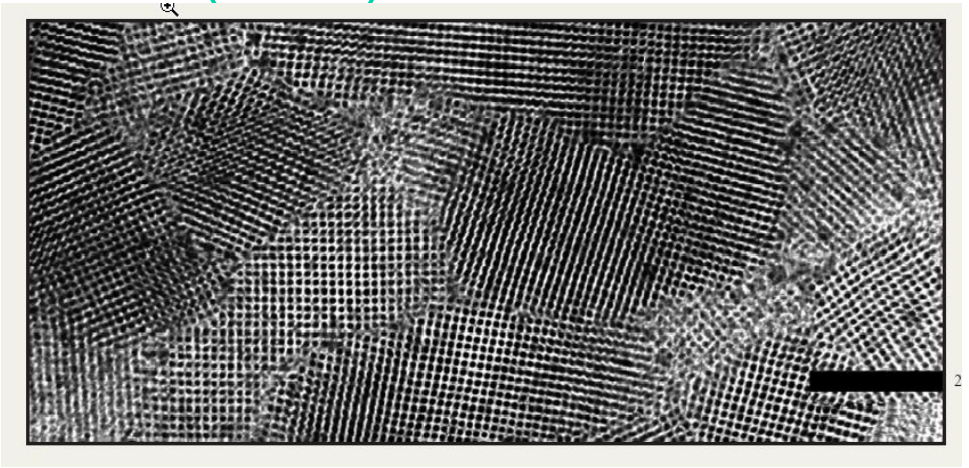
- Nanocrystal structures
- Coherent X-ray diffraction
- Crystal strain as complex density
- Strain induced by surface reactions
- Ultrafast snapshots of moving matter
- Crystallographic phase determination

Chemical Synthesis of Nanocrystals



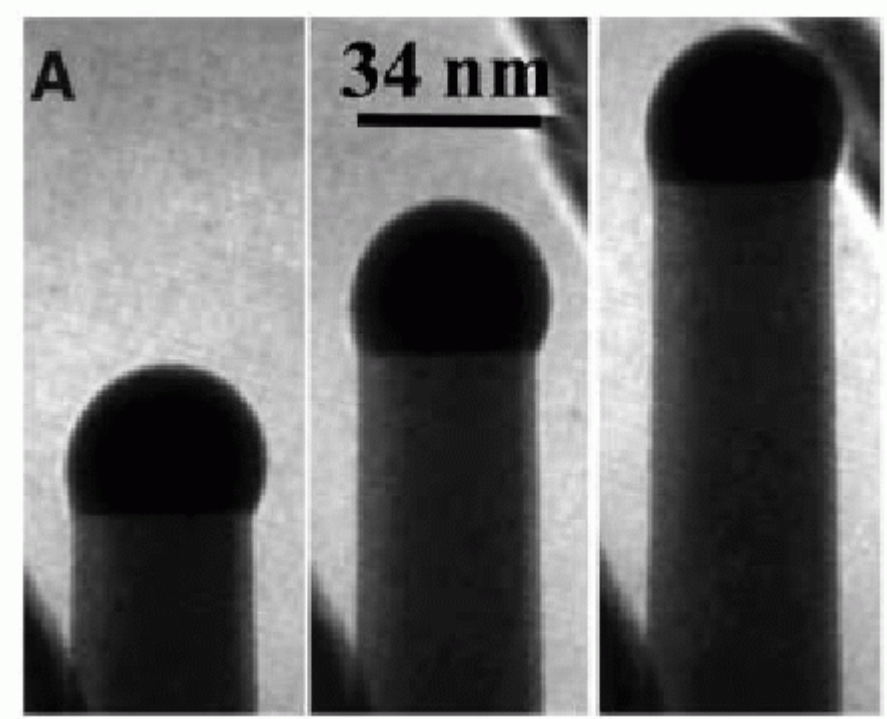
- Reactants introduced rapidly
- High temperature solvent
- Surfactant/organic capping agent
- Square superlattice (200nm scale)

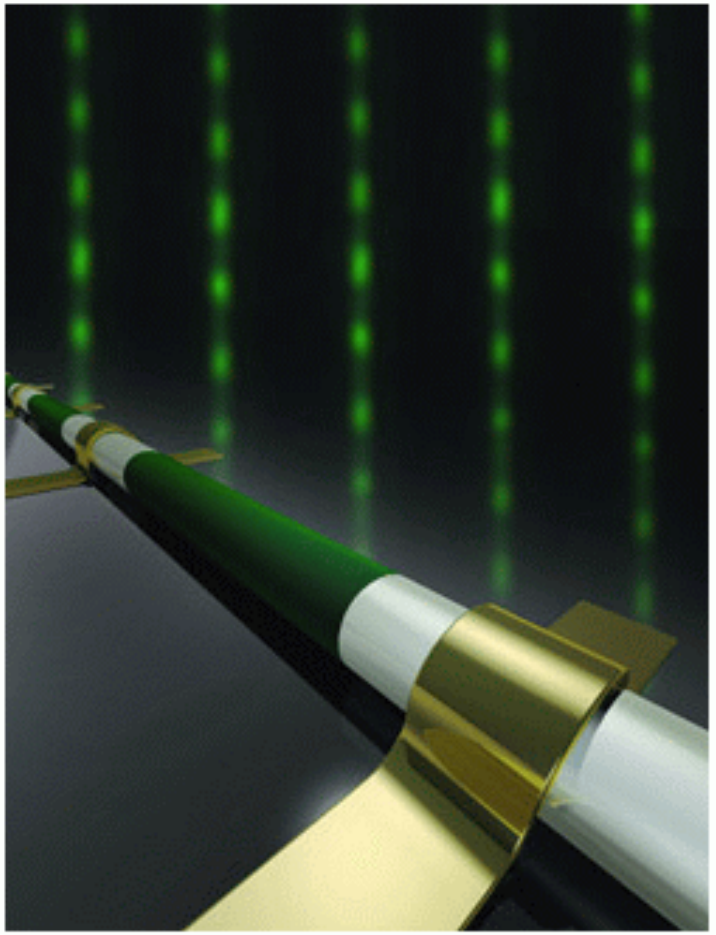
C. B. Murray, IBM J. Res. & Dev. **45** 47 (2001)



VLS growth of nanowires

S. Kodambaka et al., Science 316 729 (2007)

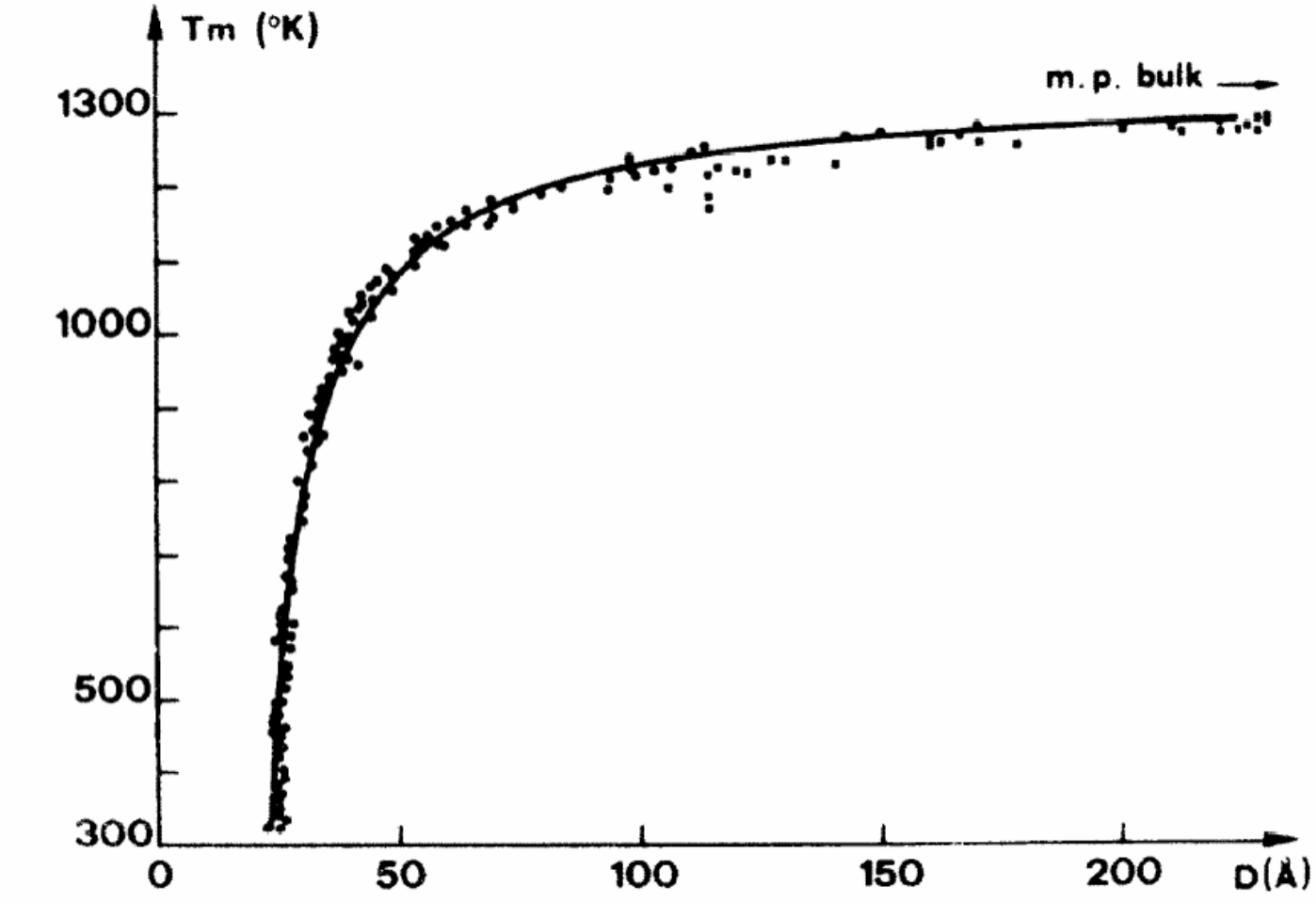




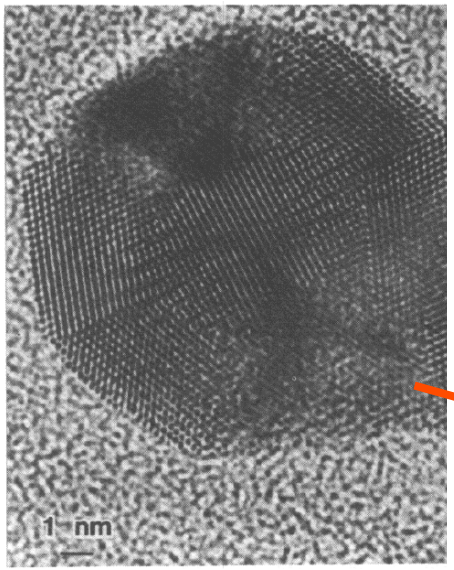
NiSi/Si nanowire heterostructure devices. *Nature* **430**, 61 (2004).

Size-dependent Melting of Au Particles

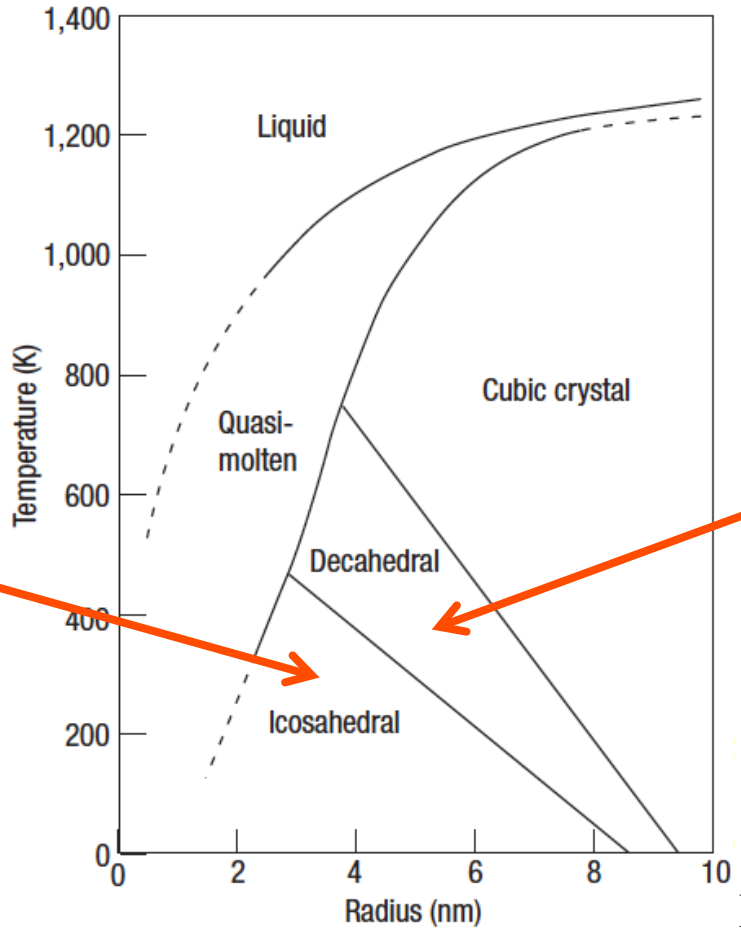
P. Buffat and J-P. Borel, Phys. Rev. A 2287-97 (1975)

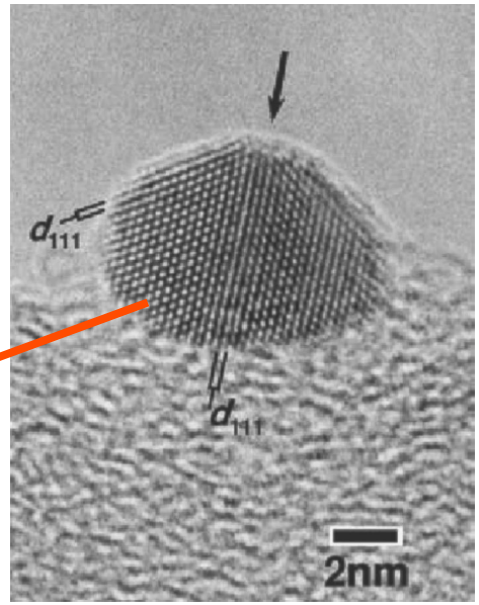


Structure of Gold vs Size



L. D. Marks, RPP (1994)





Koga and Sugawara (2003)

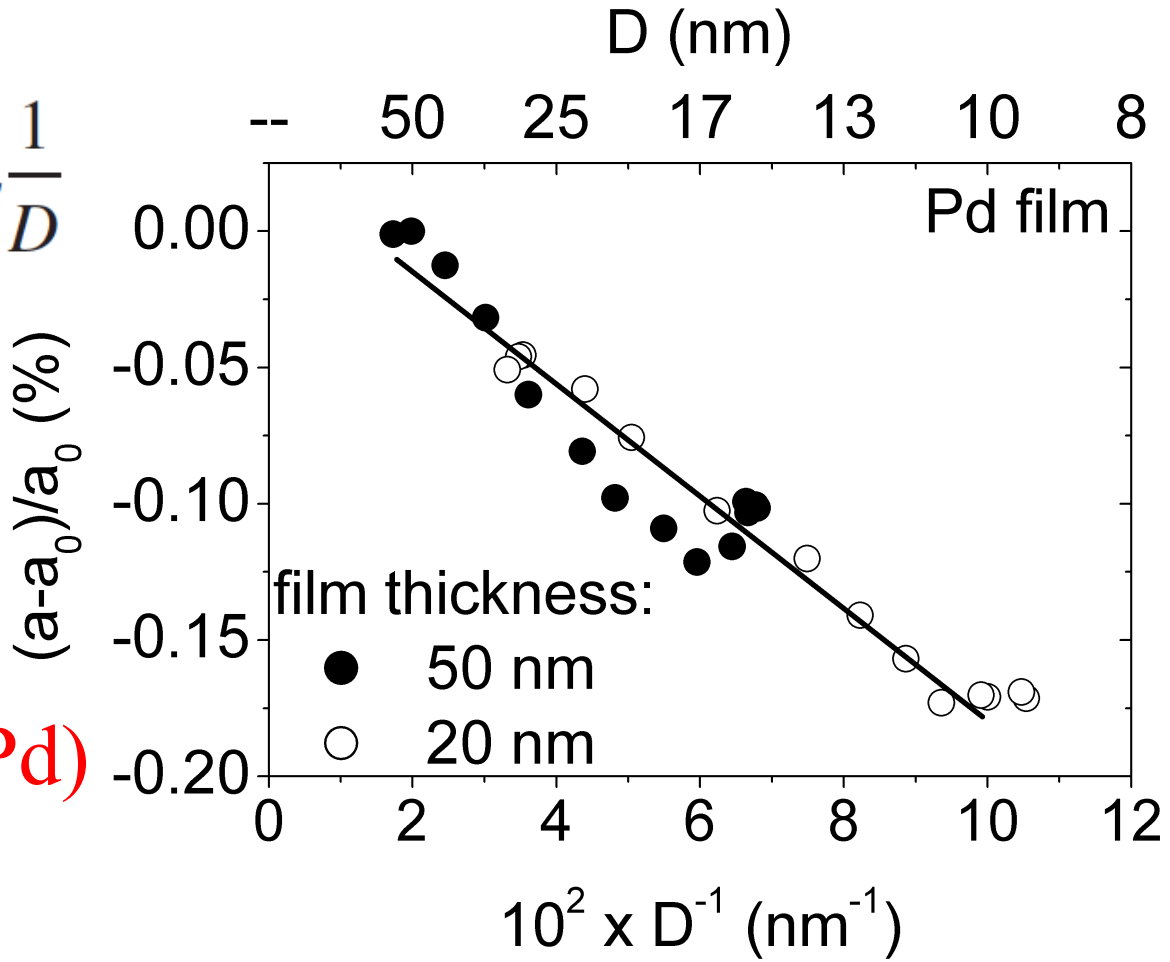
Contraction of Small Particles

Gibbs Thomson pressure + Bulk modulus

Sheng, Welzel & Mittemeijer, APL 97 153109 (2010)

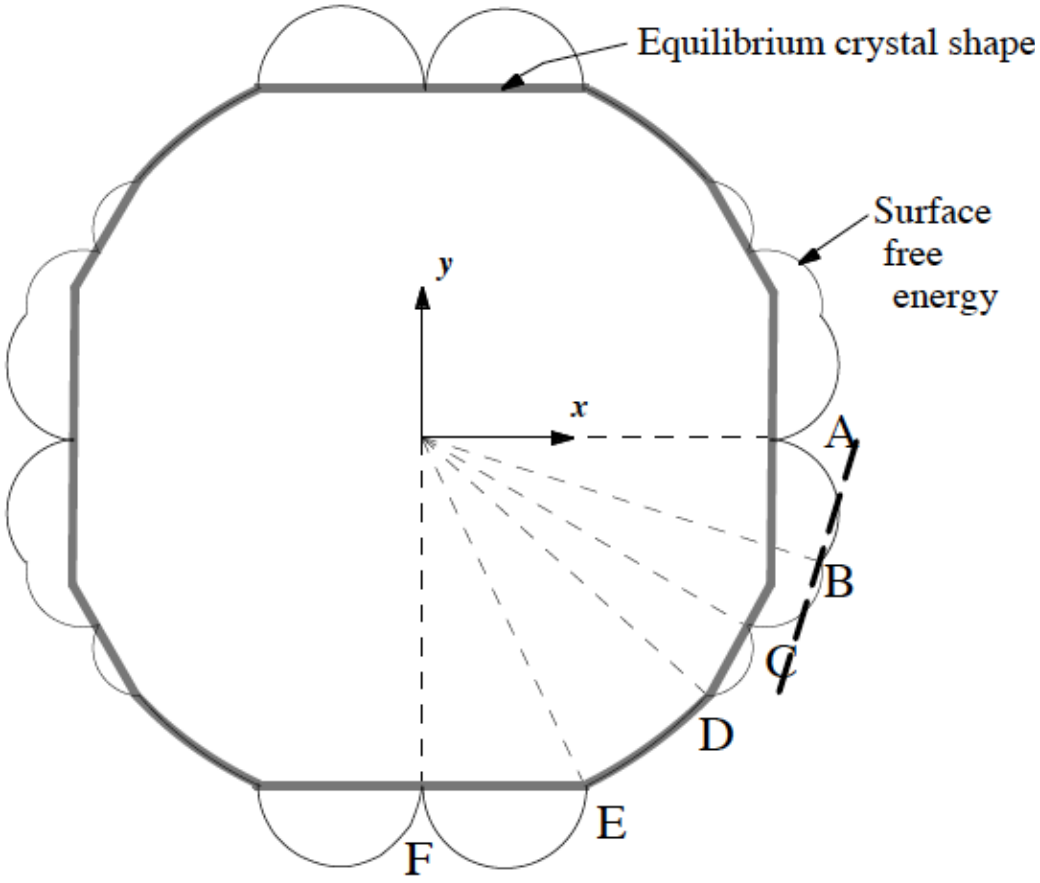
$$\frac{a - a_0}{a_0} = -\frac{4}{3K}\sigma_S\frac{1}{D}$$

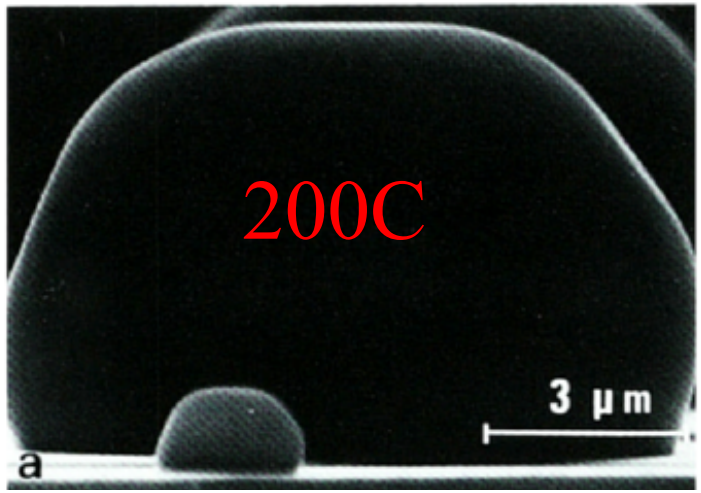
$\sigma_S = 2\text{N/m}$ (Pd)

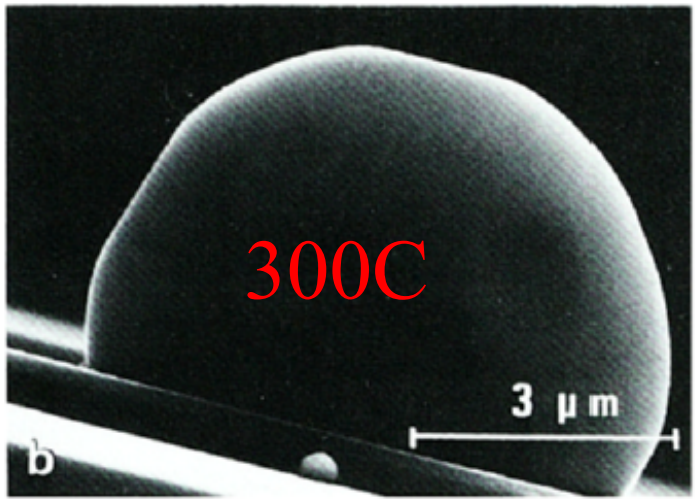


Equilibrium crystal shape

Wulff construction; Heyraud & Metois, Surf Sci 128 334 (1983)

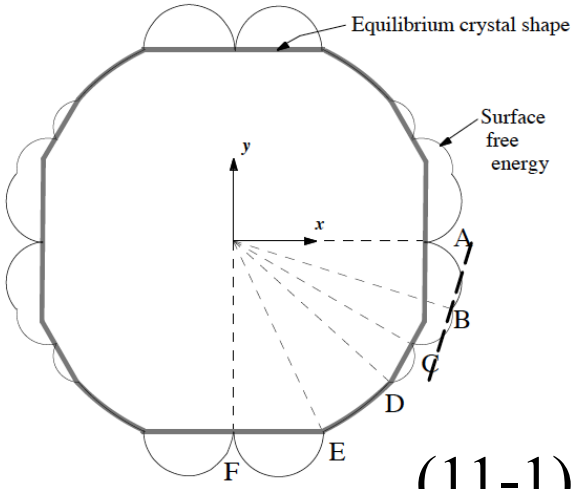






Equilibrium crystal shape

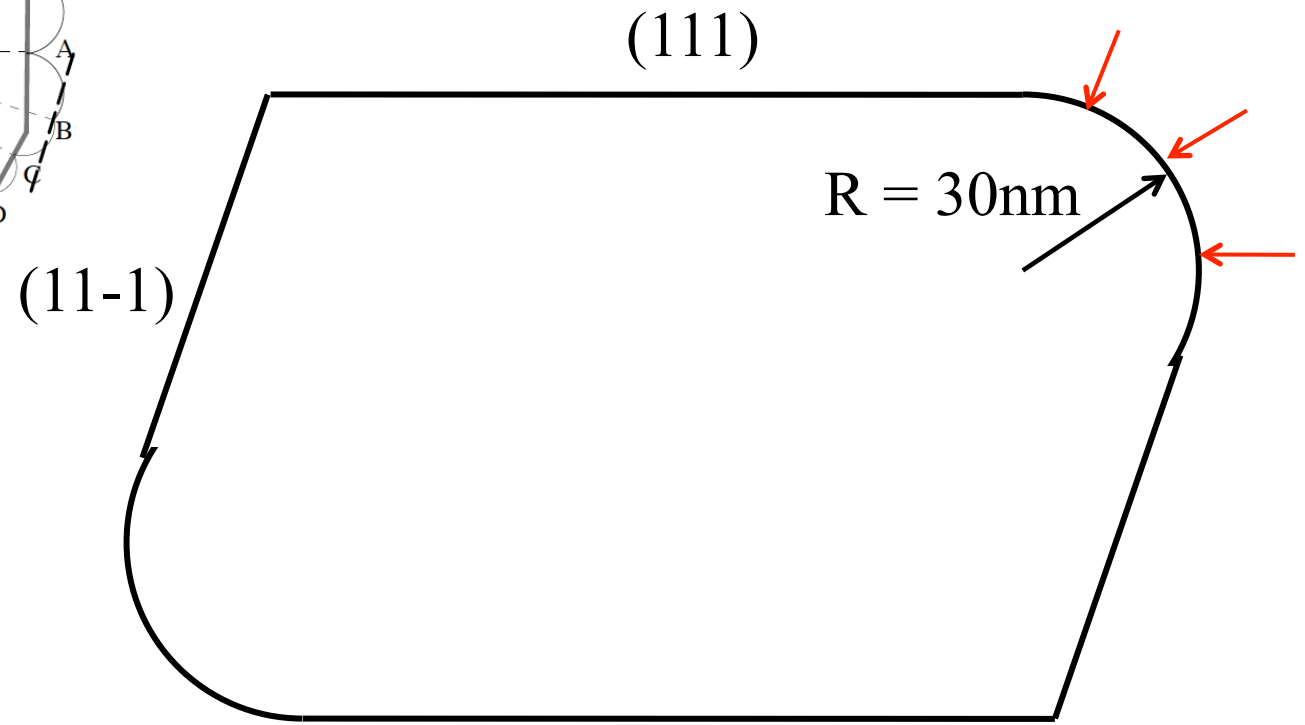
Wulff construction + Gibbs Thomson (Young-Laplace) pressure



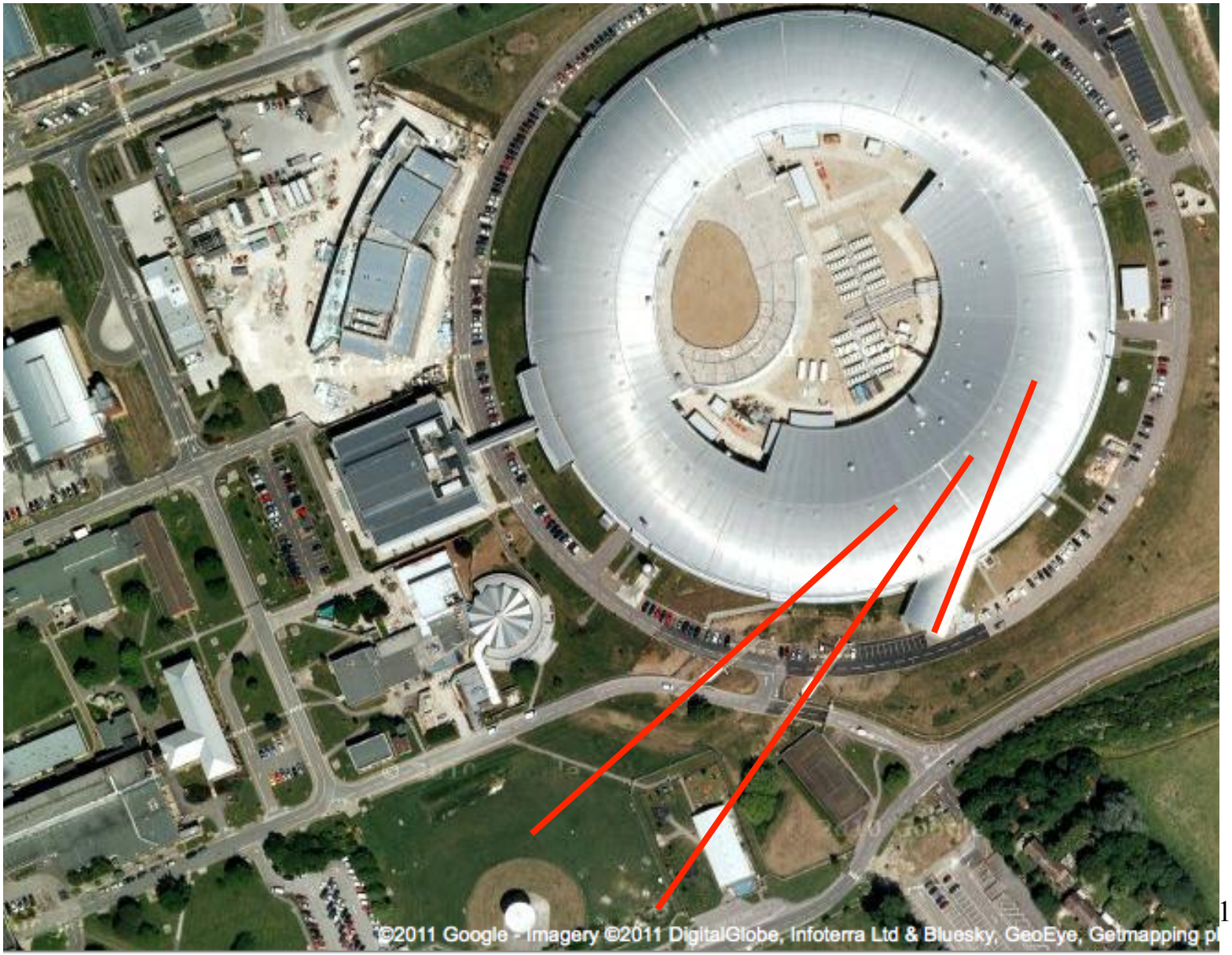
$\gamma = 2\text{N/m}$

$P = 2\gamma/R = 70\text{MPa}$

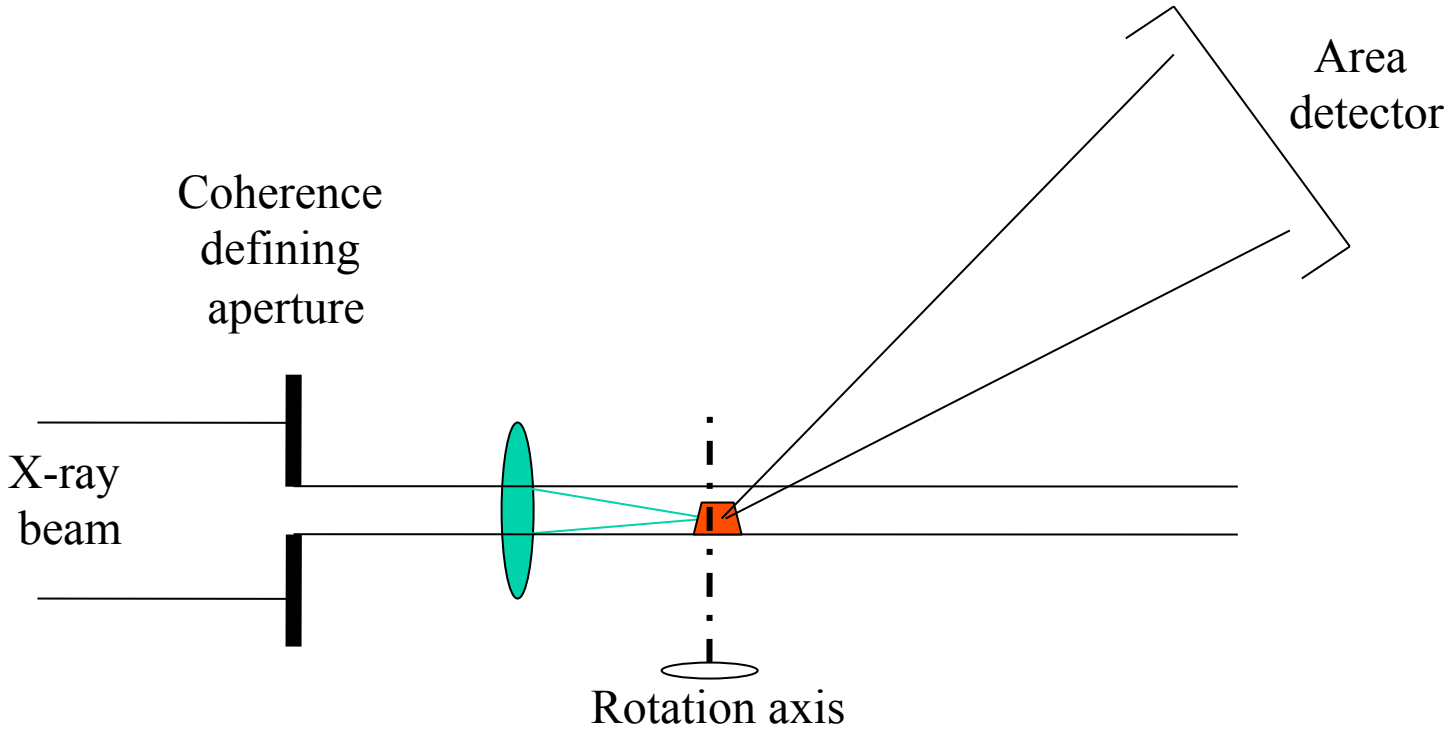
$\varepsilon = 0.1\%, \quad \Phi = 0.6\text{ rad}$

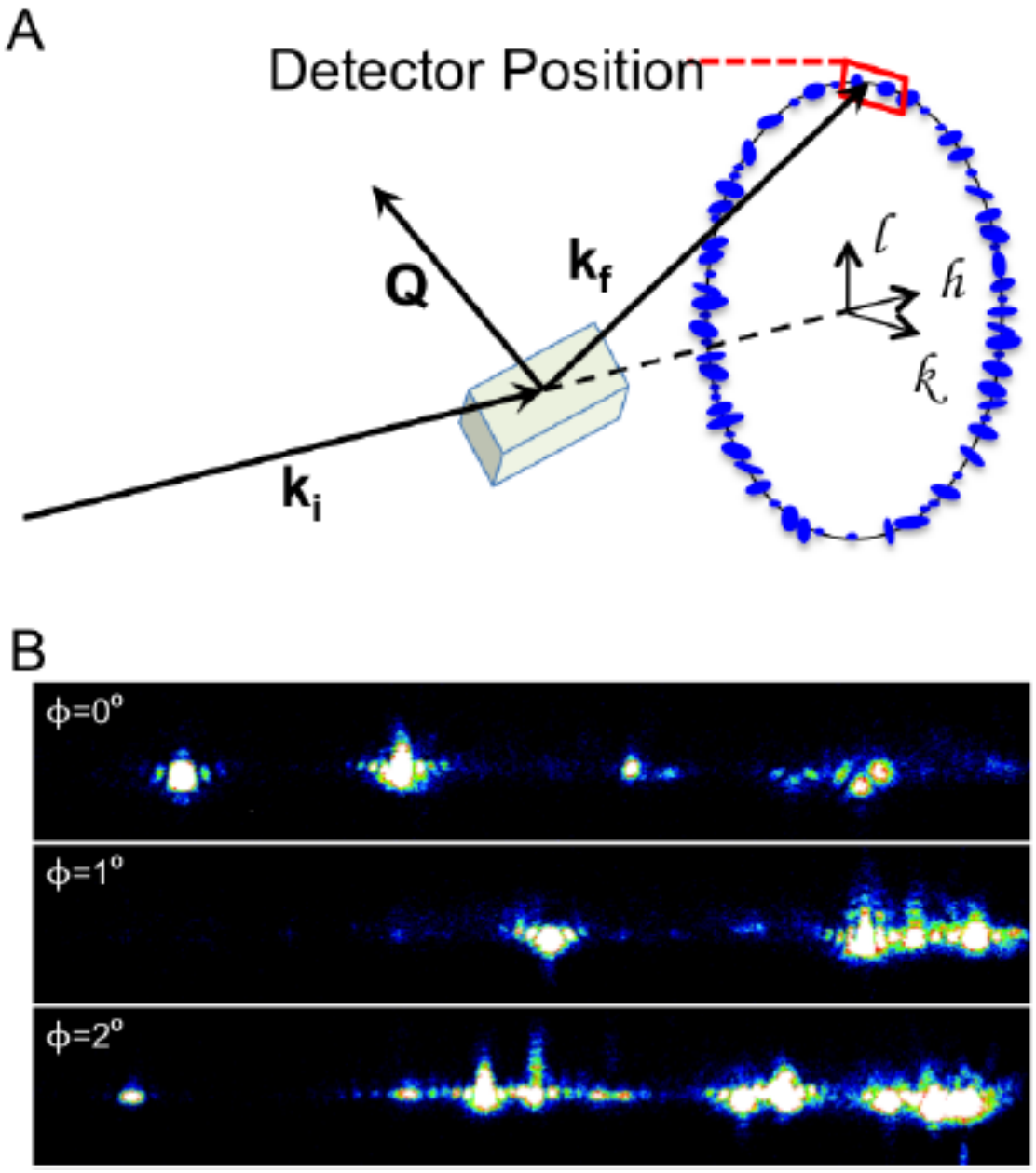


Diamond Light Source, 2009

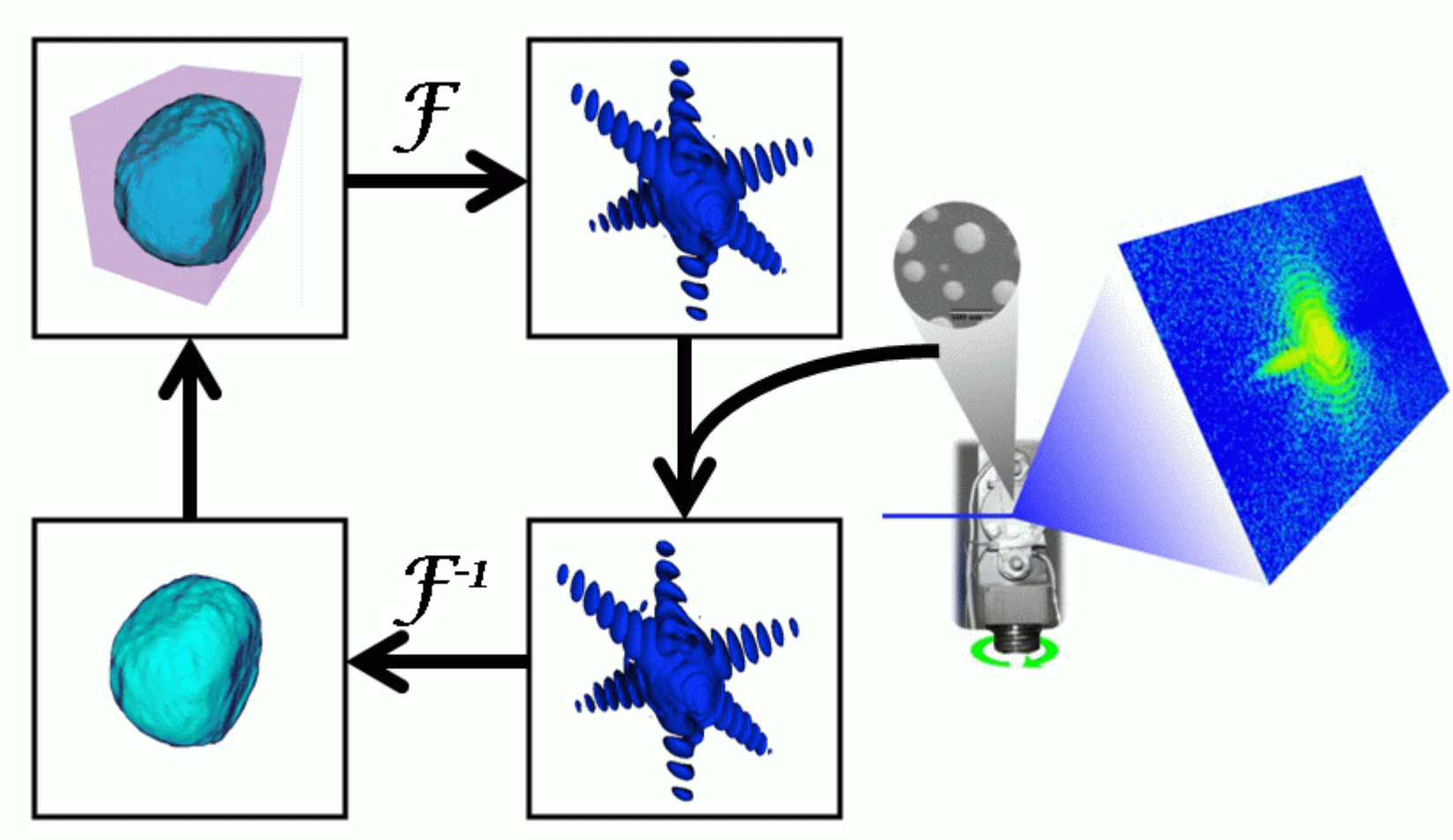


Lensless X-ray Microscope, 2003




A
Detector Position
Q
k_f
k_i
l
h
k
B
ϕ=0°
ϕ=1°
ϕ=2°

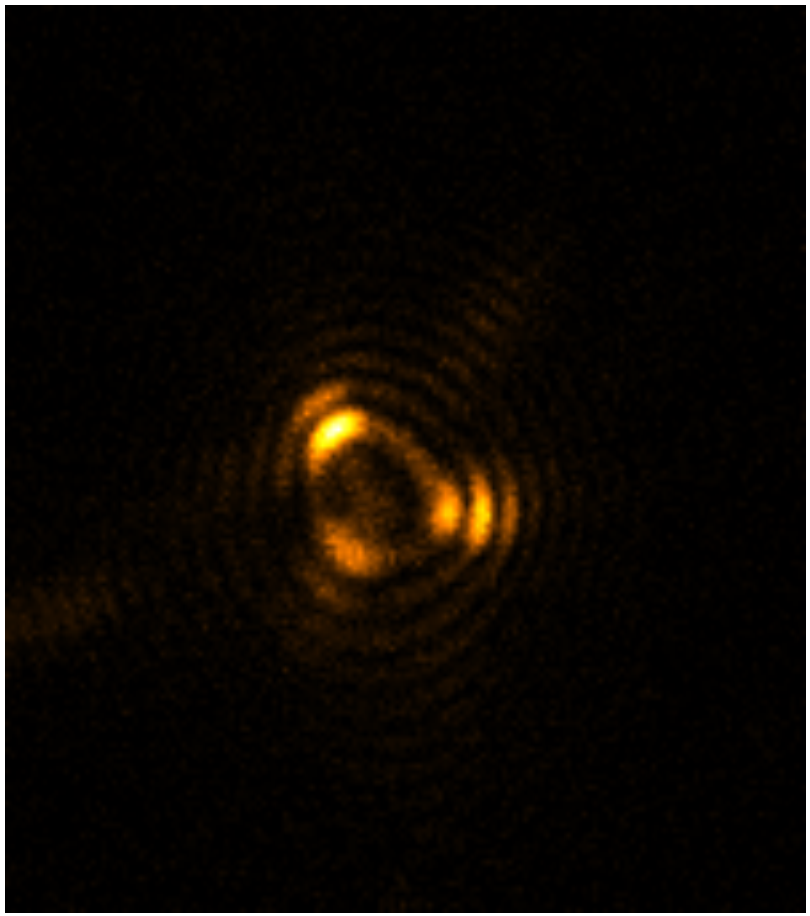
Generic "Error Reduction" method

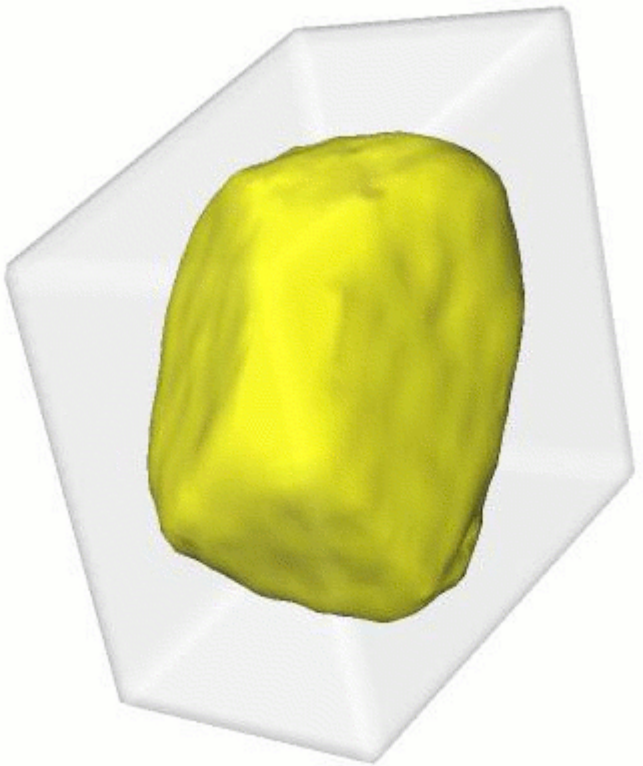


J. R. Fienup Appl. Opt. 21 2758 (1982)
R. W. Gerchberg and W. O. Saxton Optik 35 237 (1972)

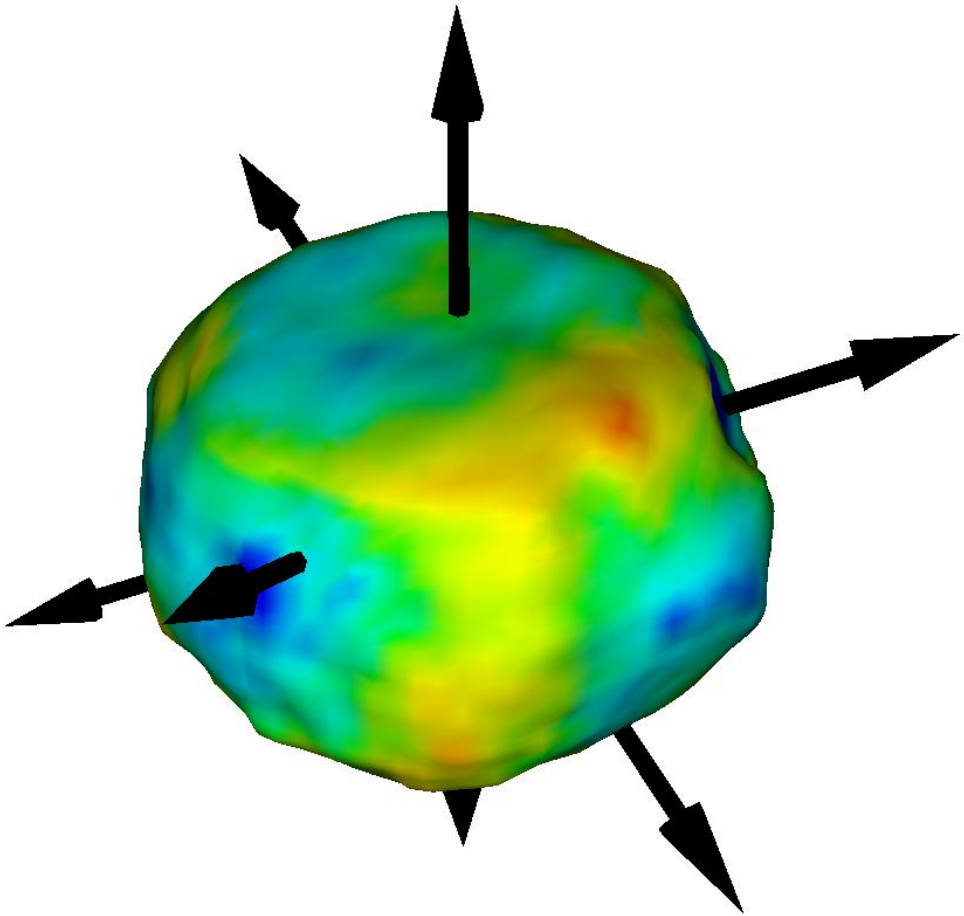
Gold nanocrystal reconstruction

showing support used for 20 HIO followed by 10 ER



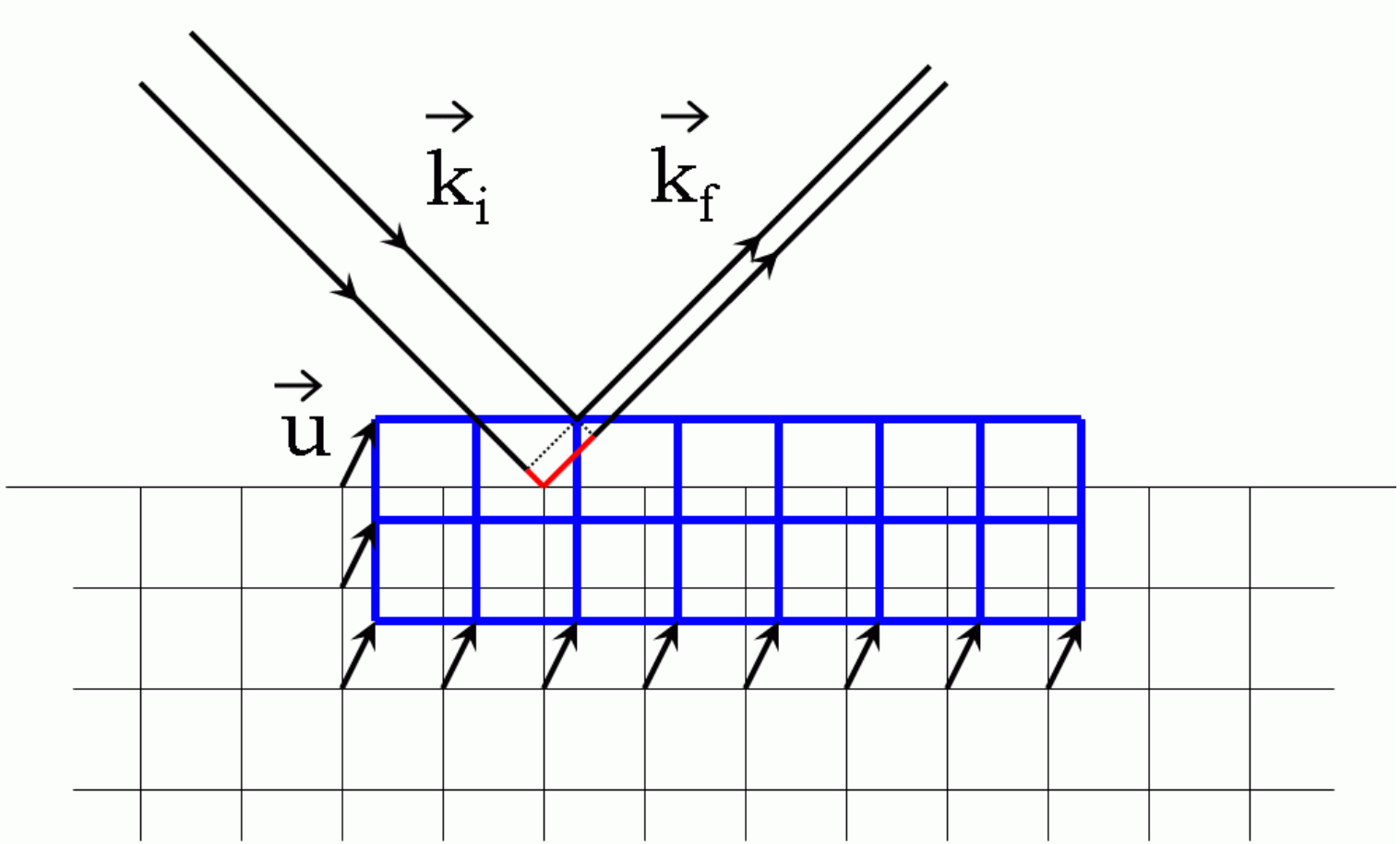


Phase isosurface of residual strain



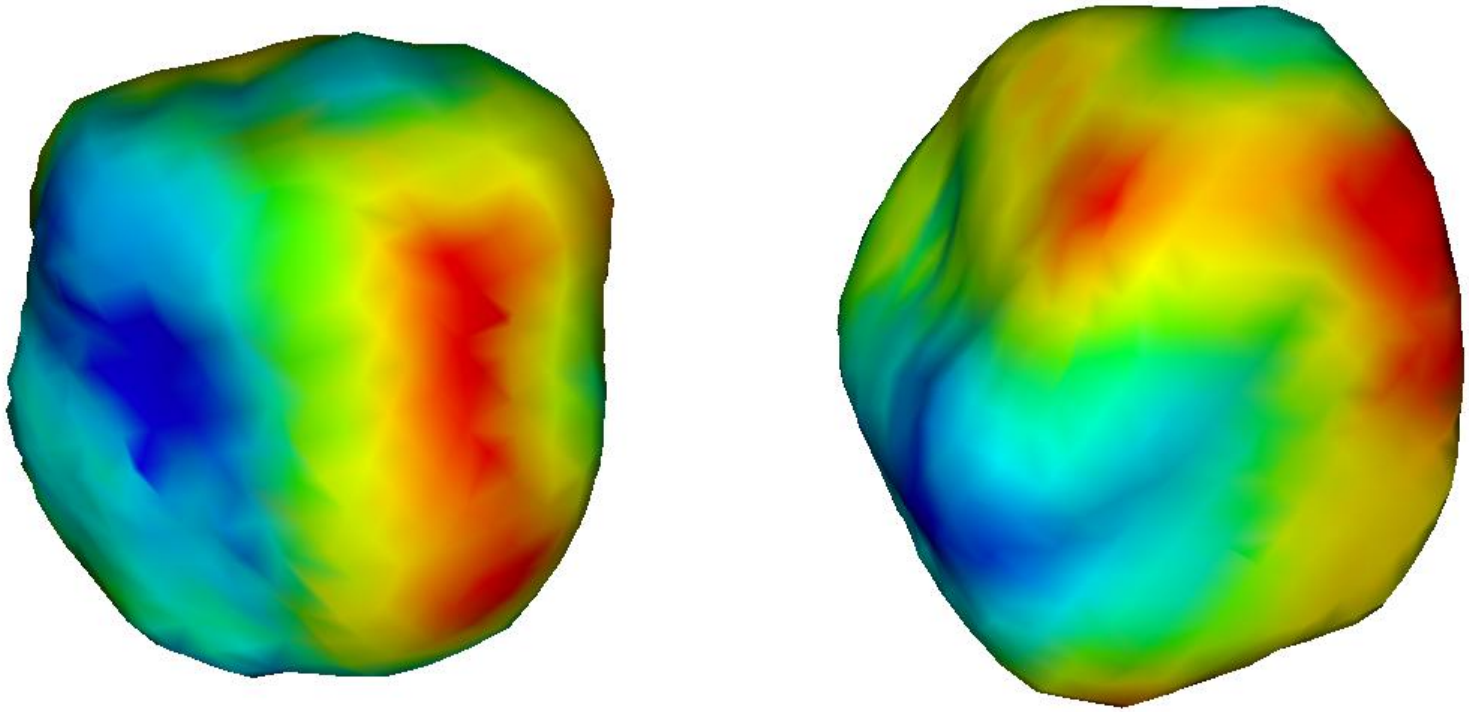
Sensitivity to strain

$$\Delta\varphi = \mathbf{k}_f \cdot \mathbf{u} - \mathbf{k}_i \cdot \mathbf{u} = \mathbf{Q} \cdot \mathbf{u}$$



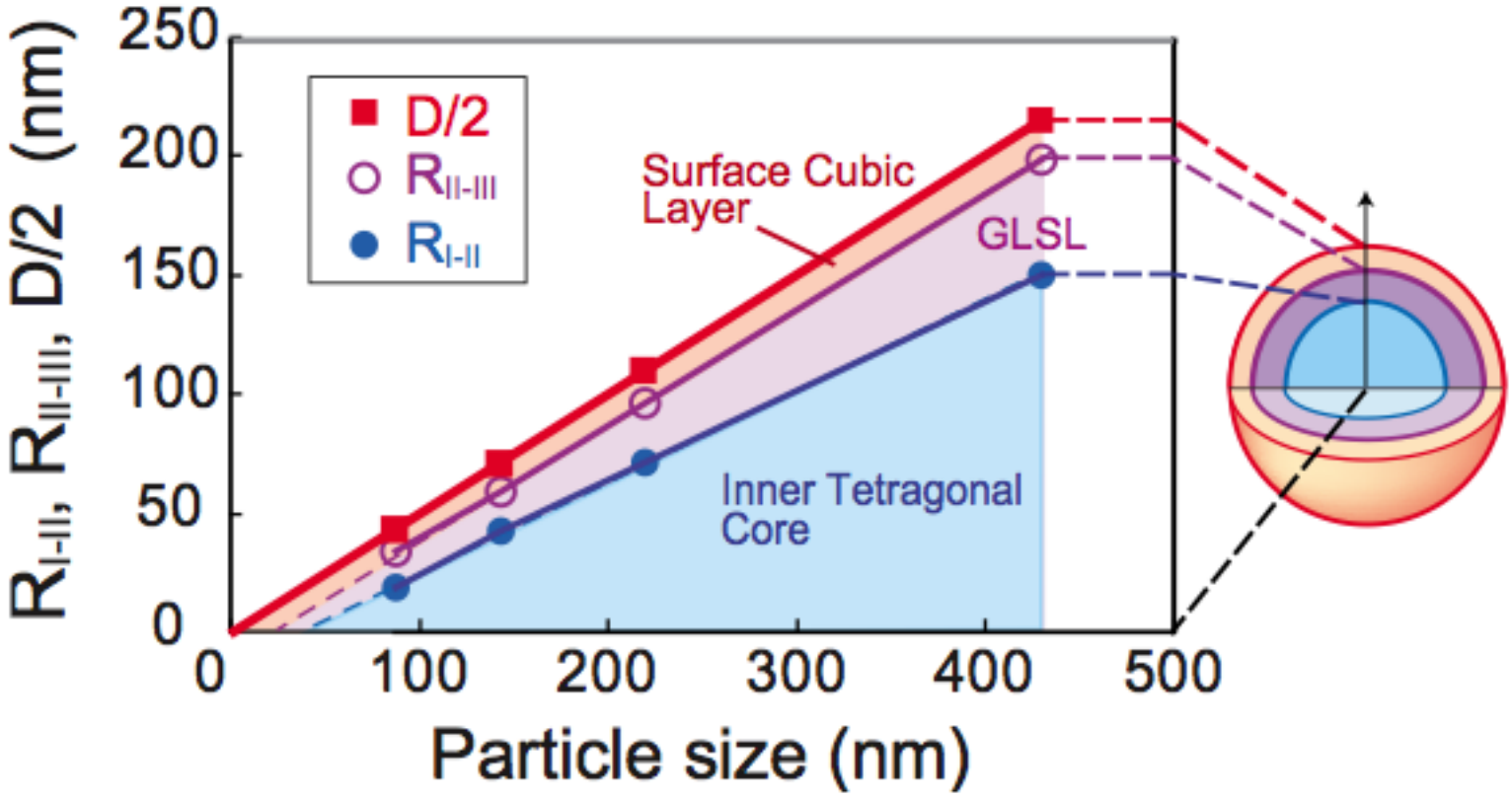
Phase isosurface of residual strain

200nm Barium Titanate (BTO) crystals



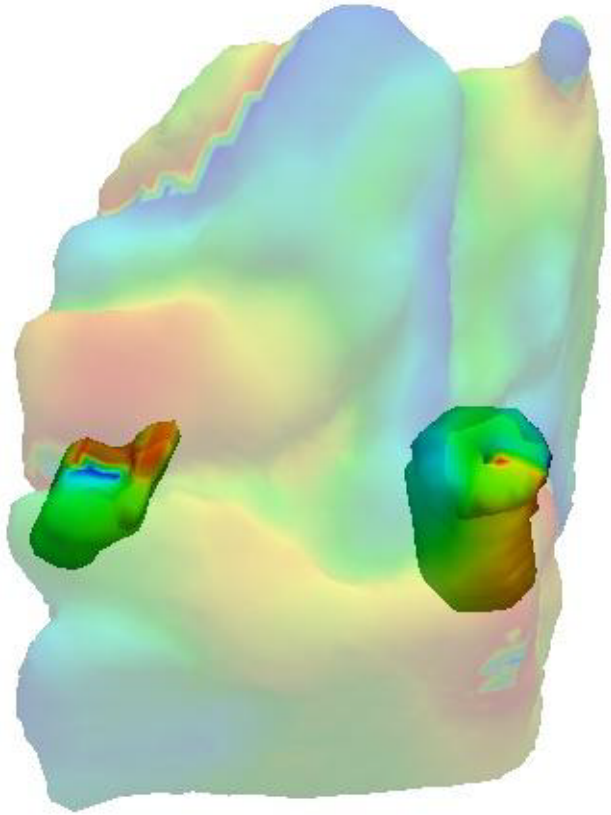
Core-shell structure of BTO

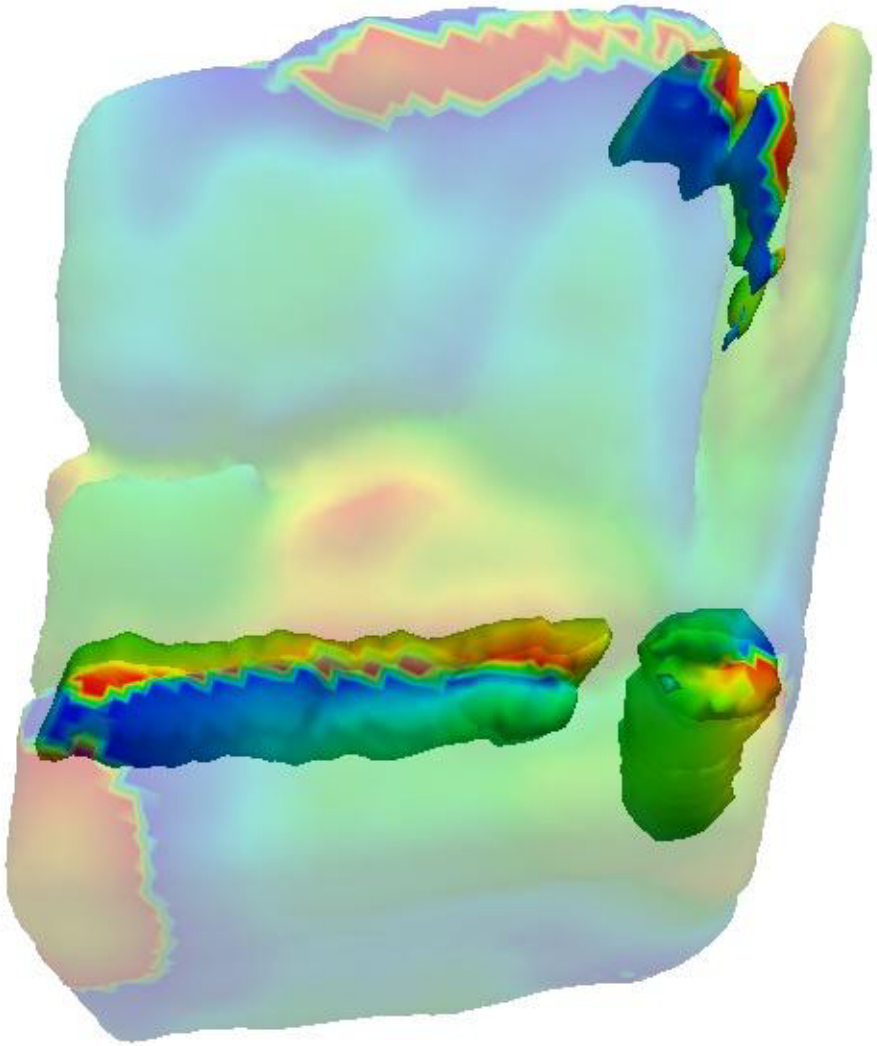
Takuya Hoshina, et al Appl. Phys. Lett. 93, 192914 (2008)



Calcite crystal growth

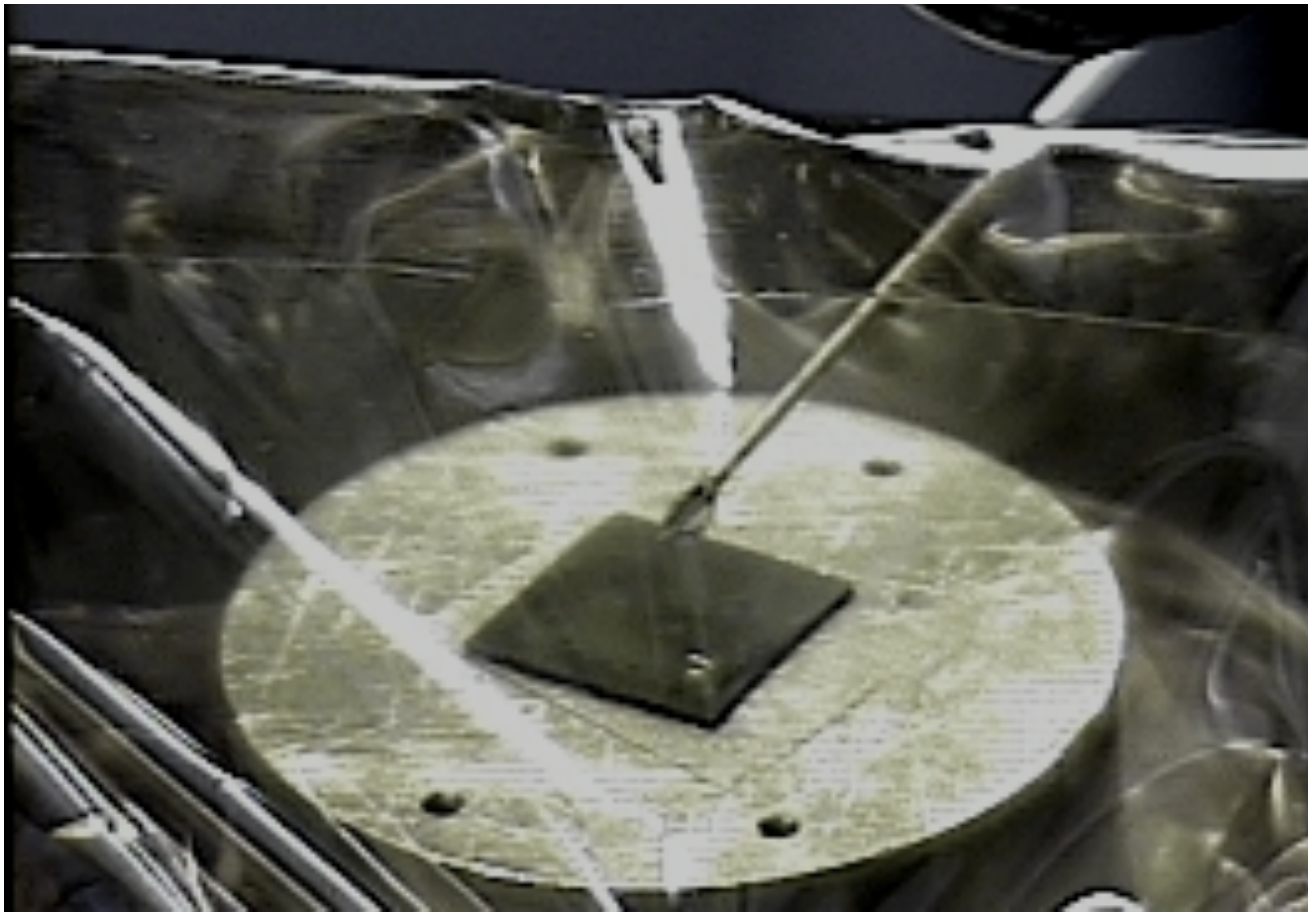
Jesse Clark and Johannes Ihli unpublished





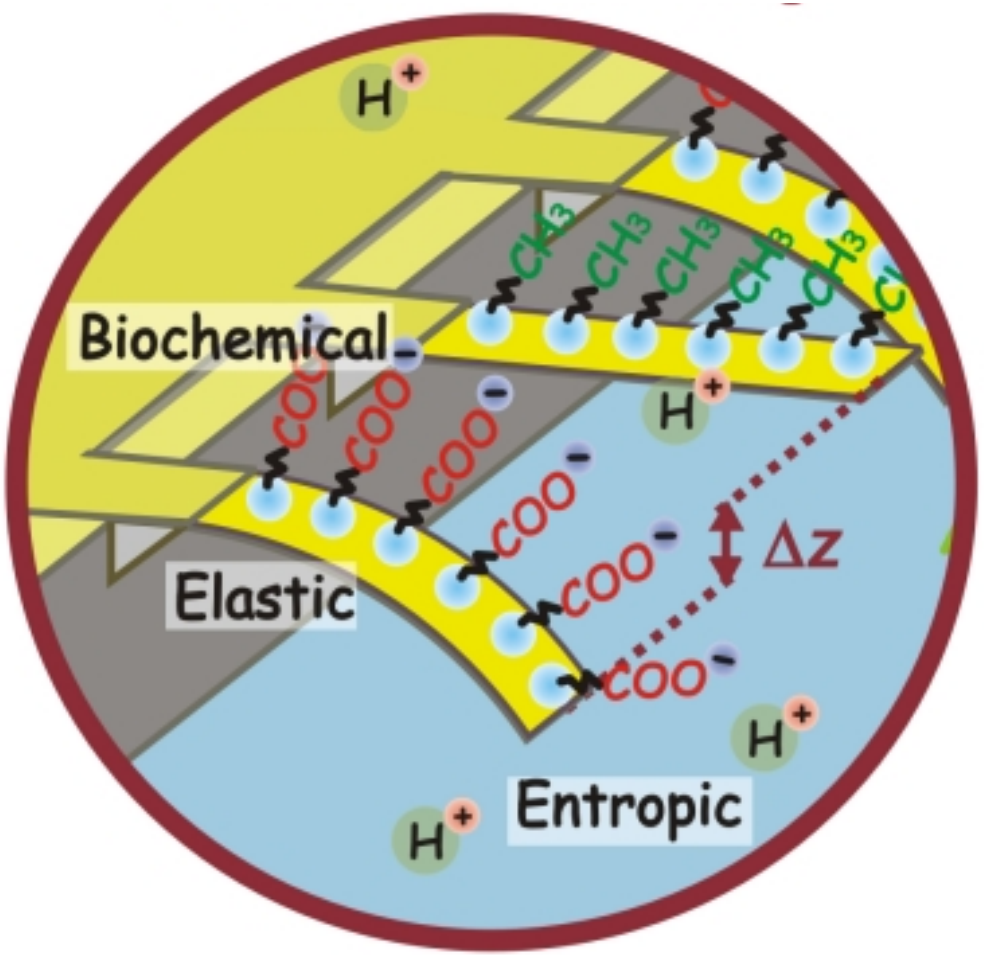
Dosing with C_3H_7SH in ethanol

Moyu Watari, Rachel McKendry, Manuel Voegtli, Gabriel Aeppli, Yeong-Ah Soh, Xiaowen Shi, Gang Xiong, Xiaojing Huang, Ross Harder and Ian Robinson, Nature Materials (2011)



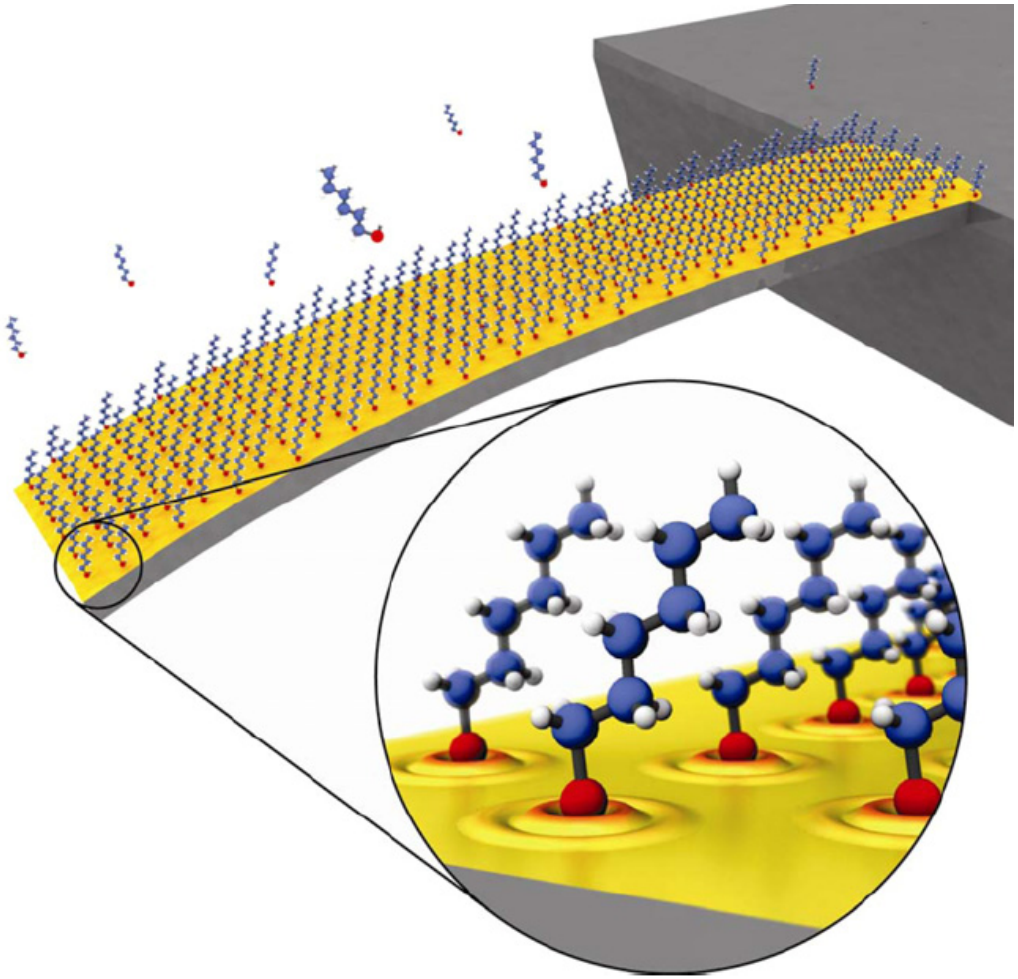
Nanocantilevers

Dr Rachel McKendrie



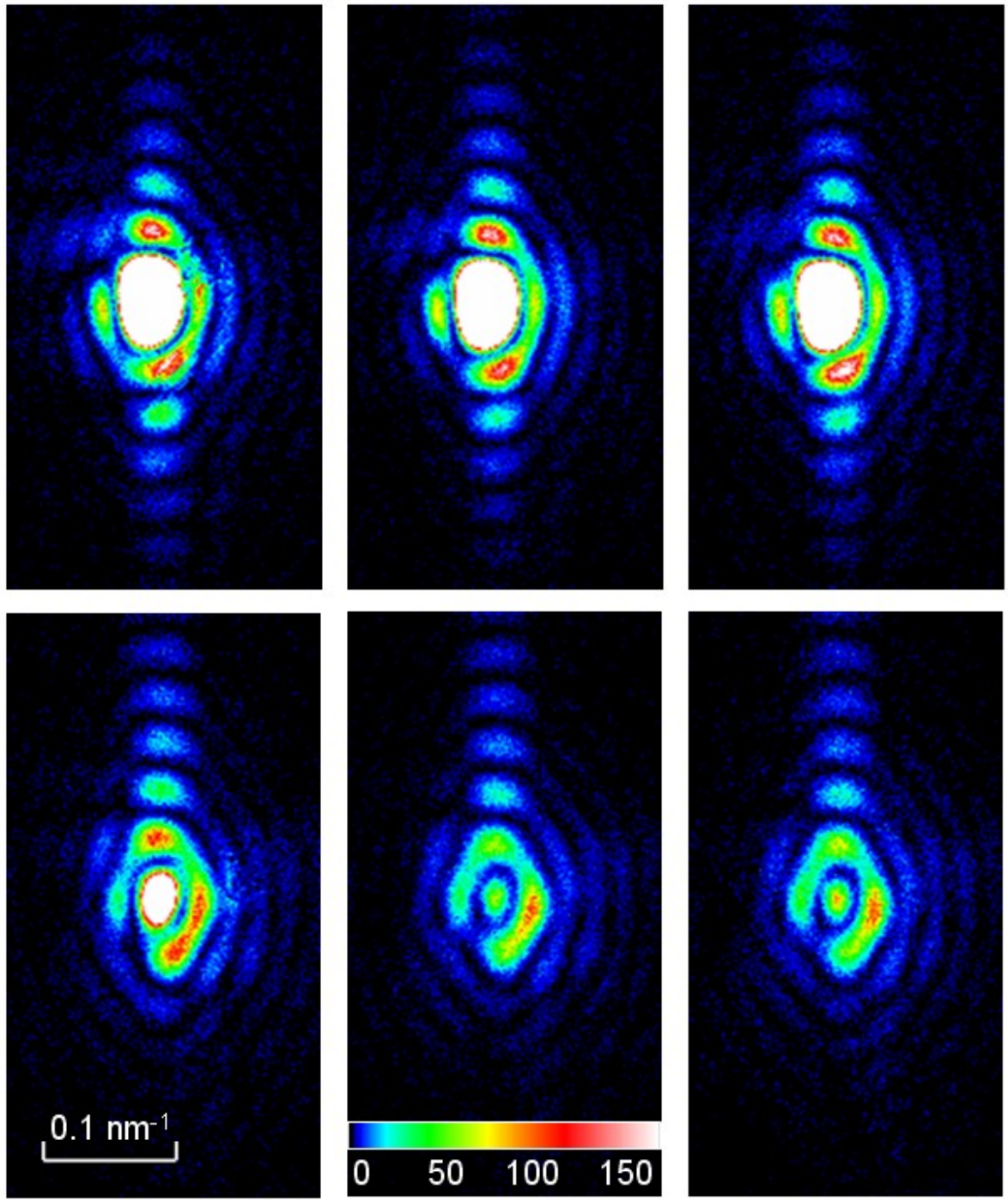
Formation of SAM on cantilever

M. Godin et al, Nanotechnology 21 (2010) 075501



Rocking curve of Au crystal

Before dosing / dose 1 / dose 2

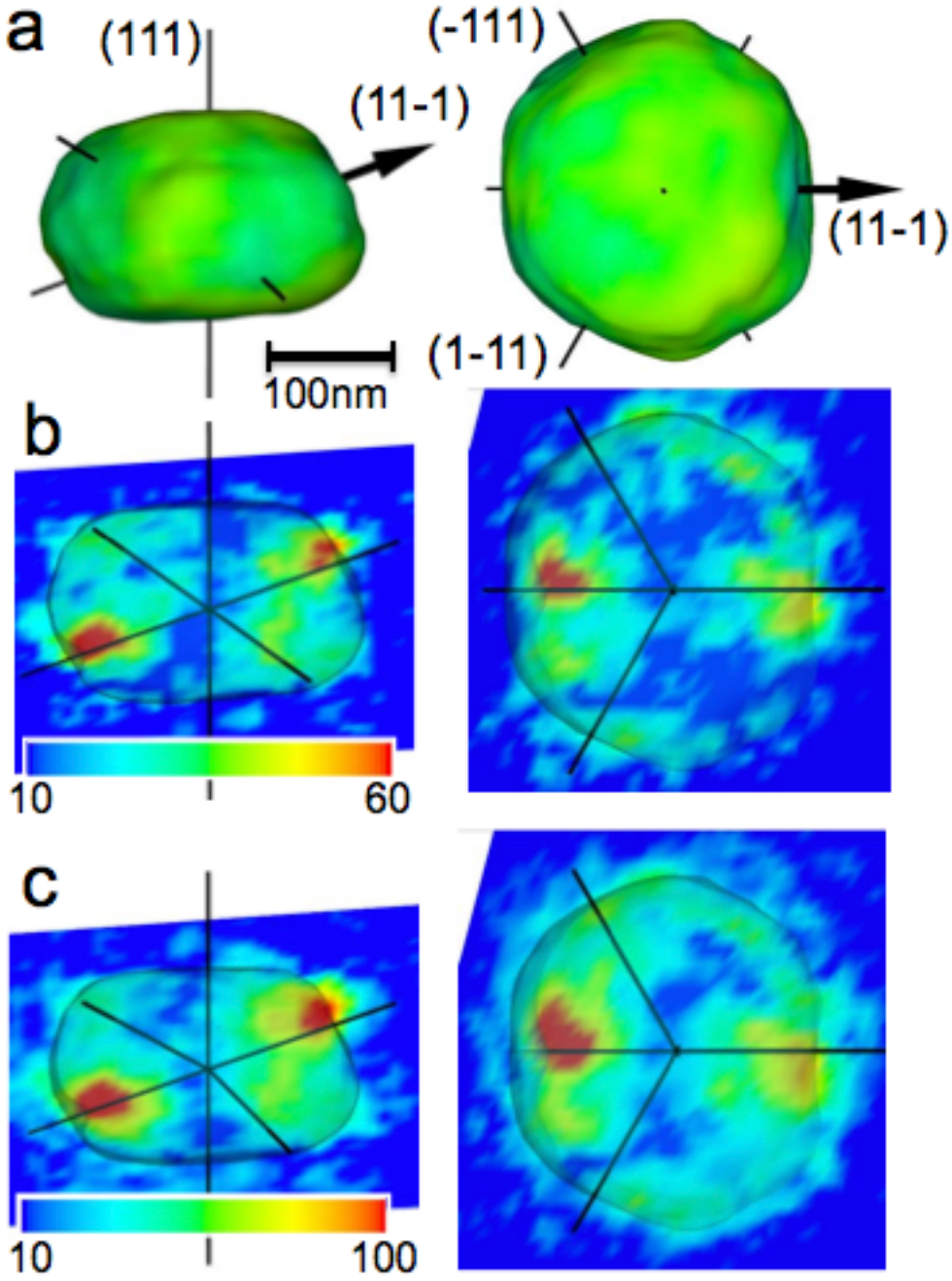


I.

Difference Fourier:

Magnitude of phase change caused by dosing

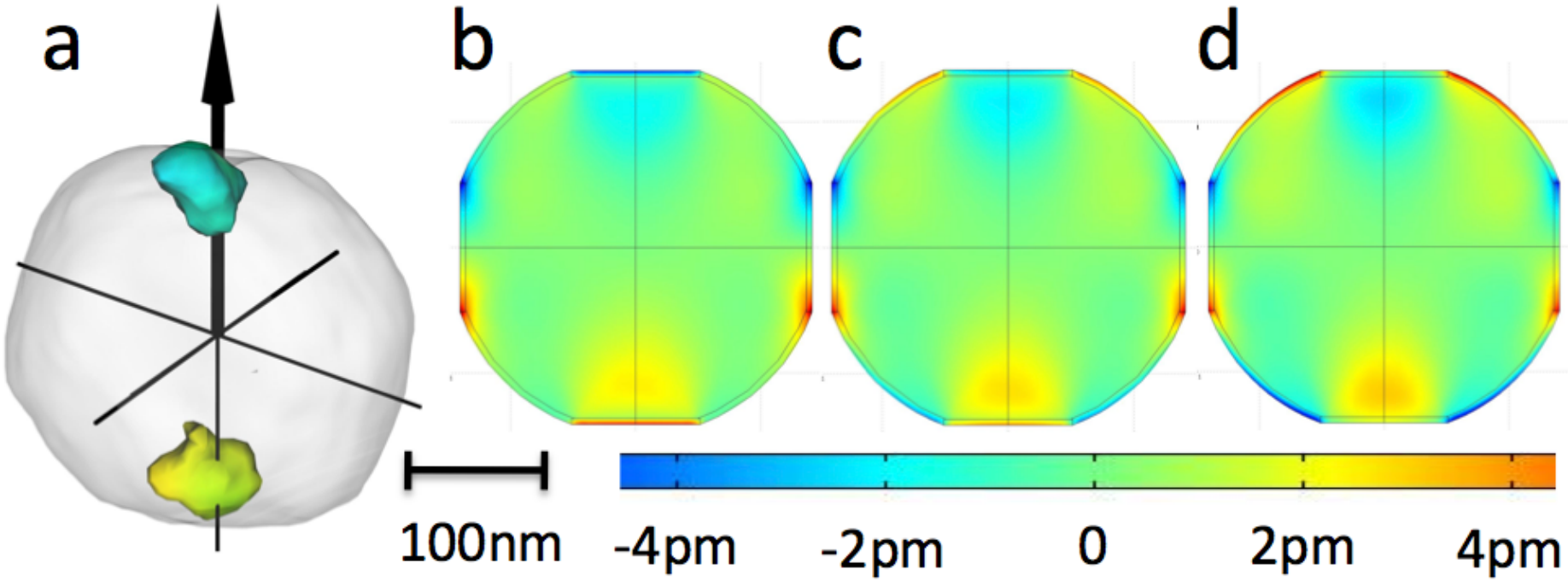
Moyu Watari et al, Nature Materials (2011)



Finite-element Analysis

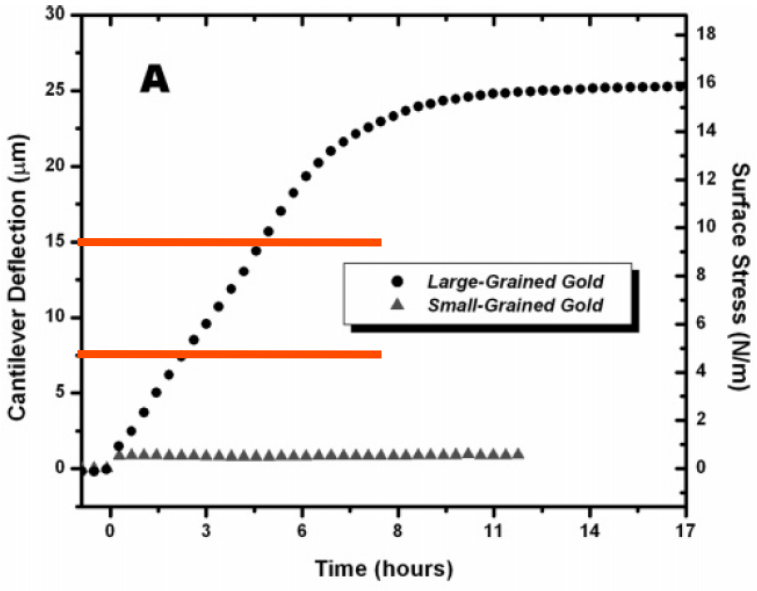
Differential stress introduced in "skin"

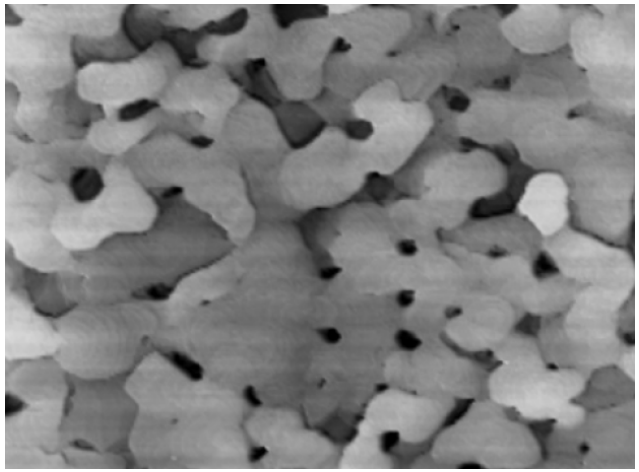
+/- 3×10^8 Pa in 5nm thickness = 1.5N/m



Cantilever strain experiment

M. Godin et al Langmuir **20** 7090 (2004)

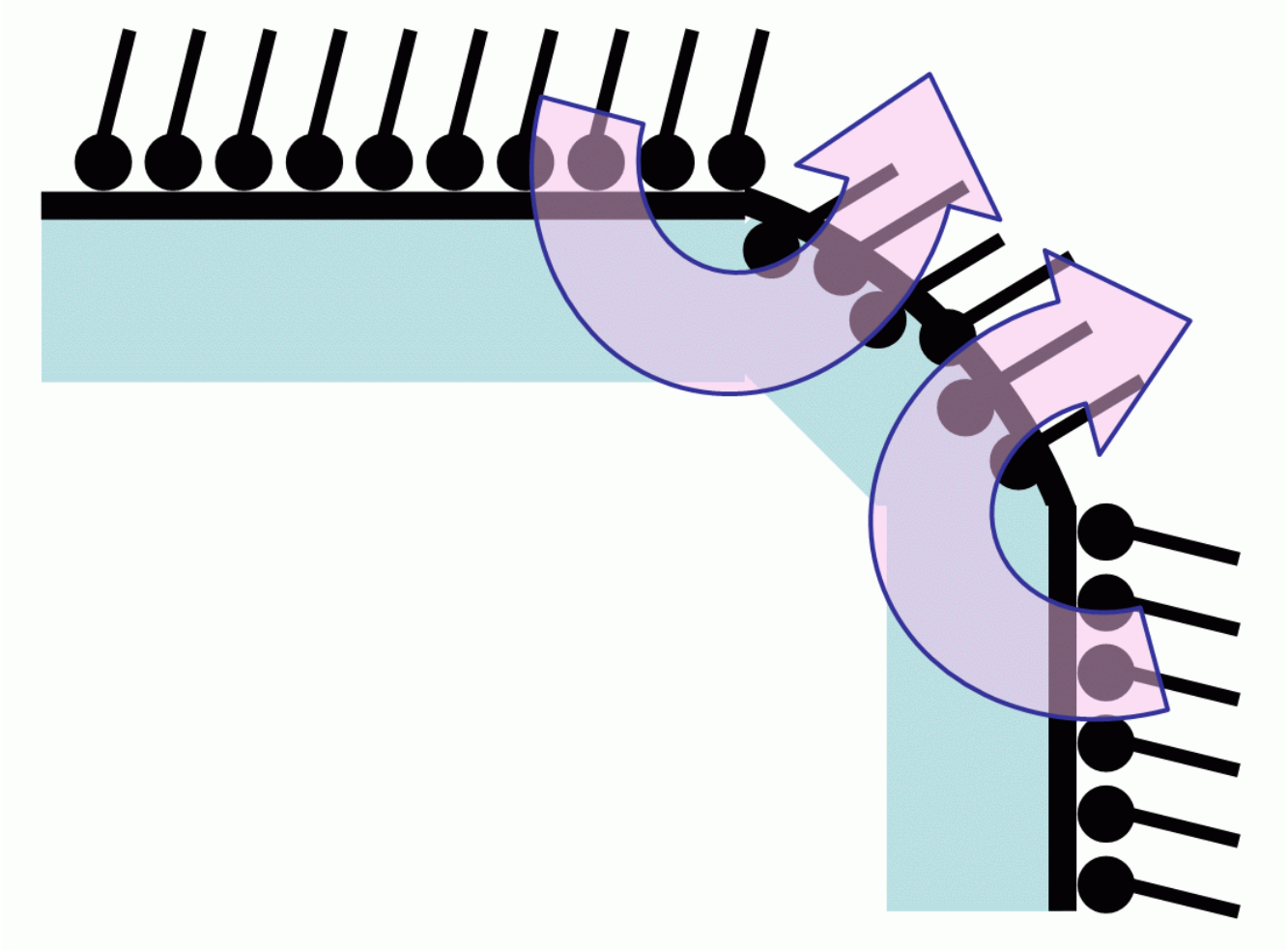




3×2μm STM image

Calculated surface stress from FEA analysis:
4.5±2N/m after 1hr, 9.5±3N/m after 2hrs

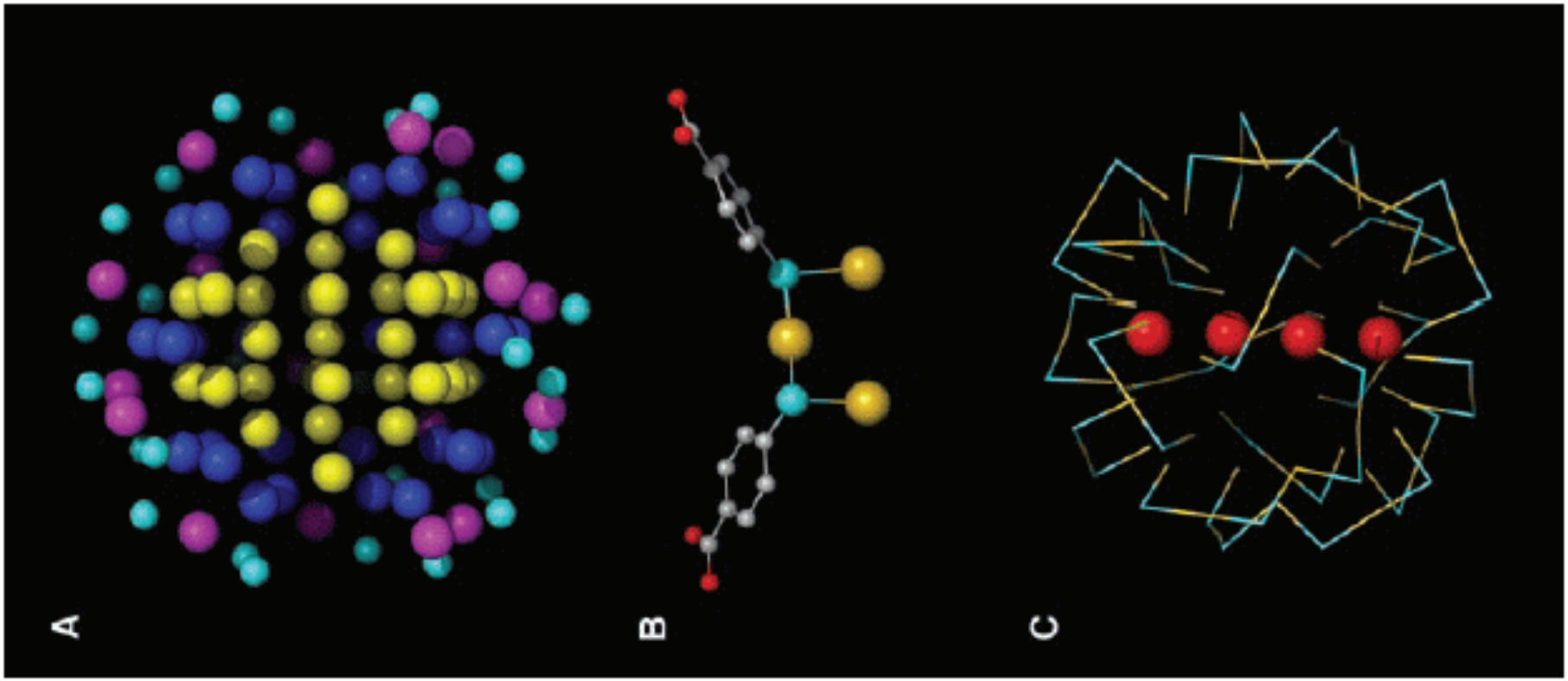
Differential adsorption, thiol/Au

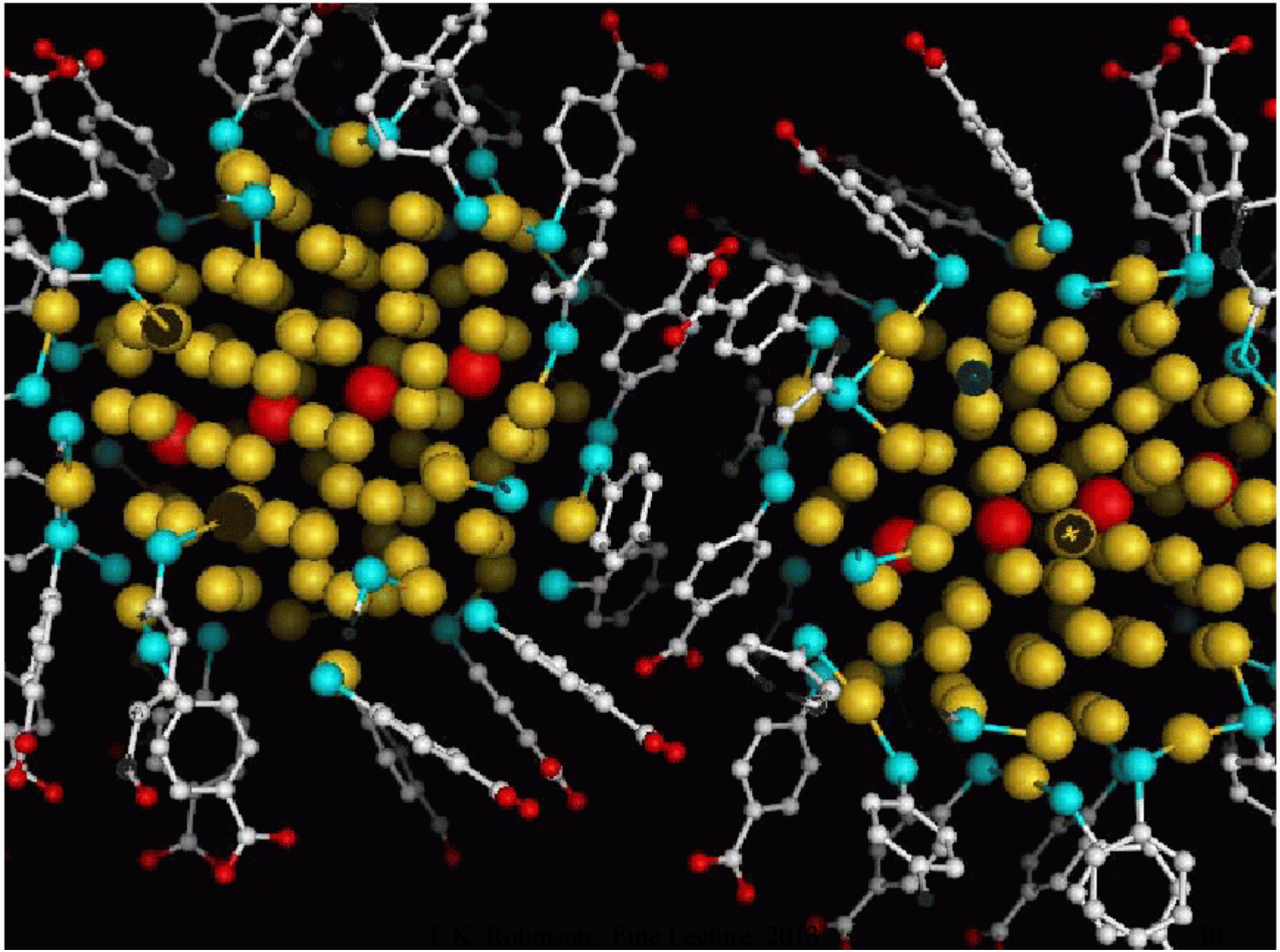


Gold-sulphur network in 1.6nm NC

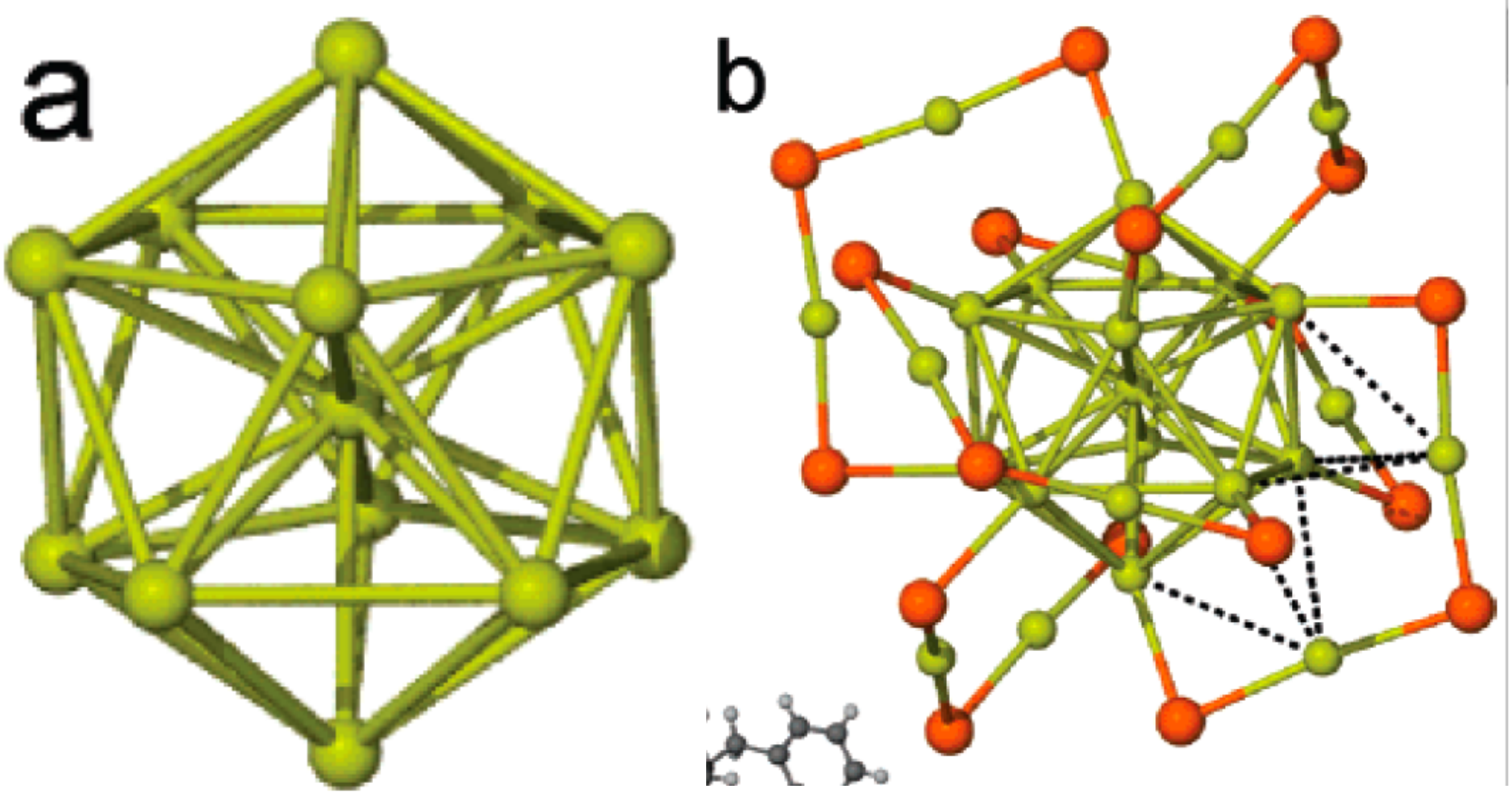
p-mercaptobenzoic acid (p-MBA)

P. D. Jadzinsky et al, Science 318, 430 (2007)



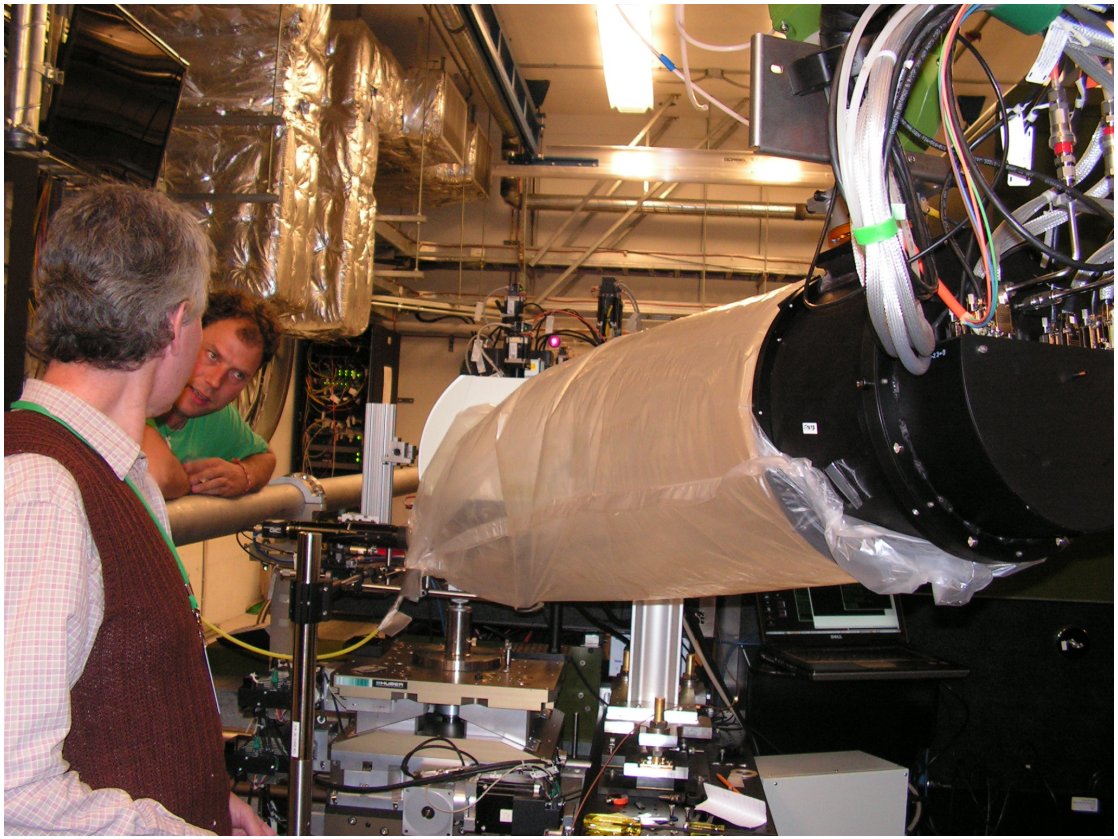


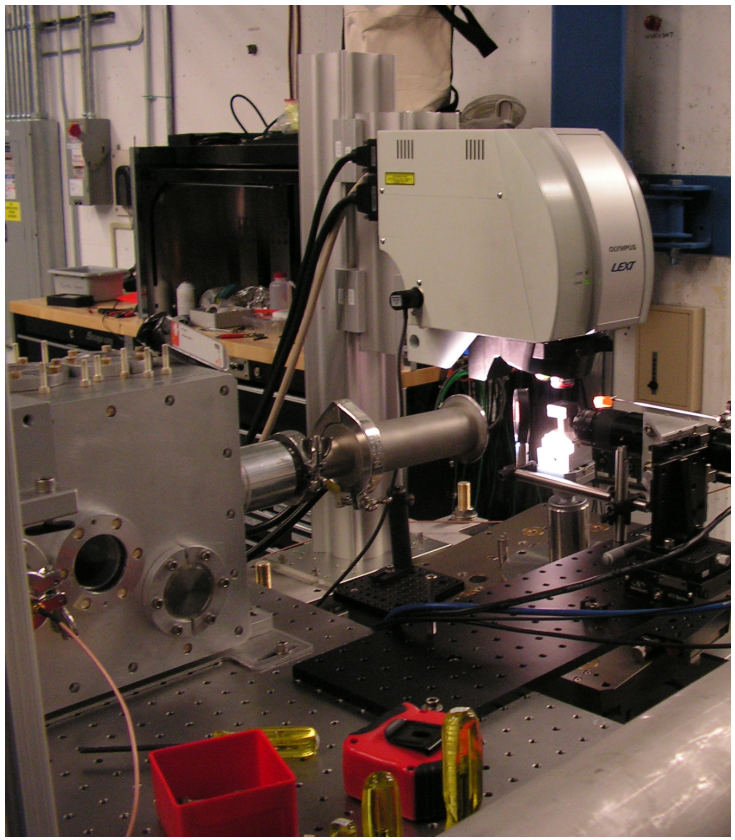
Gold-sulphur structure $Au_{13}(Au_2RS_3)_6$ $=Au_{25}RS_{18}$ Heaven et al, JACS 130 3755 (2008)



Pump-probe at LCLS (XPP)

Justin Wark, Loren Beitra, Alexander Korsunsky, Ross Harder, David Fritz ,
Sebastien Boutet, Jesse Clark, Garth Williams, Brian Abbey, Andy Higginbotham,
Diling Zhu, Henrick Lemke, Mattieu Chollet, Marc Messerschmidt

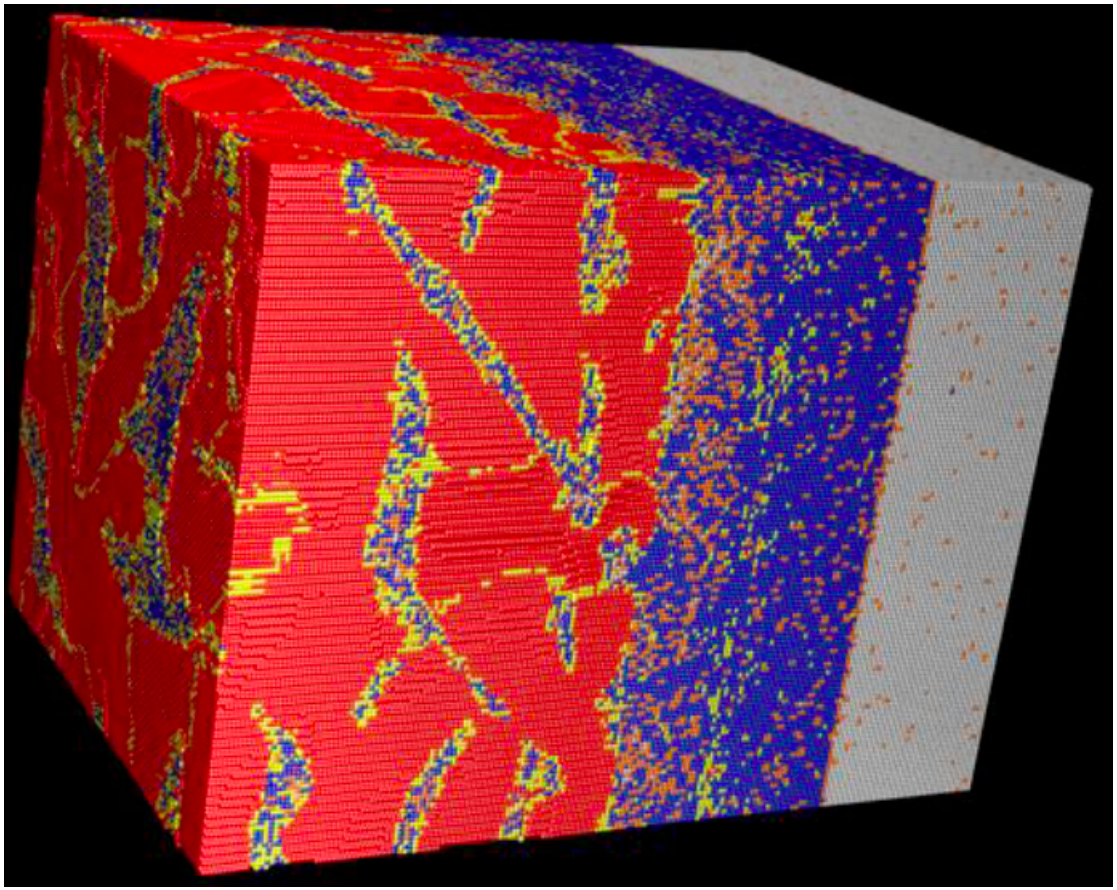




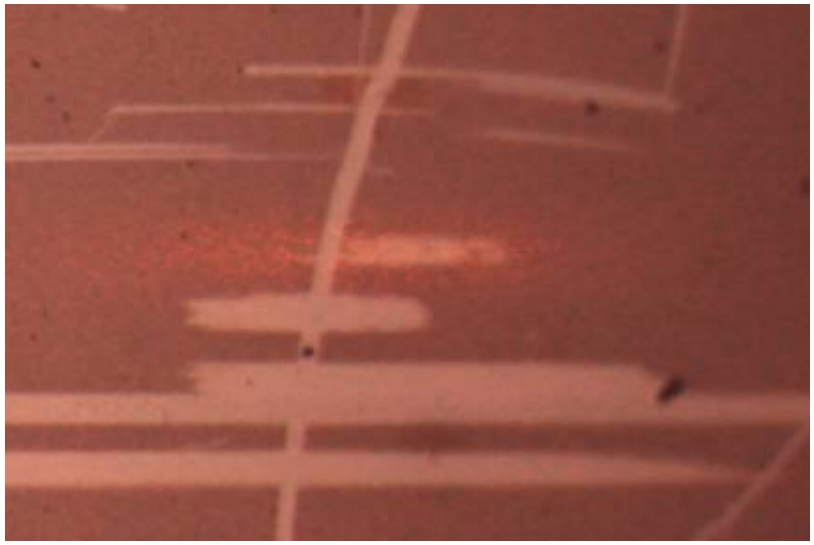
MD simulation of Shock Wave

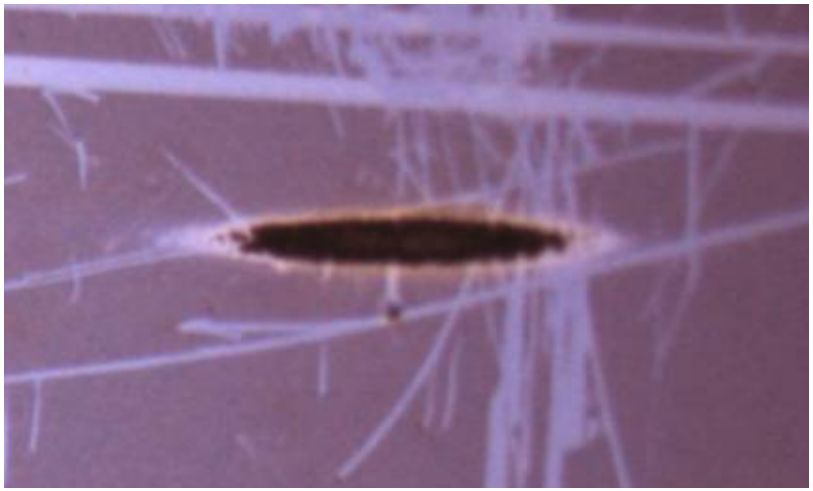
Damage in Fe along (001) direction

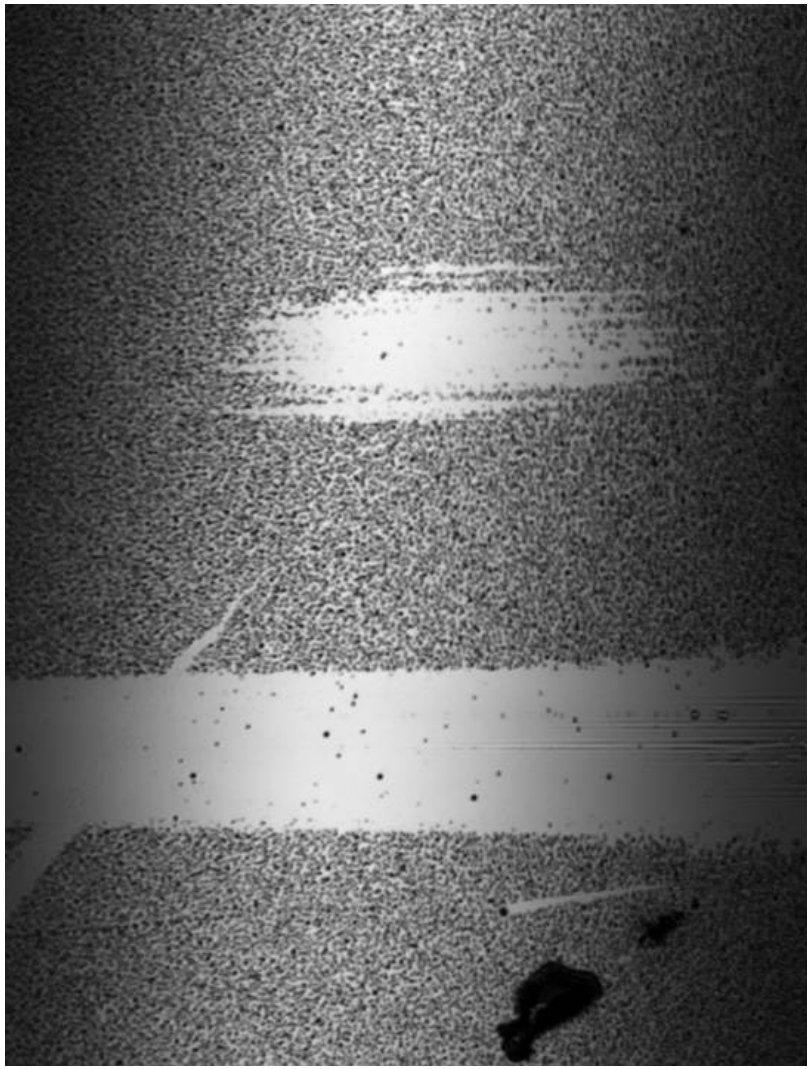
K Kadau, TC Germann, PS Lomdahl, and BL Holian. Science, 296 1681 2002



Visible and Confocal microscopy







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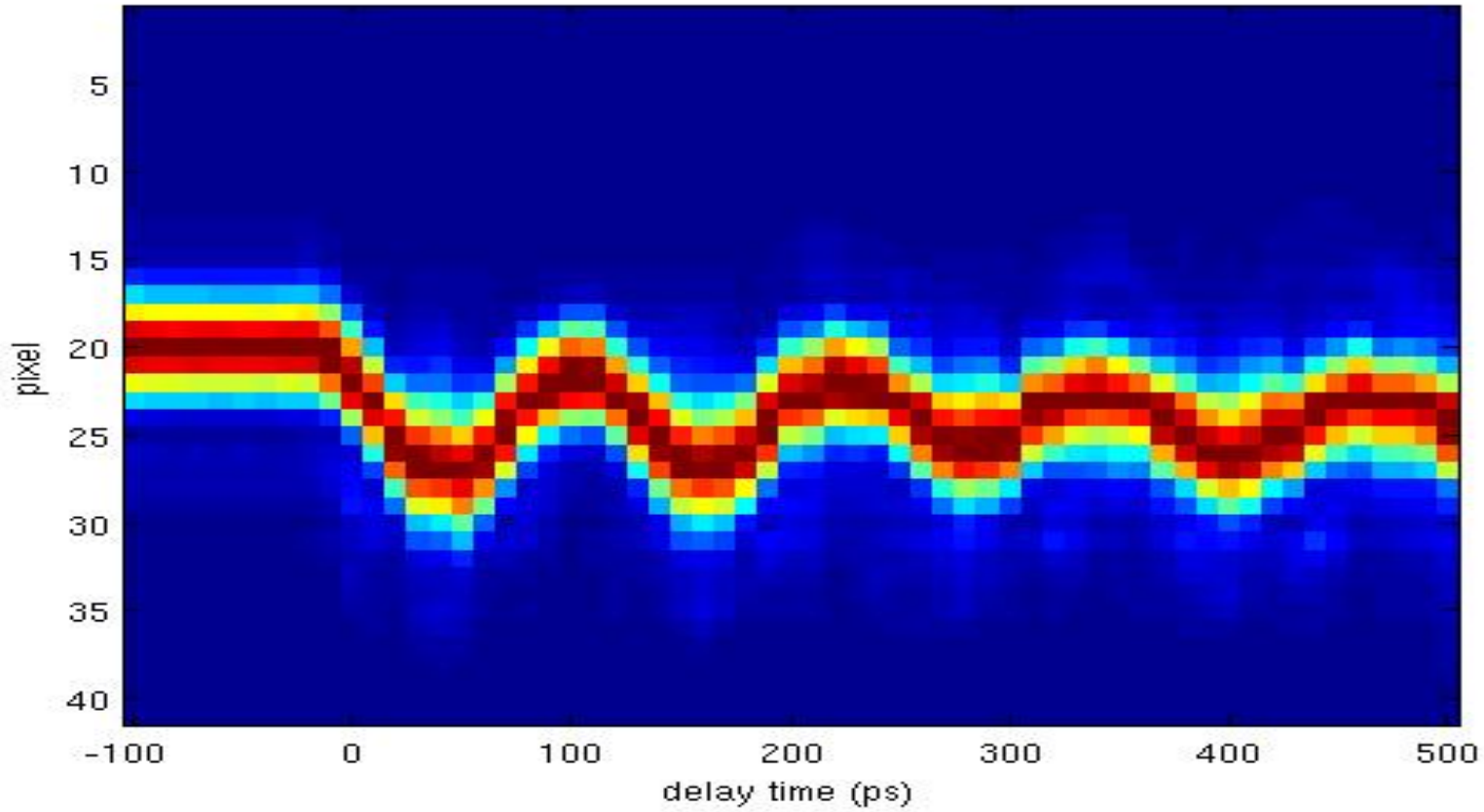
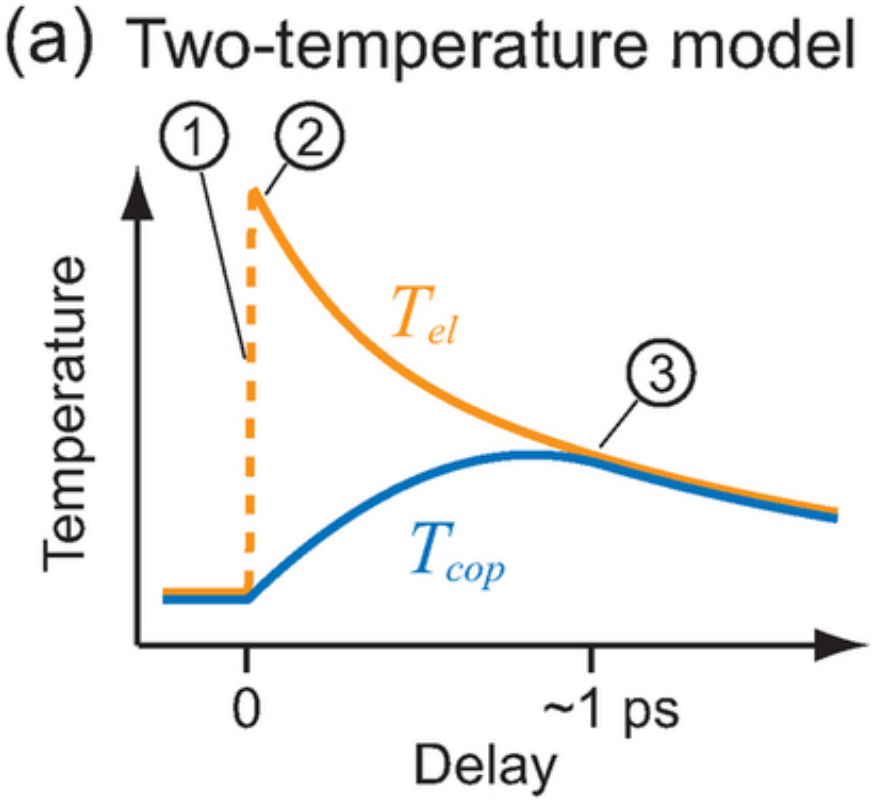
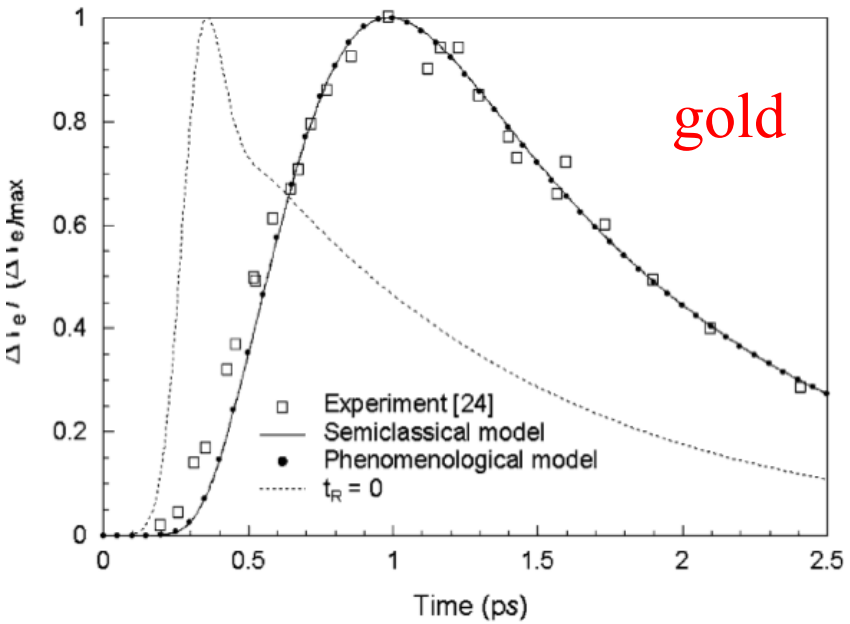


"Two-temperature" model

Y. Ishida et al, Nature Scientific Reports 1 64 (2011)

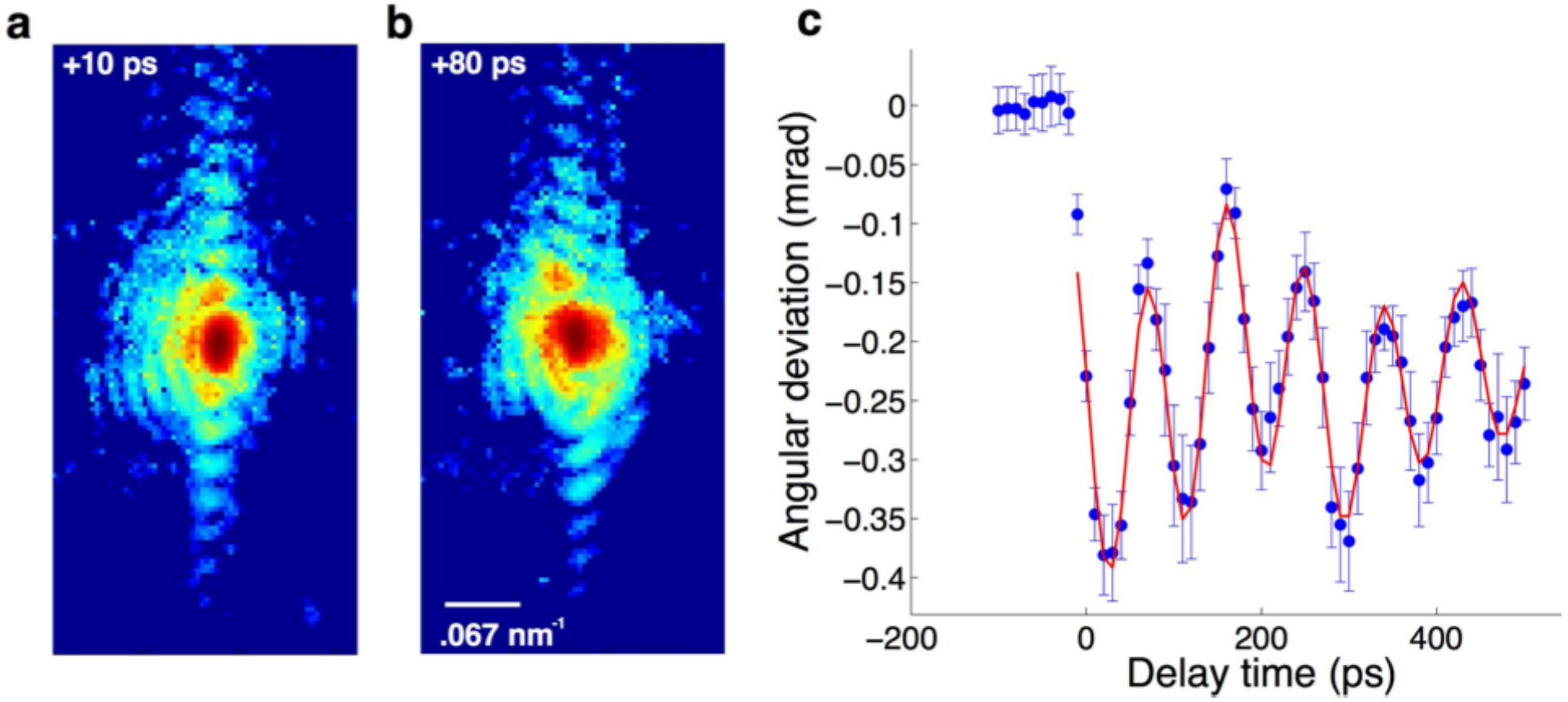
J.K. Chen et al, Int J. Heat Transfer 49 307 (2006)





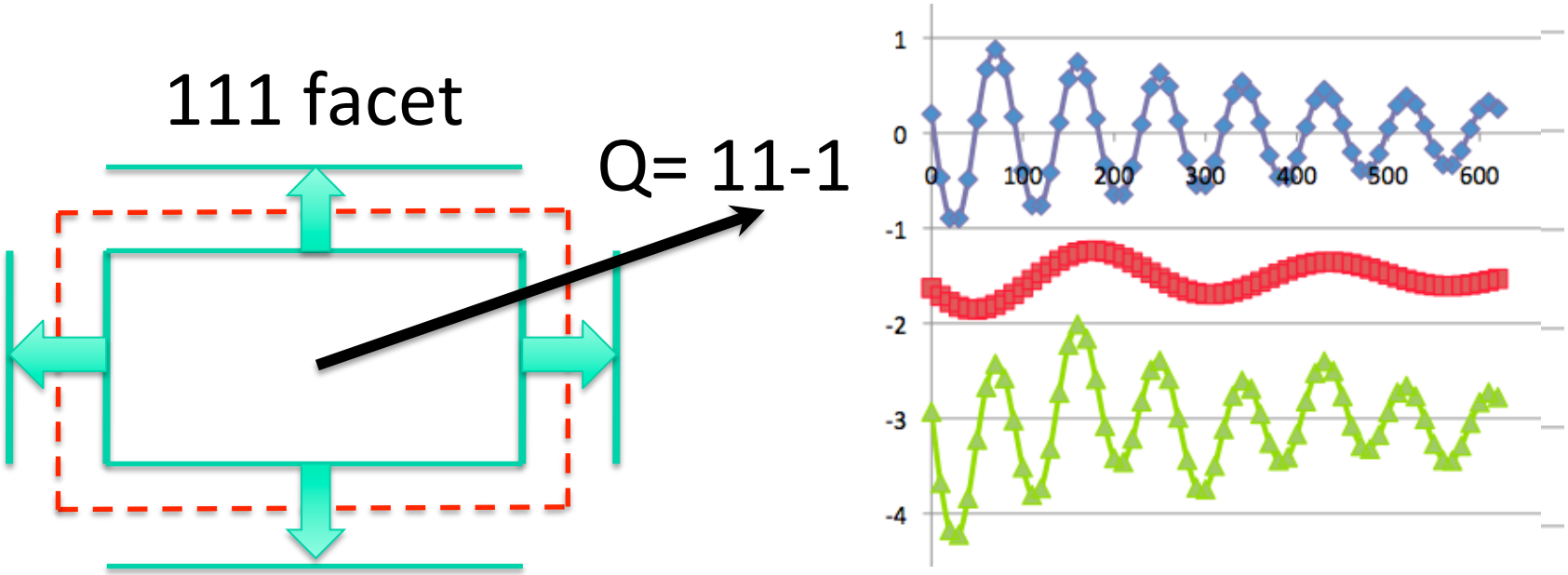
ig. 2. Comparison of the change in electron temperature at
e front surface of an 80-nm gold film irradiated by a 2.8 mJ/
n^2, 800 nm, 150-fs laser pulse.

Time resolved Bragg peak position



Two Normal Modes of Vibration

$$S(\tau) = \sum_{n=1}^{N} A_n \exp\left[-(\tau/\tau_{d,n})^2\right] \cos(\omega_n \tau + \varphi_{0,n})$$



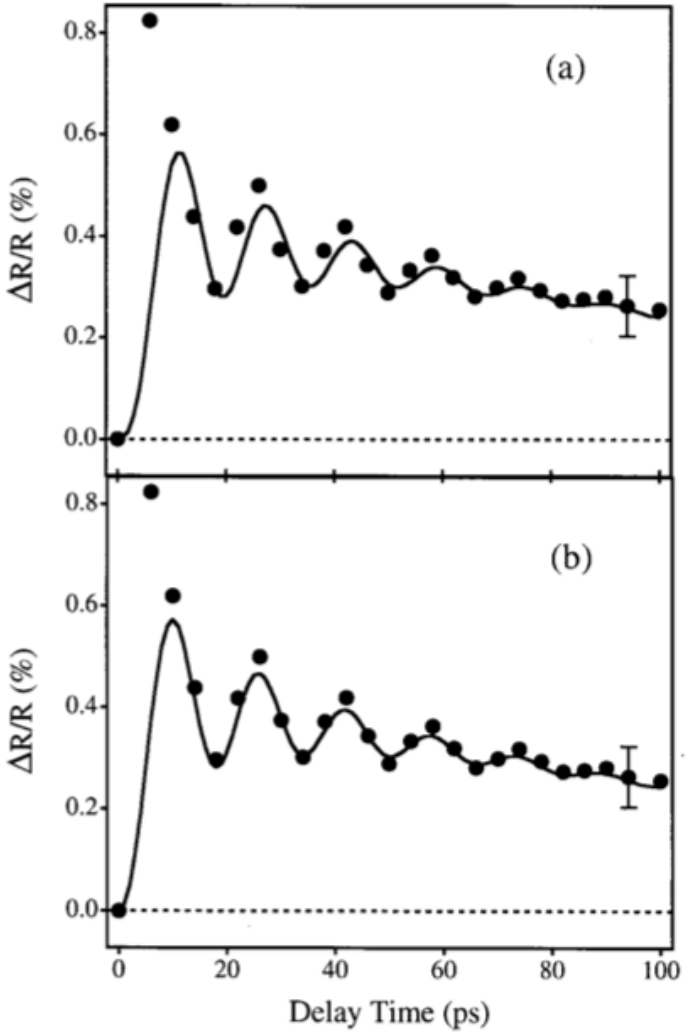
T_1 = 90ps h_1 = 145nm c_s = 3240 m/s

T_2 = 259ps h_2 = 420nm

Ultrafast Absorption Spectroscopy

24nm Au nanoparticles in H_2O, converted to radius

G. V. Hartland, J. Chem. Phys. 116, 8048 (2002)

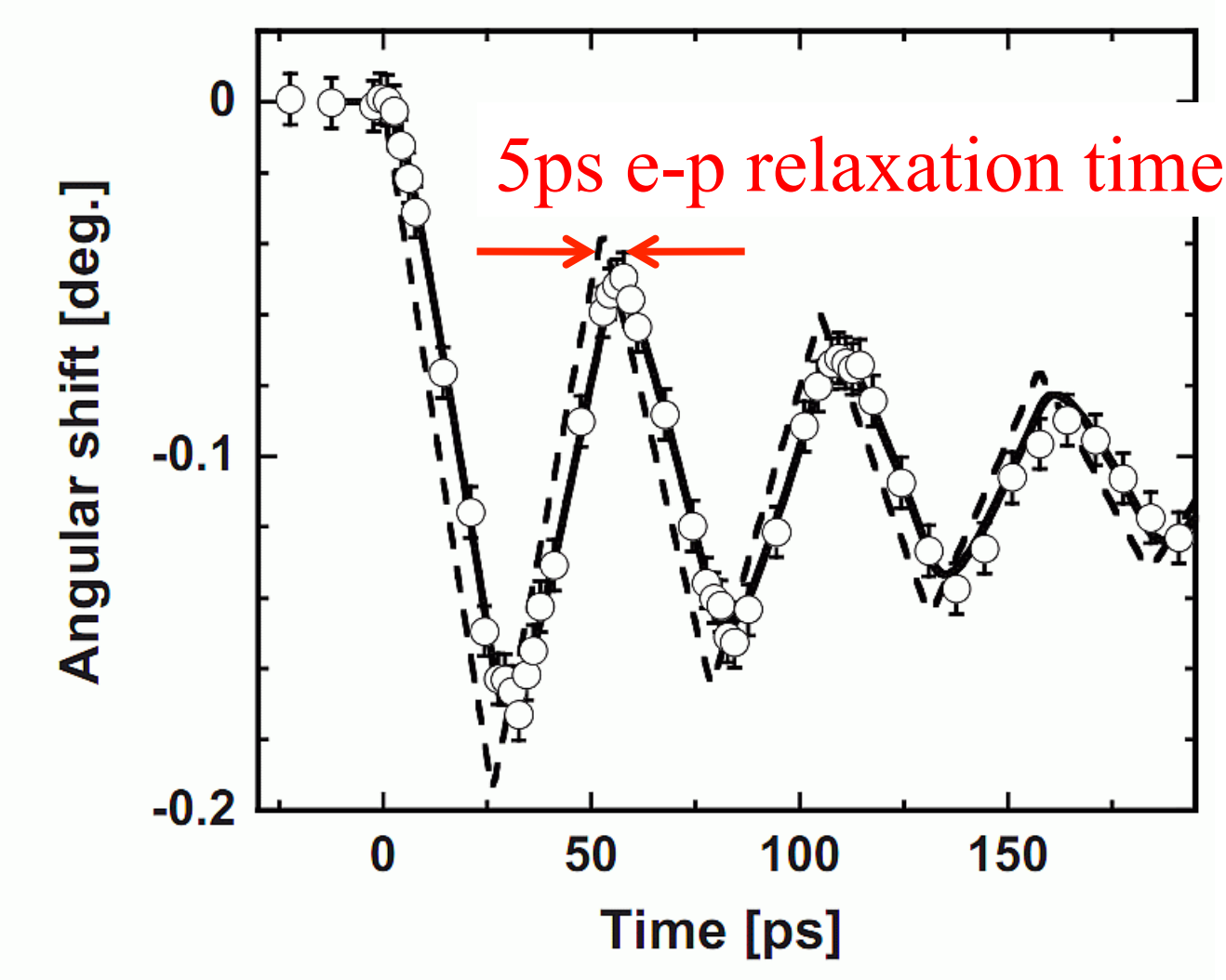


(a) Simple normal mode analysis with impulse heating.

(b) Corrected by adding 2T model and electronic contribution to thermal expansion coefficient.

Plasma Source on 90nm Au film

M. Nicoul et al, APL 98 191902 (2011)

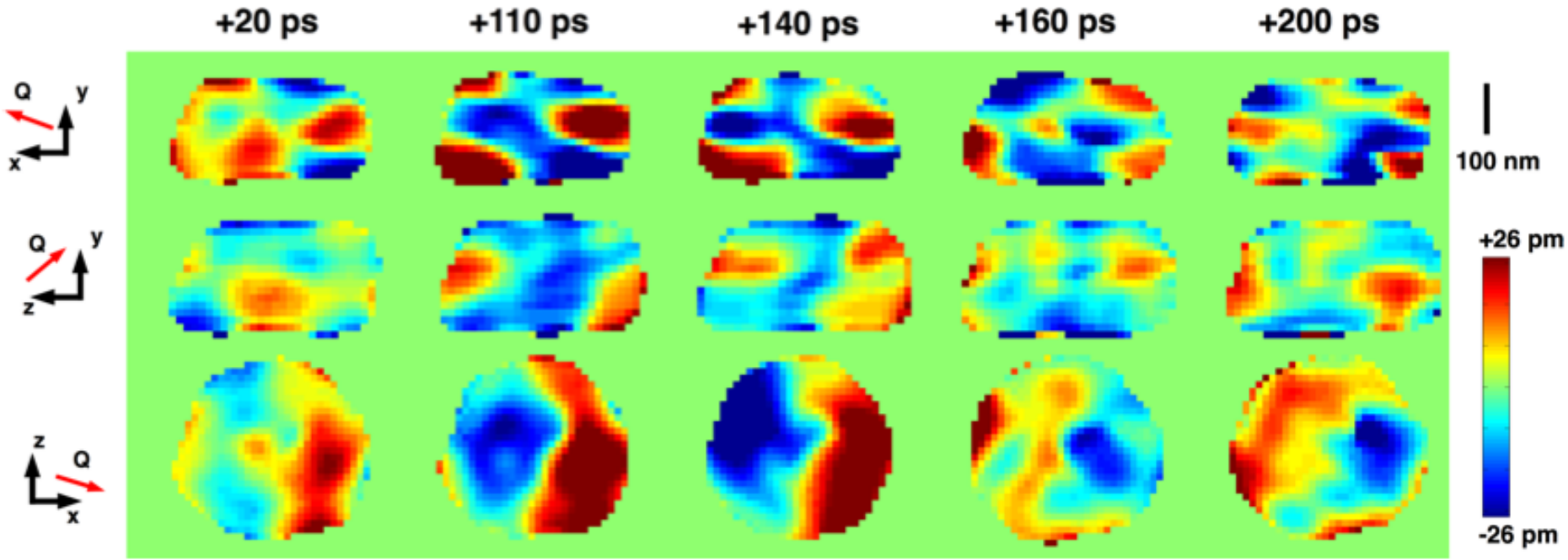


Dynamic imaging of displacements

CDI inversion of 3D diffraction patterns

1000 frames averaged at each point of rocking curve

Jesse Clark et al, Science 341 56 (2013)

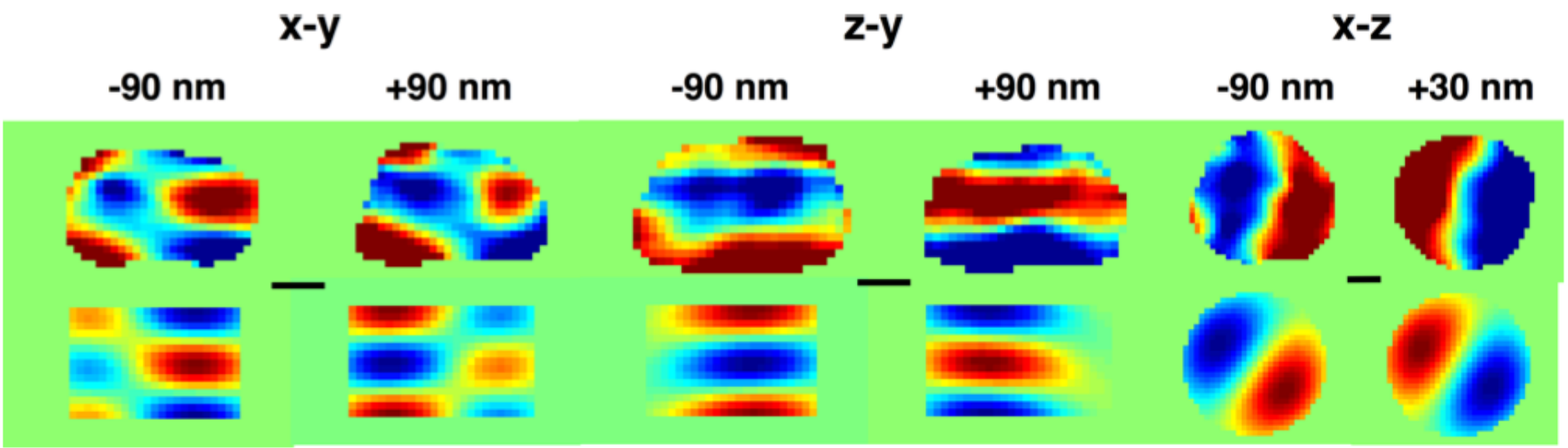


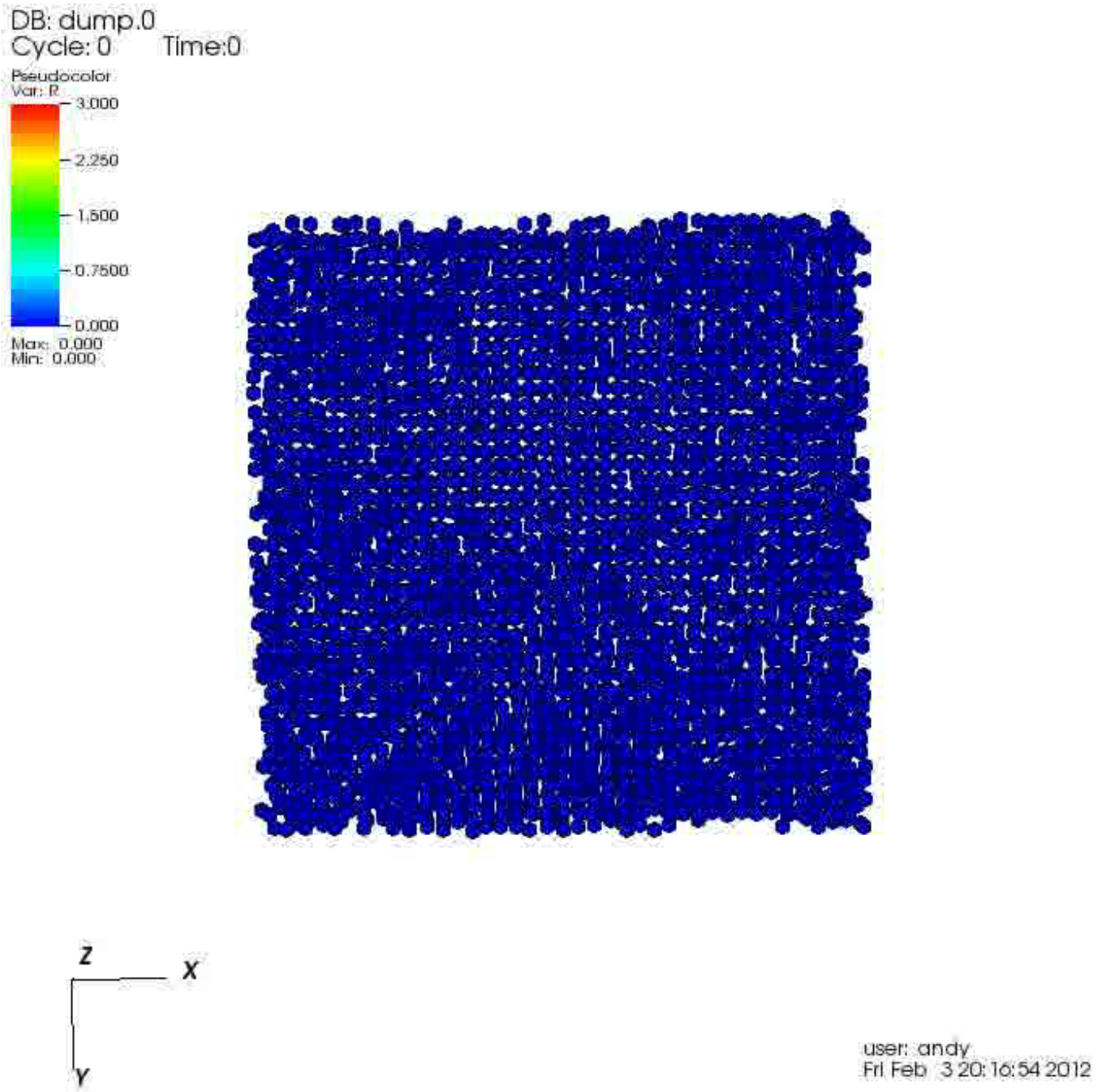
Dynamic imaging of displacements

CDI inversion of 3D diffraction patterns

Comparison with (1,1) normal mode of cylinder

Jesse Clark et al, Science 341 56 (2013)

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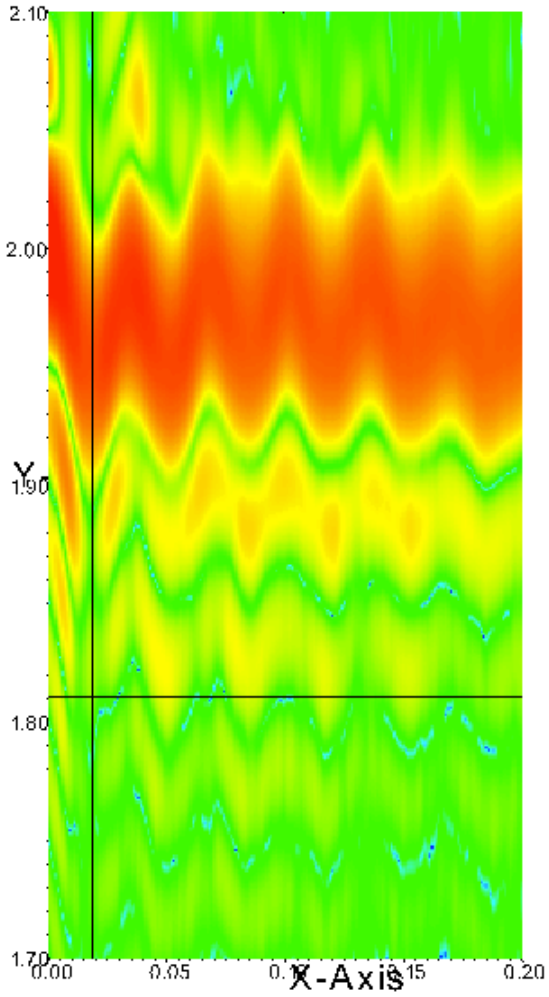
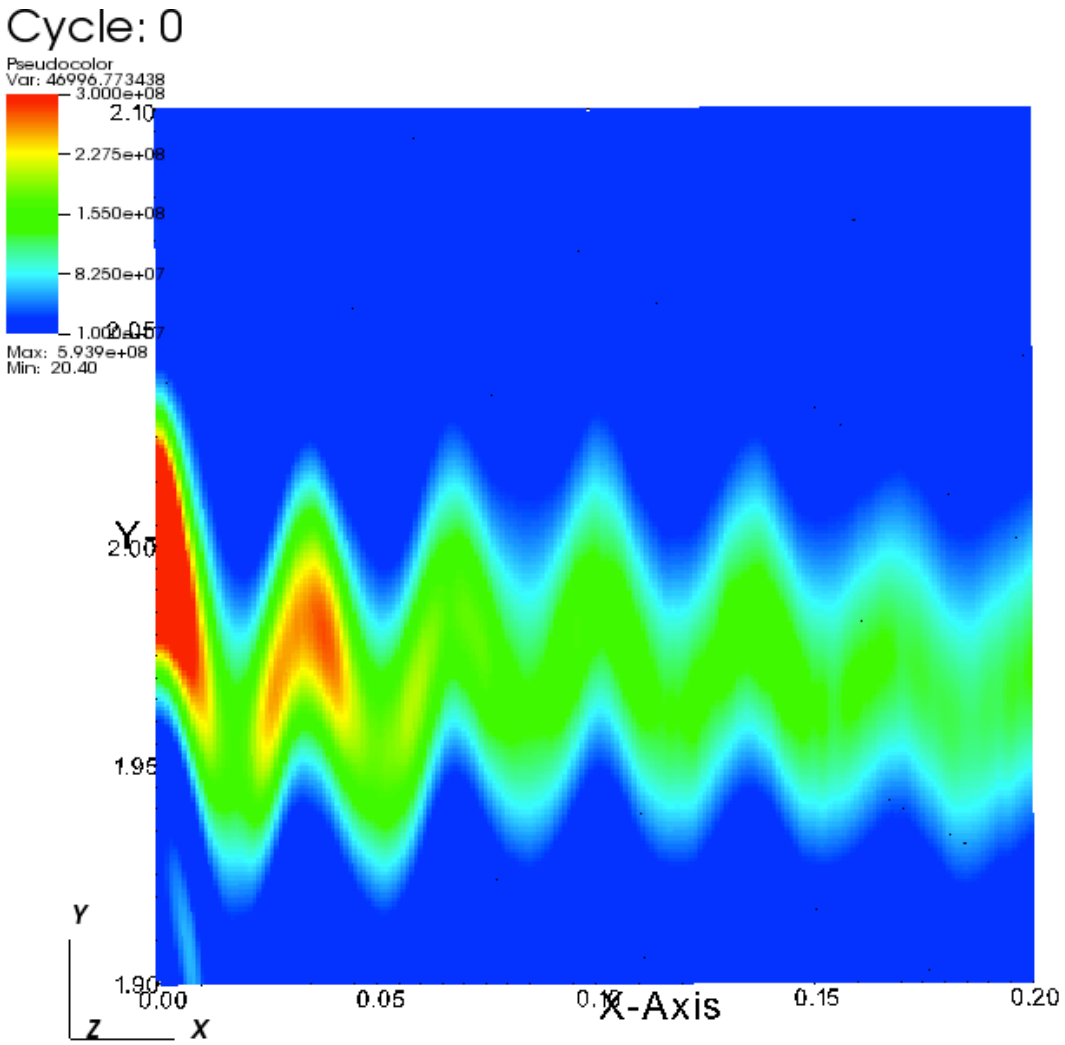
Fri Feb 3 20:16:54 2012

MD Simulation (LAMMPS)

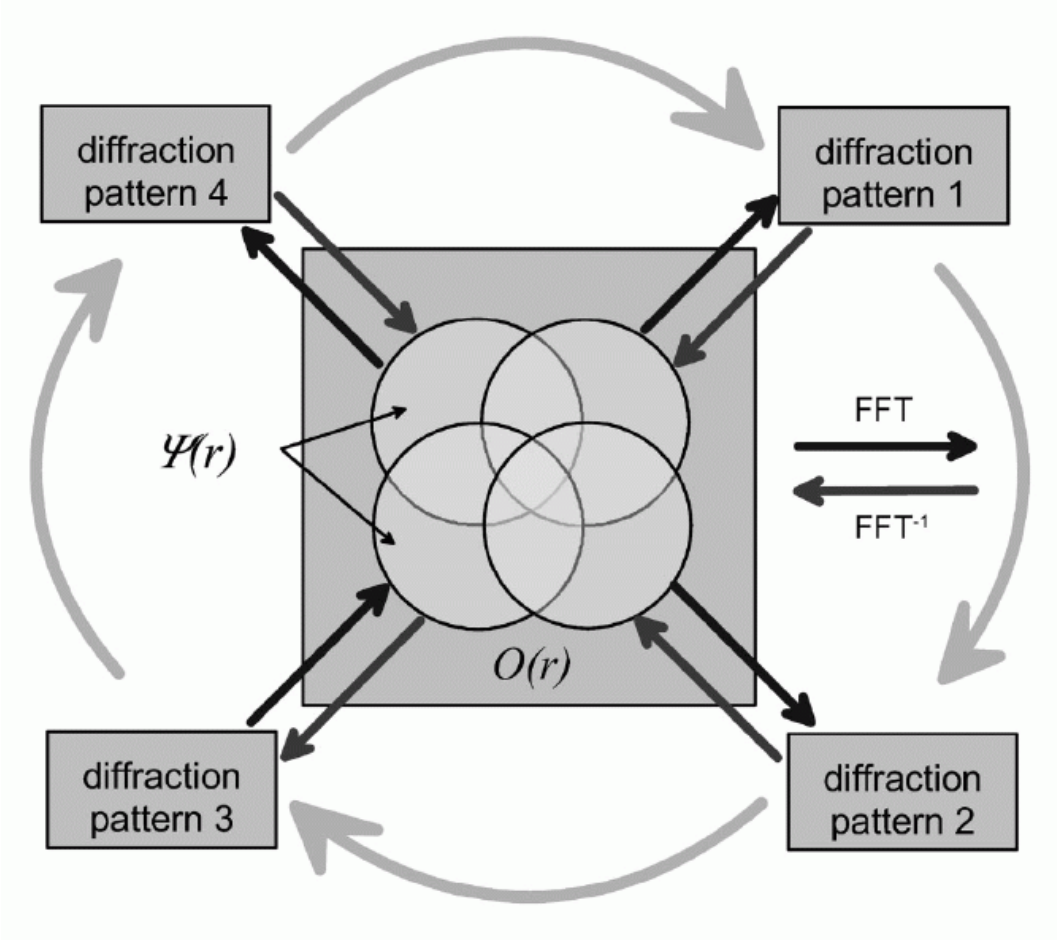
Andy Higginbotham and Loren Beitra





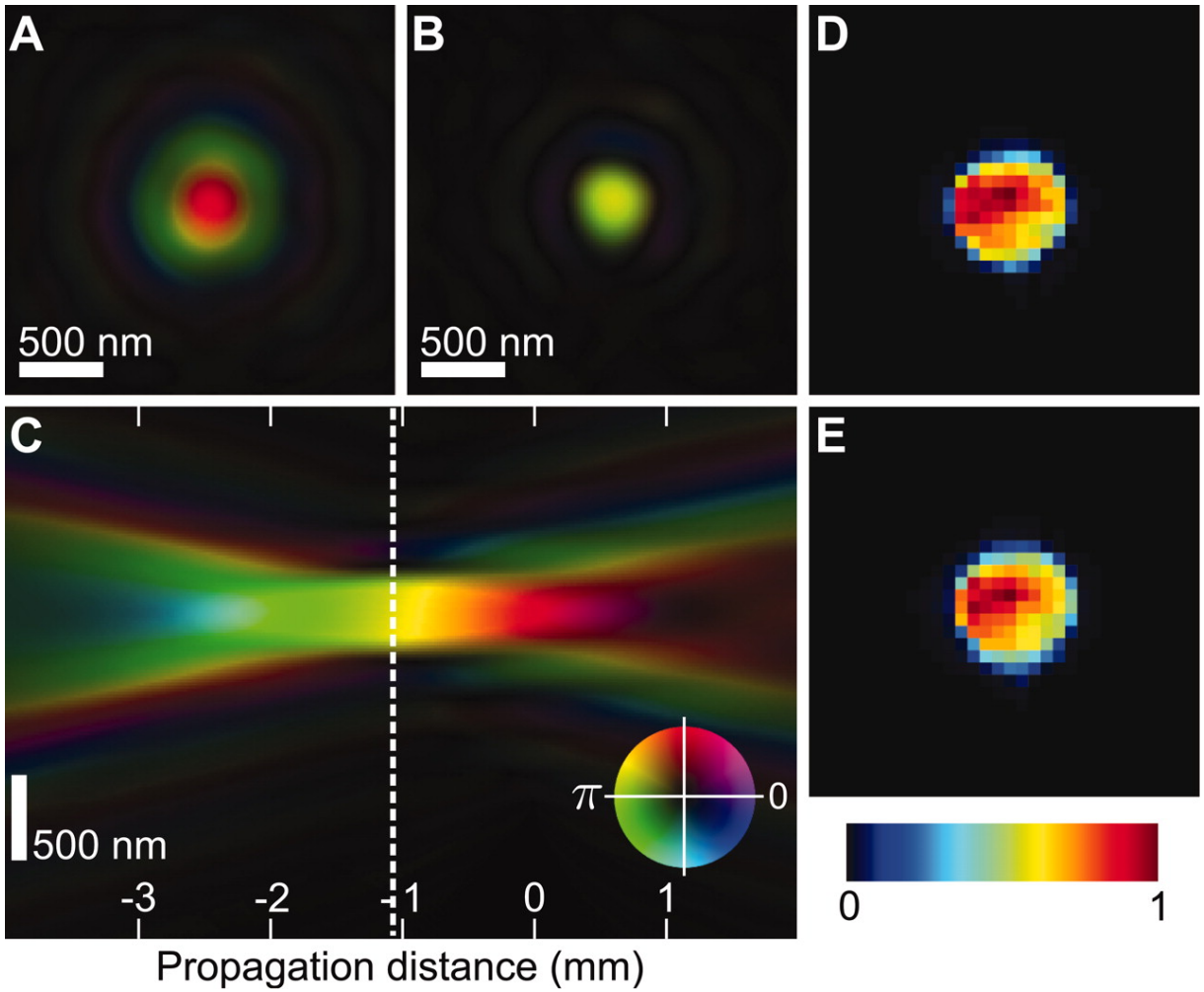
X-ray Ptychography

J. Rodenburg et al, PRL 98, 034801 (2007)



Reconstruction of Probe

P. Thibault et al, Science 321 379 (2008)



Solution of the Phase Problem in the Theory of Structure Determination of Crystals from X-Ray Diffraction Experiments

Emil Wolf*

Department of Physics and Astronomy and the Institute of Optics, University of Rochester, Rochester, New York 14627, USA

(Received 6 May 2009; published 10 August 2009)

We present a solution to a long-standing basic problem encountered in the theory of structure determination of crystalline media from x-ray diffraction experiments; namely, the problem of determining phases of the diffracted beams.

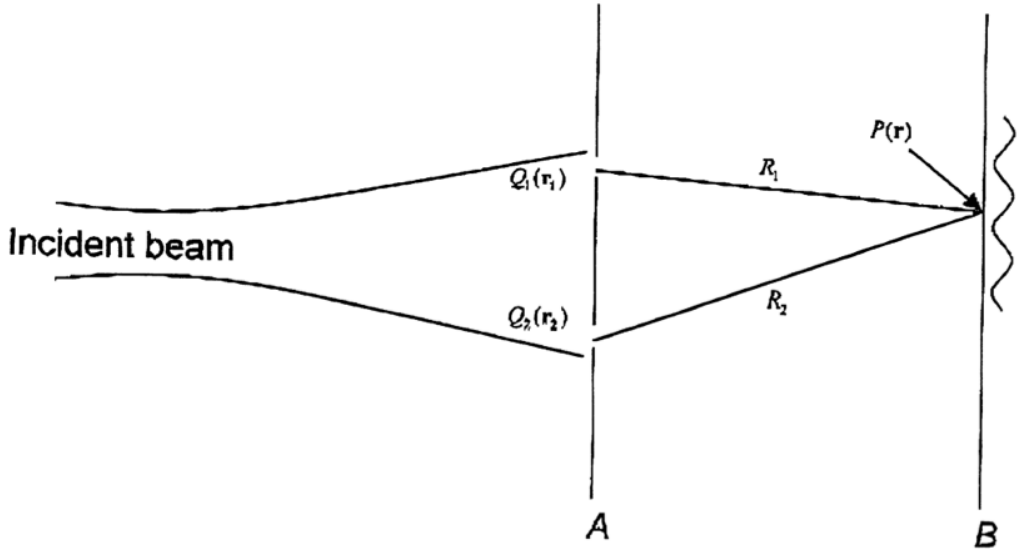
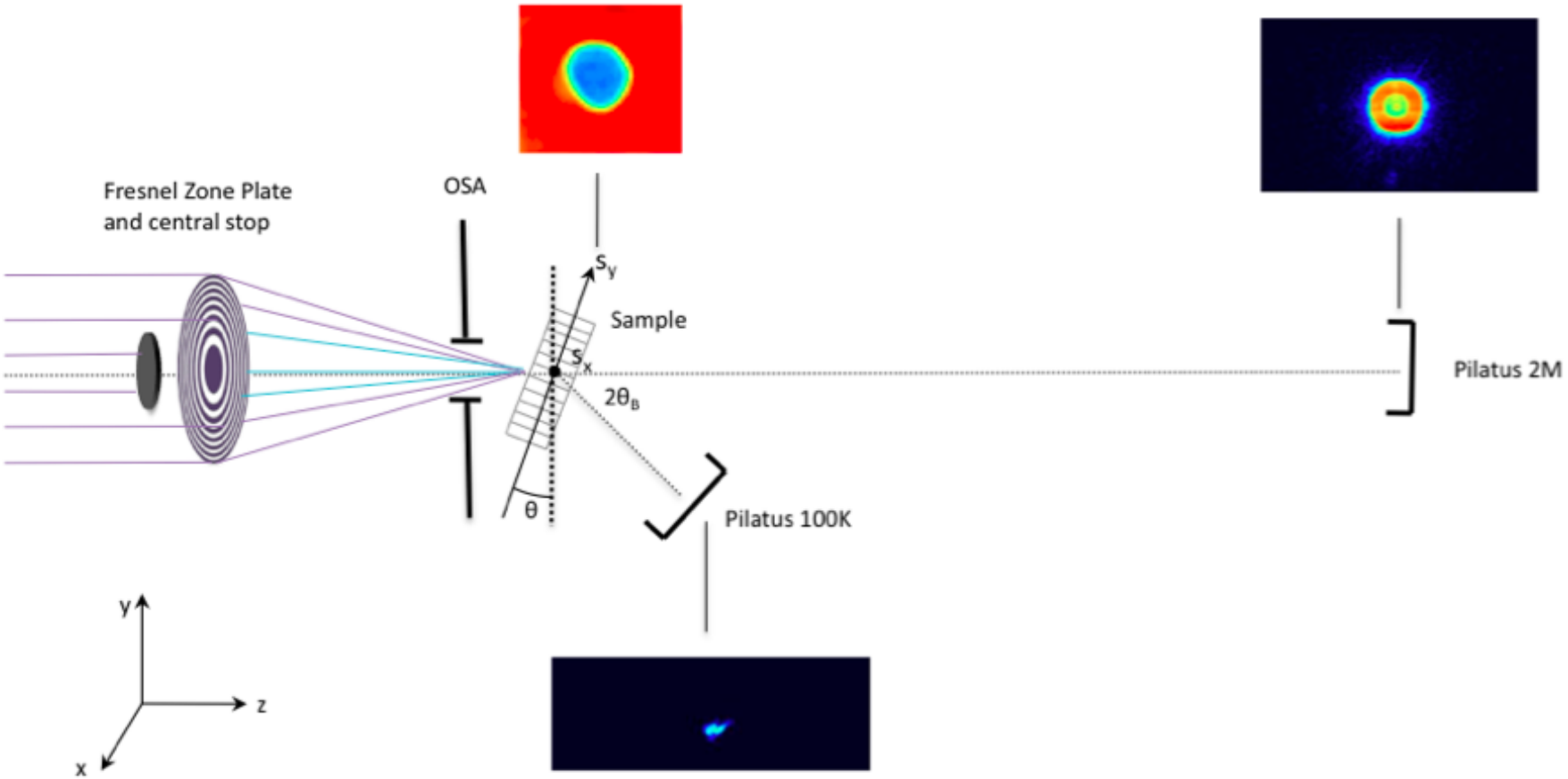


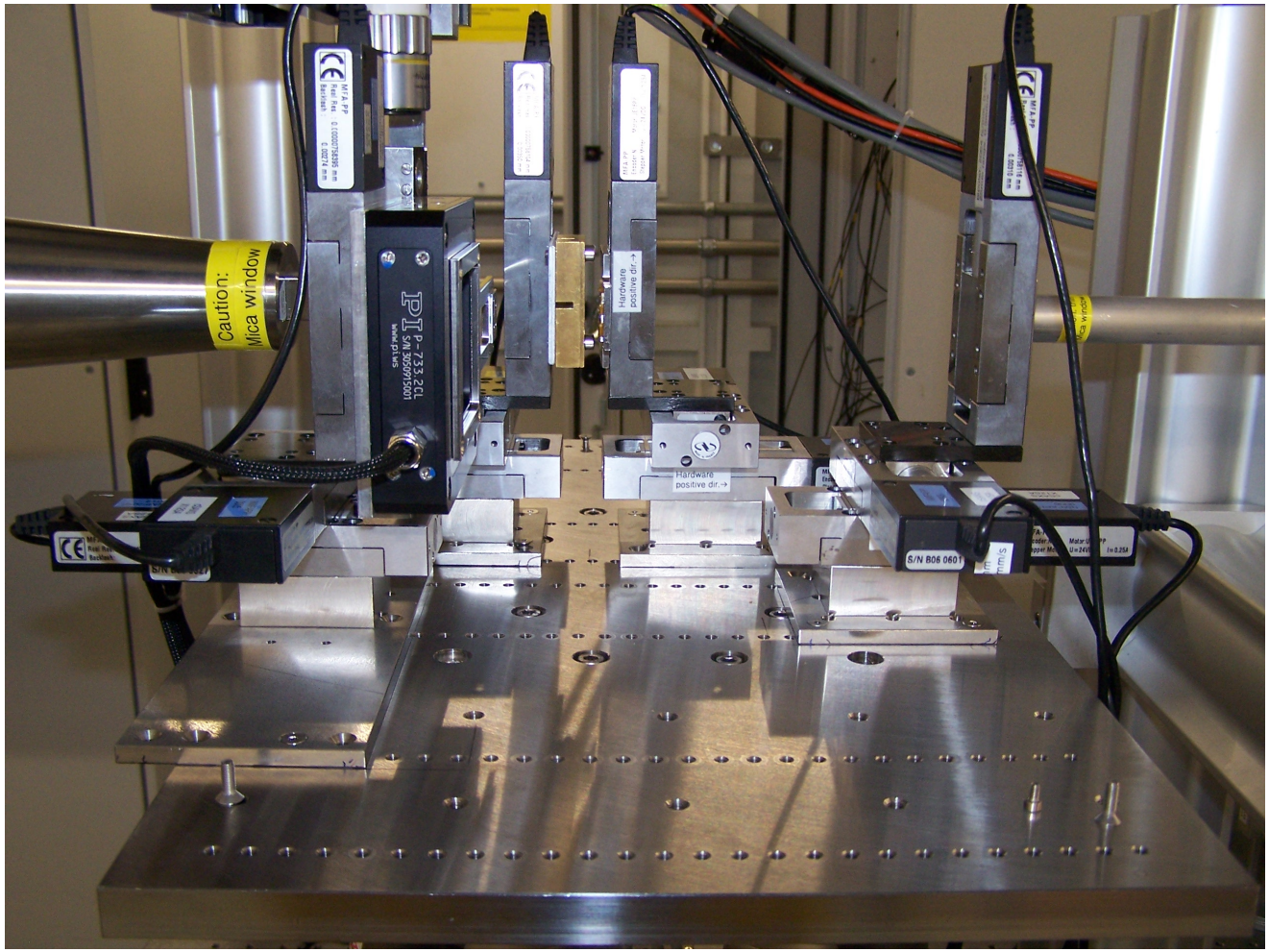
FIG. 1. Illustrating notation relating to Young's interference experiment.

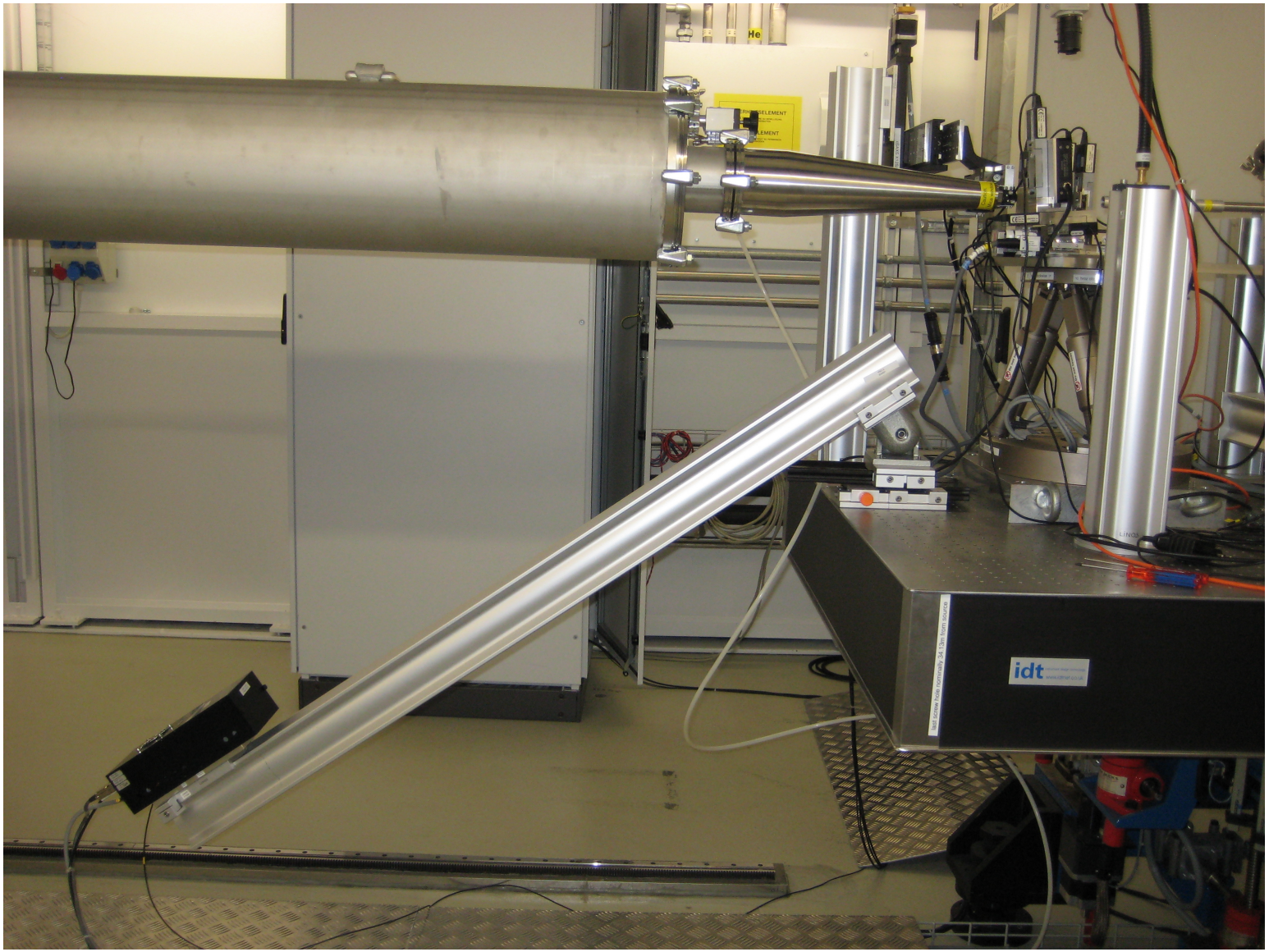
Ptychography of Gold Nanocrystals

Maria Civita, UCL, Ana Diaz, Swiss Light Source



Swiss Light Source cSAXS beamline

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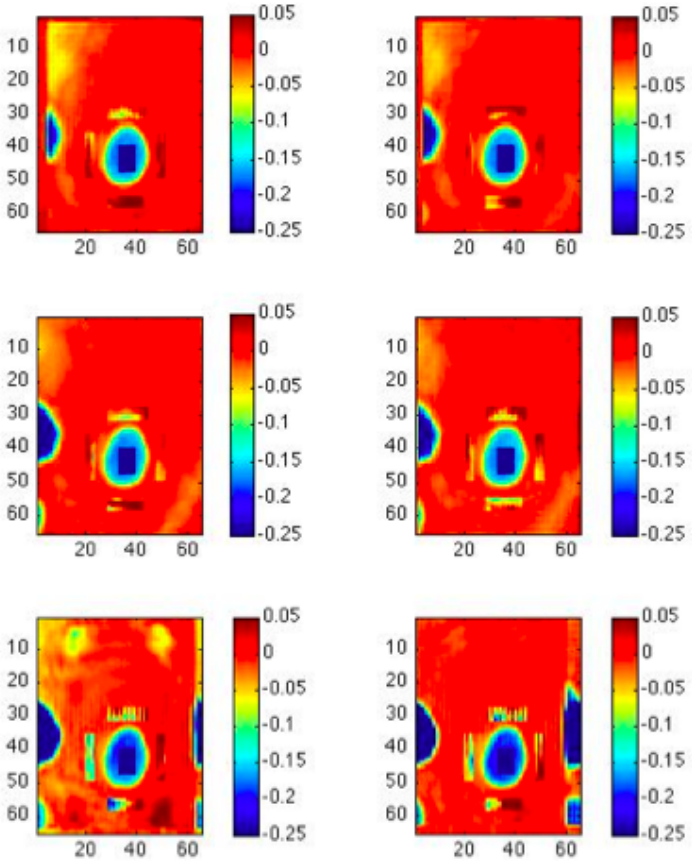
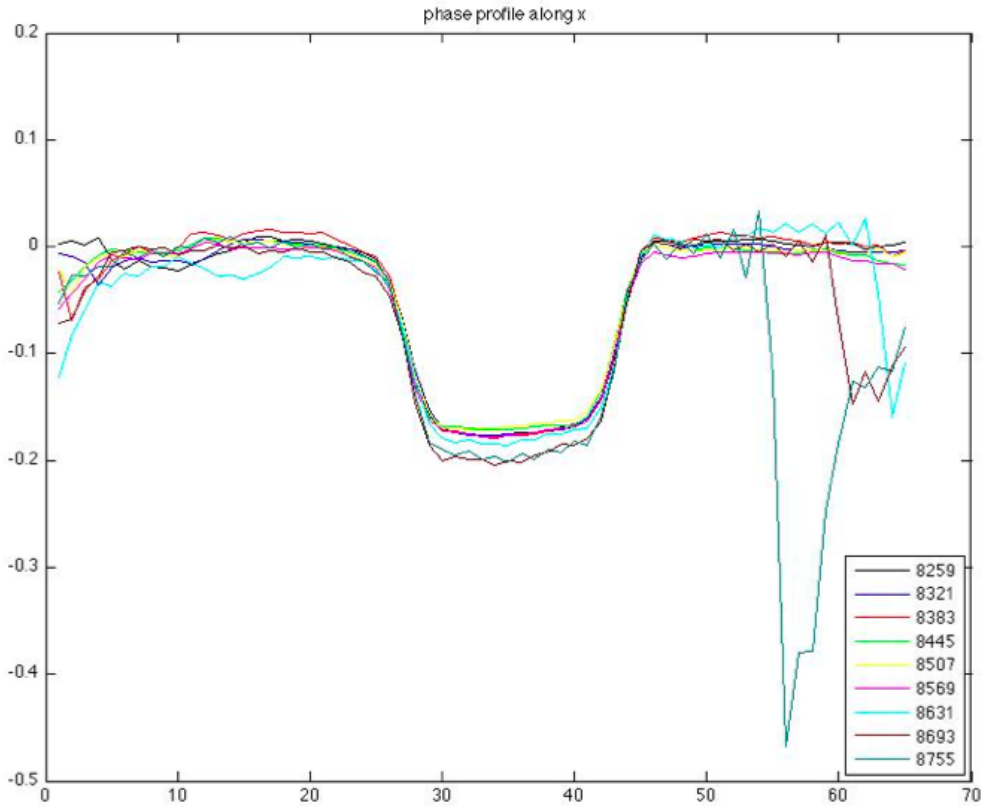
Ptychography of Gold Nanocrystals

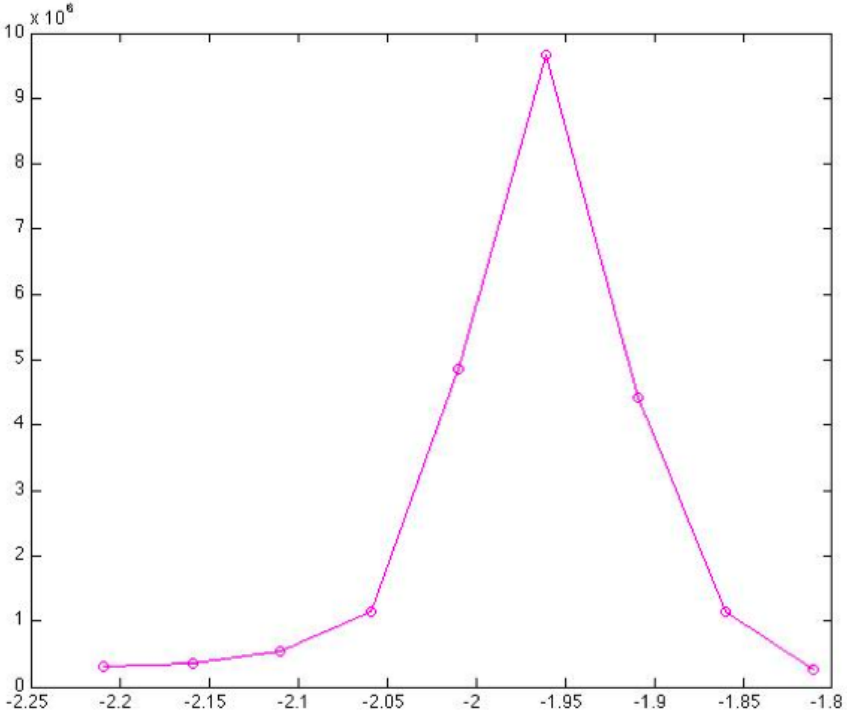
Maria Civita, UCL, Ana Diaz, Swiss Light Source

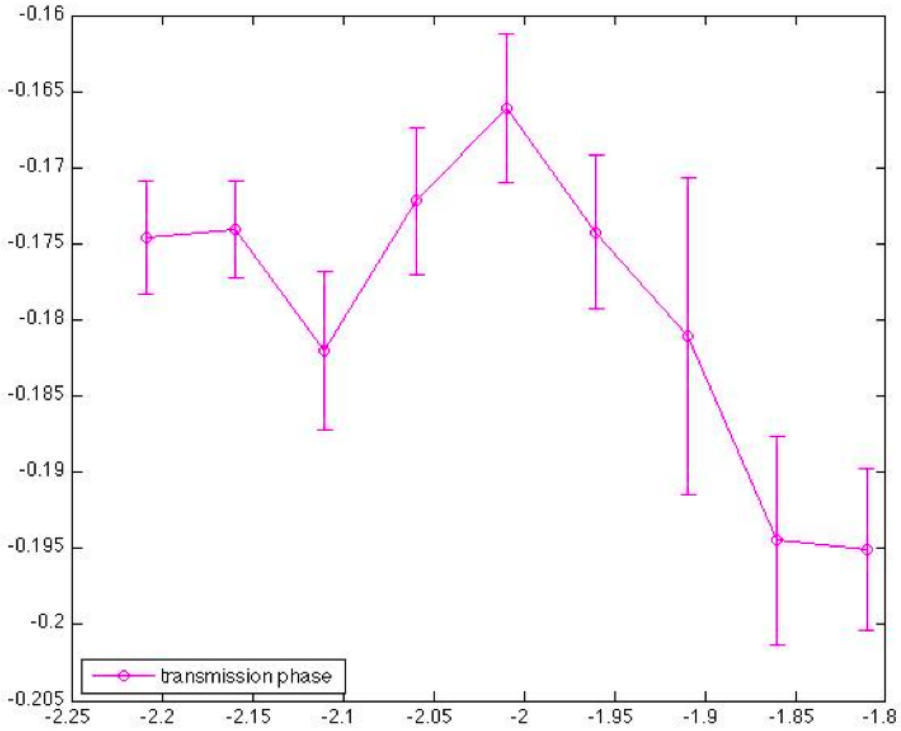




Transmitted Phase of Gold Nanocrystals

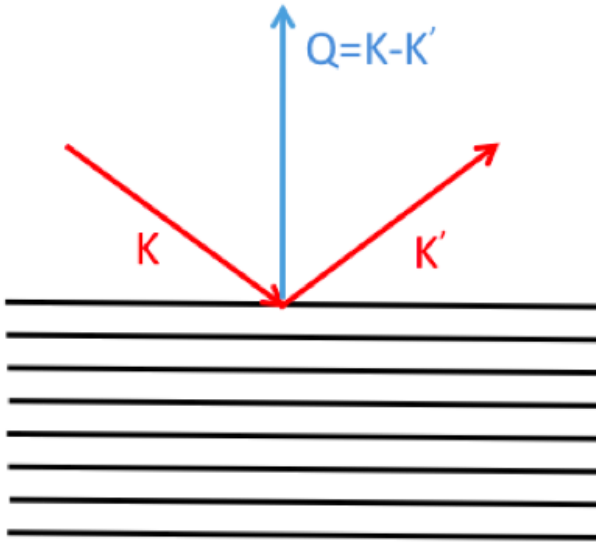
Maria Civita, UCL, Ana Diaz, Swiss Light Source

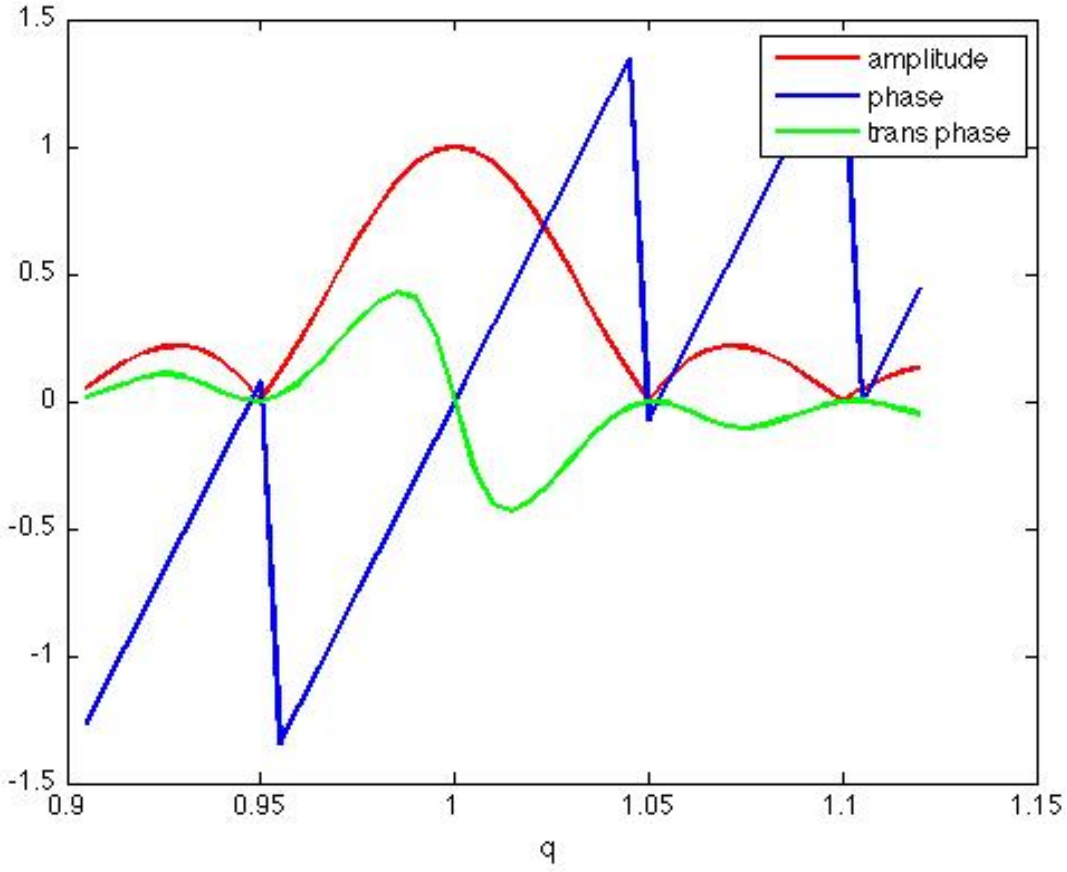




Diffraction from a Finite Slab

Als-Nielsen and McMorrow, "Modern X-ray Physics"





Diffraction from a Finite Slab

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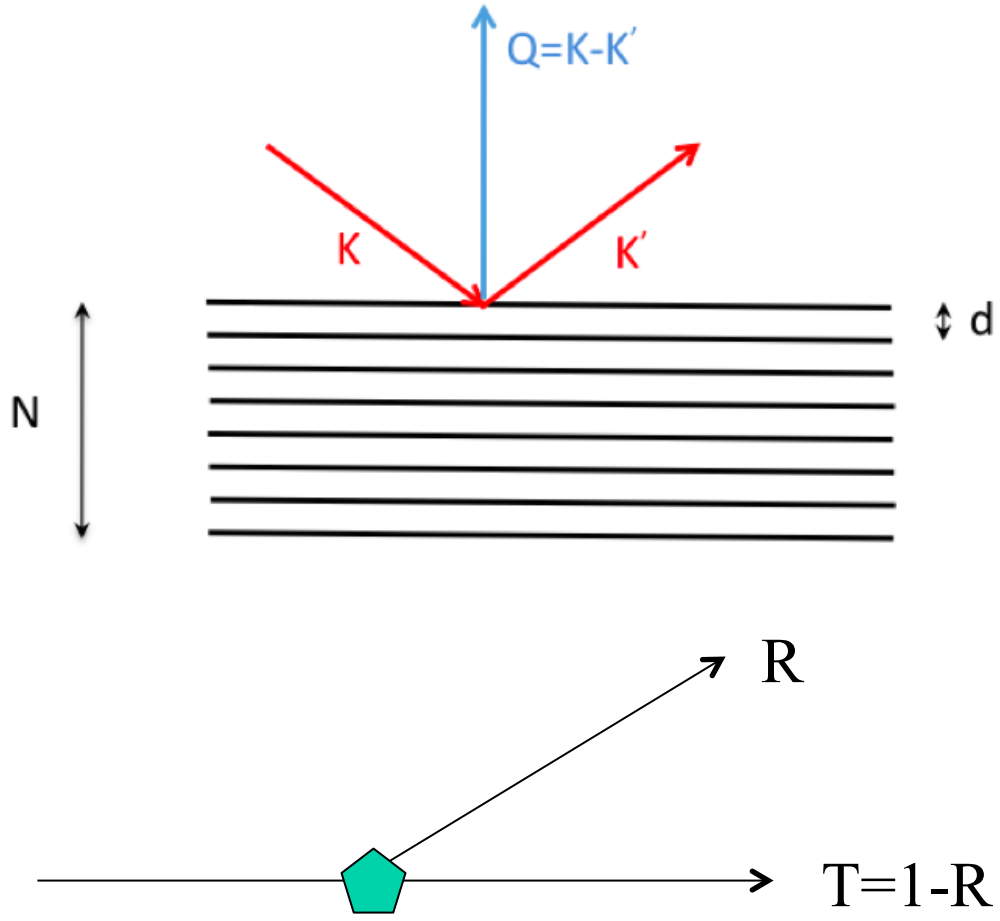
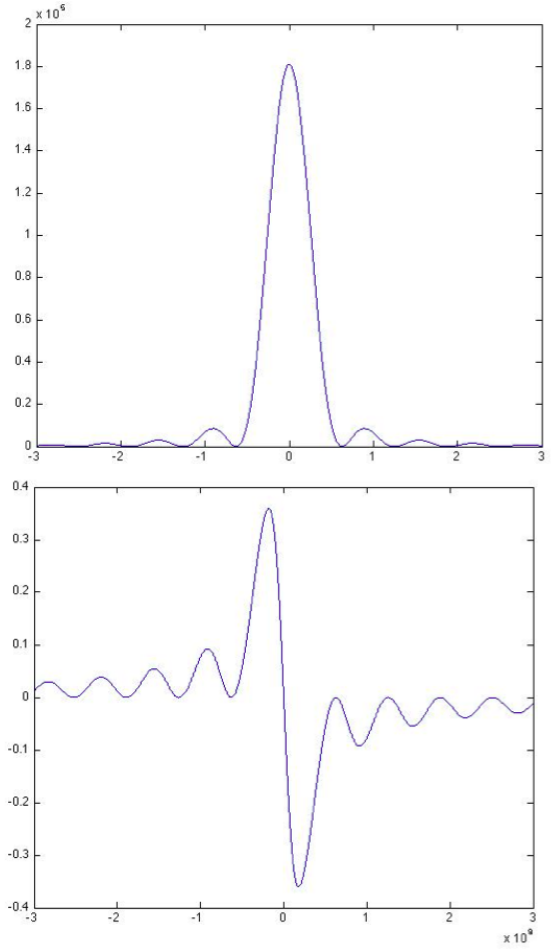
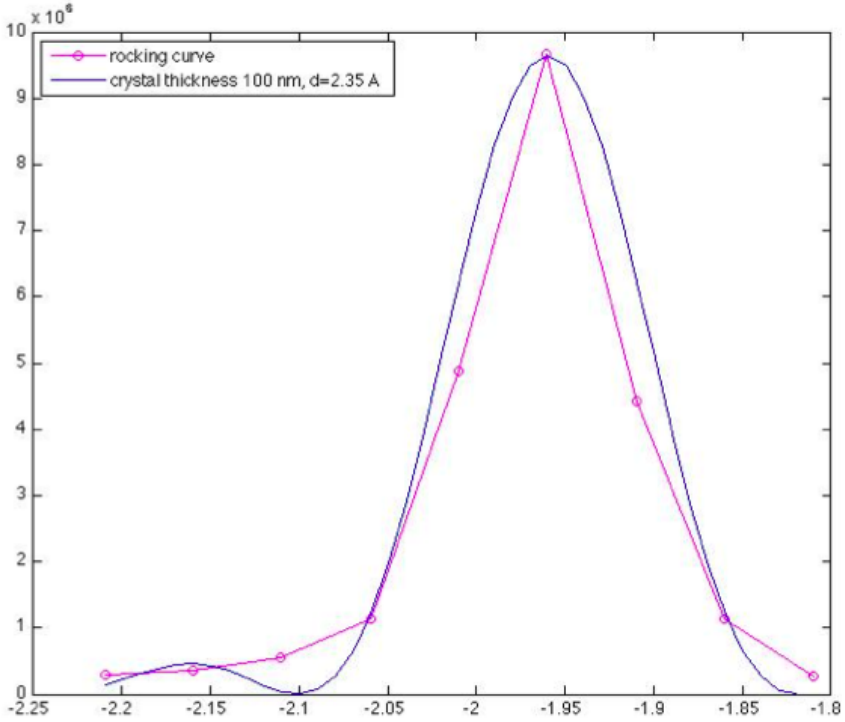
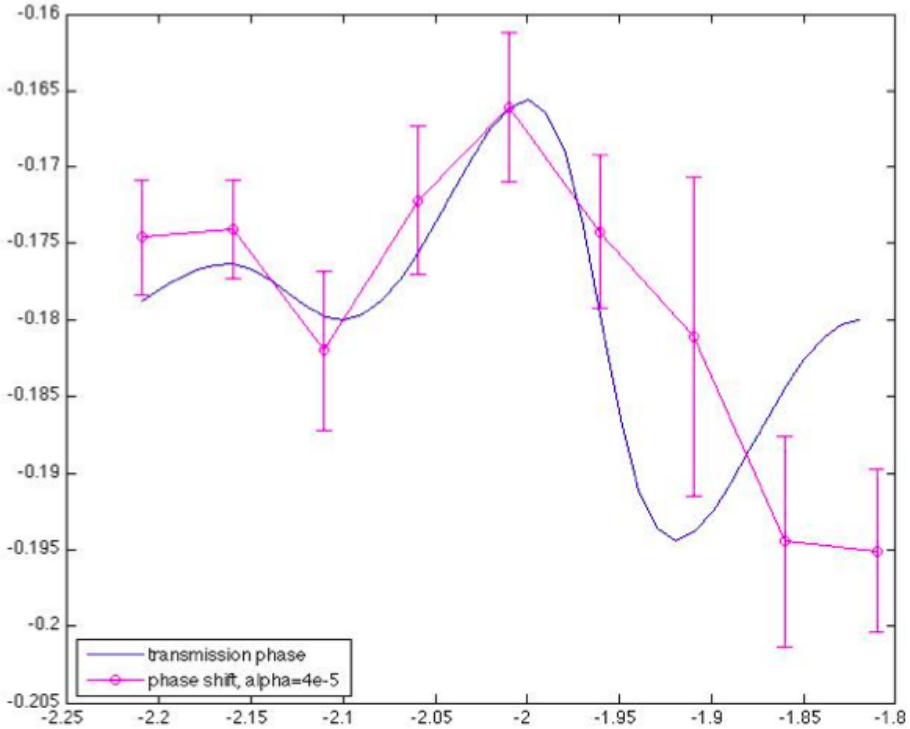




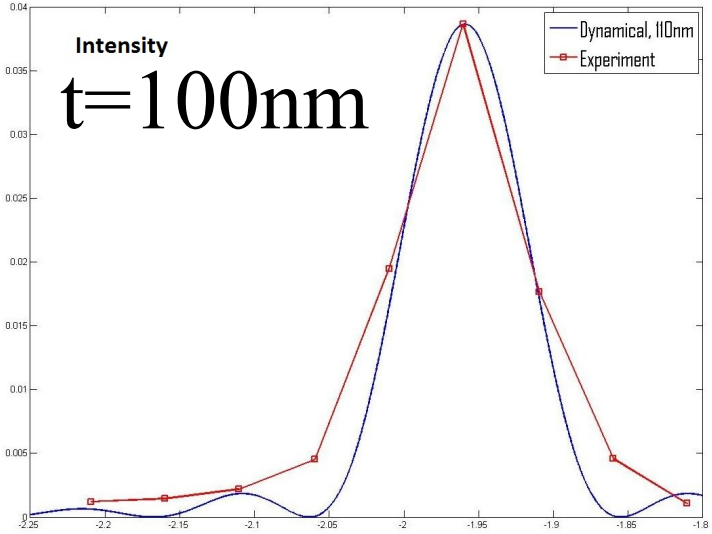
Ptychography of Gold Nanocrystals

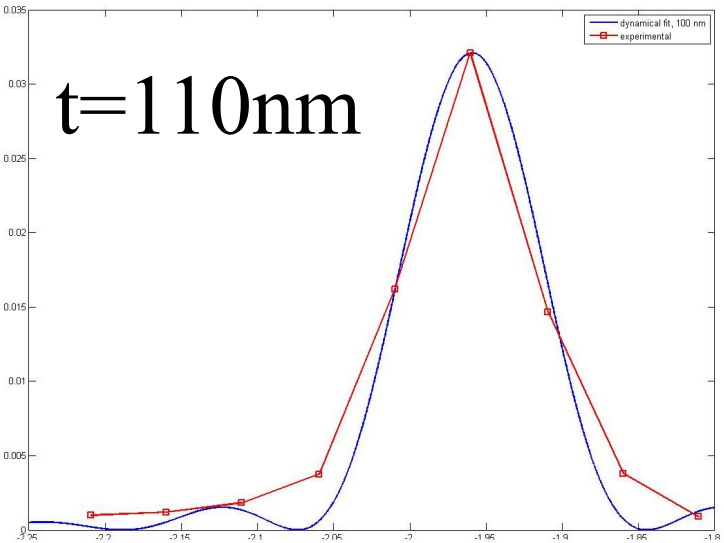
Maria Civita, UCL, Ana Diaz, Swiss Light Source

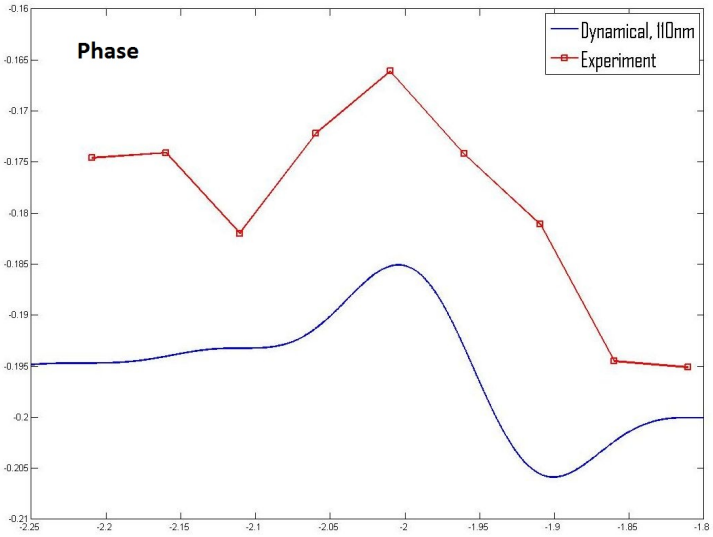


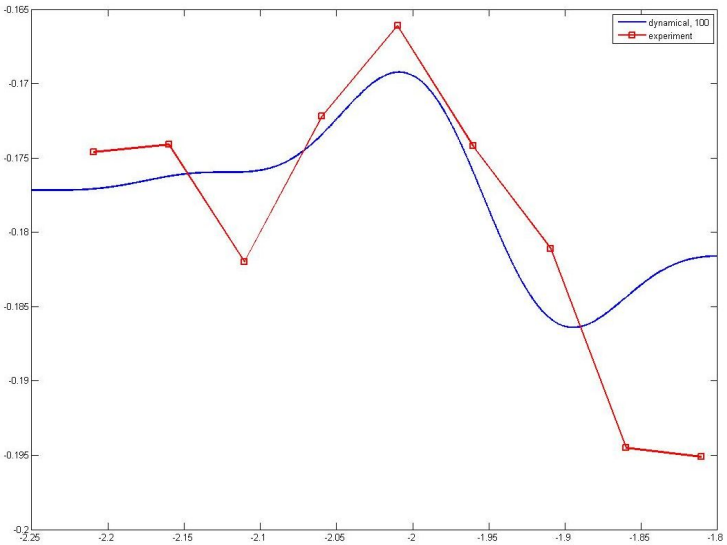


Dynamical Theory (Ivan Vartaniants)



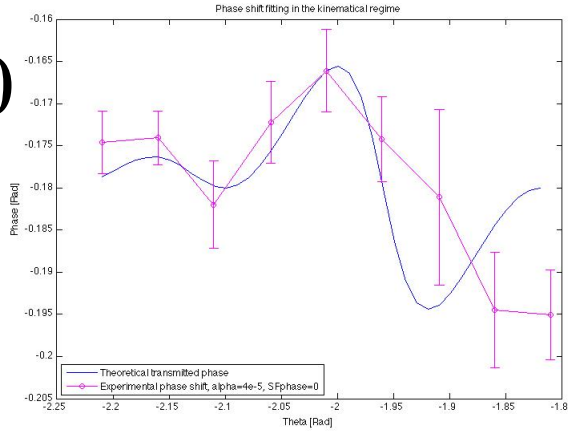




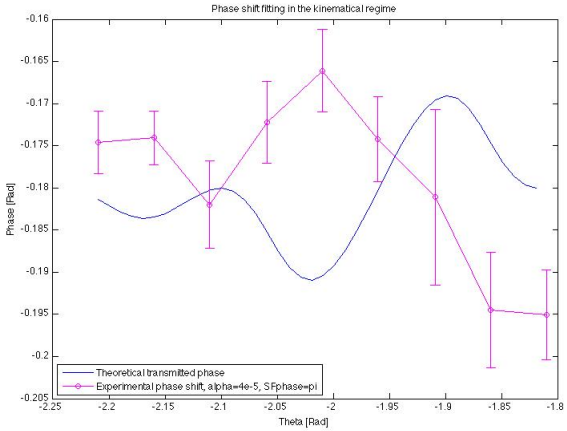


Sensitive to Crystallographic Phase

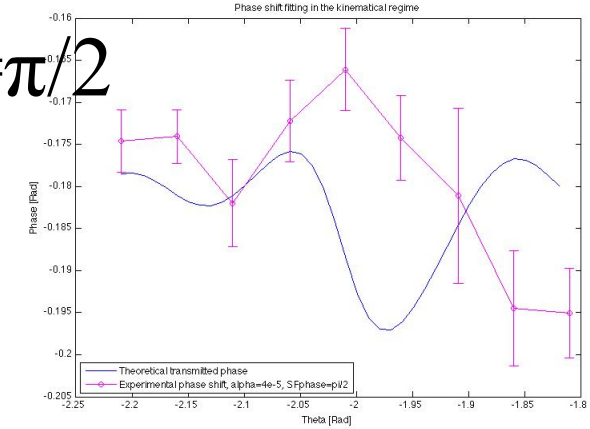
$\Phi_{SF}=0$



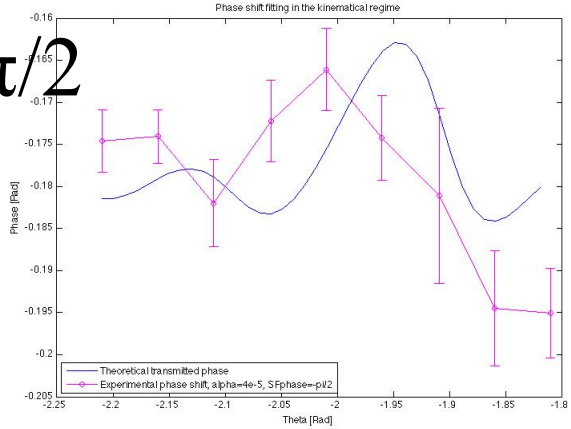
$\Phi_{SF}=\pi$



$\Phi_{SF}=\pi/2$



$\Phi_{SF}=-\pi/2$



Coherent x-ray diffraction (CXD)

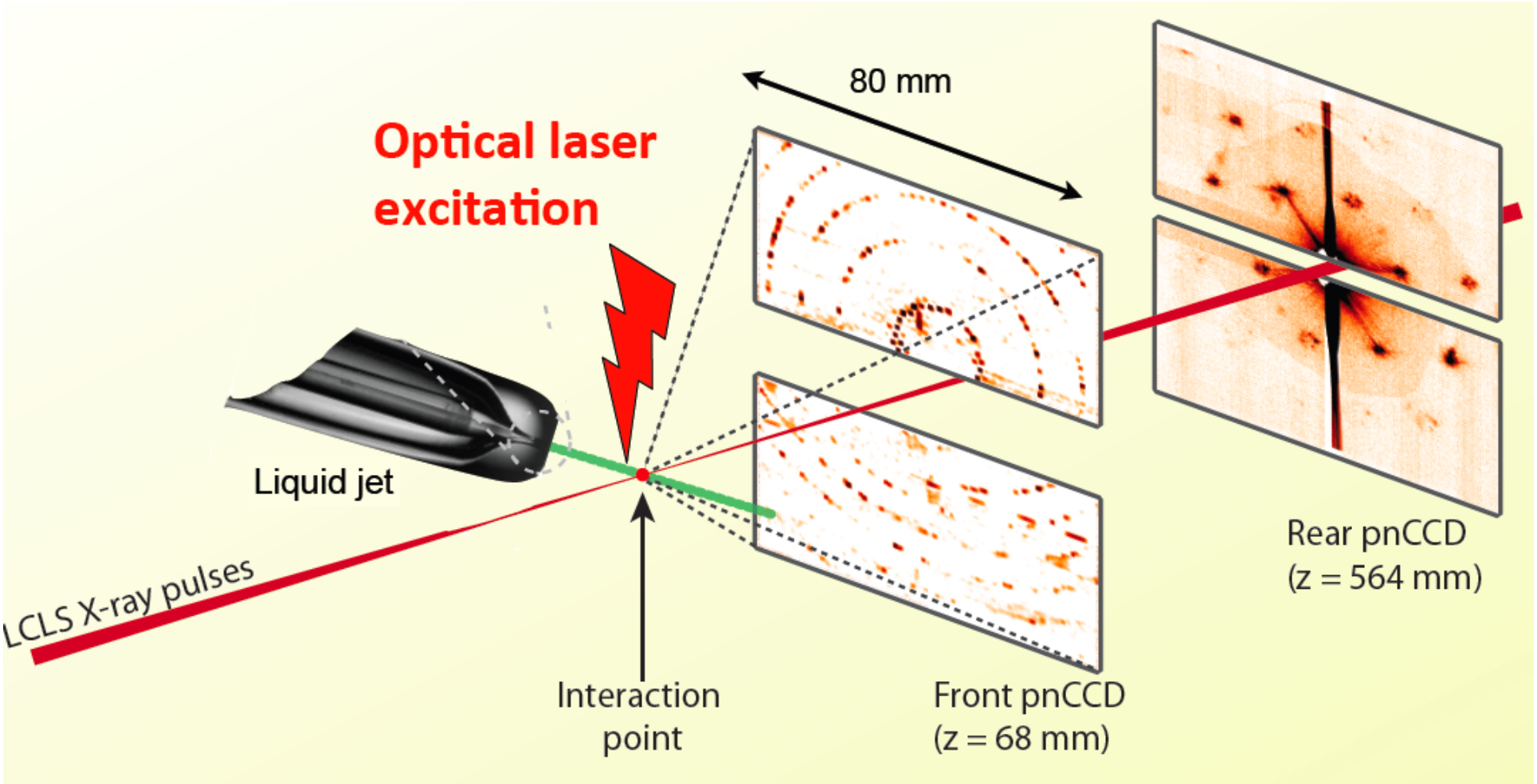
- Complex density can image strain
- Strain associated with nano-shape
- Differential strain with thiol chemistry
- Ultrafast snapshots of vibrations
- Crystallographic phase determination

Materials Science using XFELs

- Ground rule #1: €13,000 per hour
- Ground rule #2: fully automated experiment
- Ground rule #3: non-toxic, safe samples
- Ground rule #4: nanoparticles by the kg
- 27,000 pulses/sec at XFEL.EU, 2700x10
- up to 2000 frames/sec data rate = TB/hr

Pump-probe schematic

Henry Chapman, Bio-XFEL workshop Jan 2011

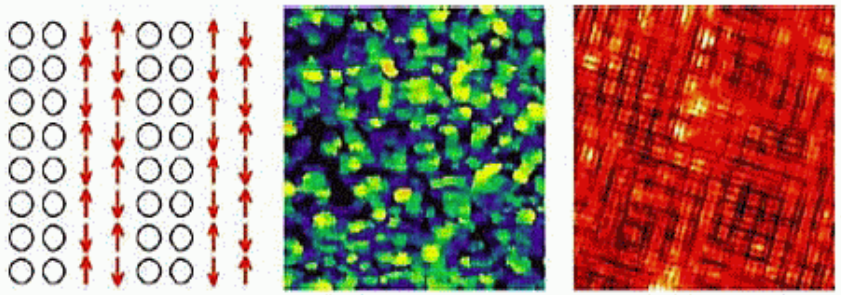


Materials Science using XFELs

- Nanoparticles injected into X-ray beam
- Synthesize samples in nanoparticle format
- Veto frames where no "hit" or diffraction
- Pump-Probe has two state variables
  - Optical fluence = sample temperature
  - Delay time after 'instantaneous' heating
- Laser "pump" pulse to create new states
- Explore transient phase diagrams

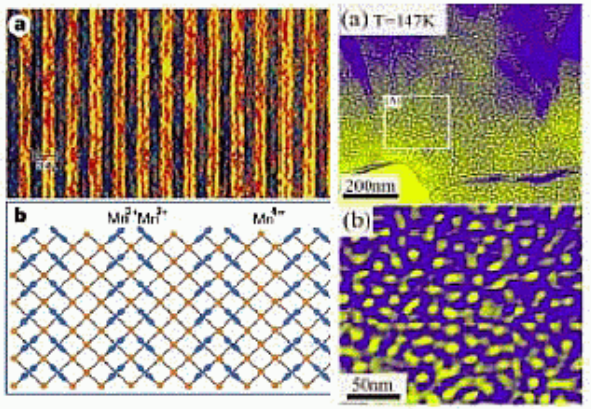
Stripes, checkerboards and zig-zags

High-Tc cuprates



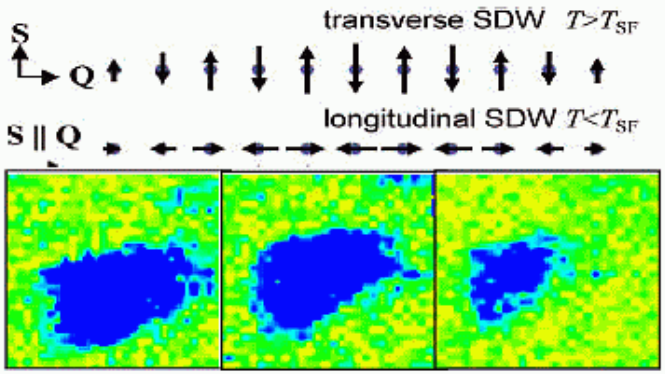
E. Dagotto, T. M. Rice, *Science* **271**, 618 (1996).
T. Hanaguri et al., *Nature* **430**, 1001 (2004).

CMR manganites

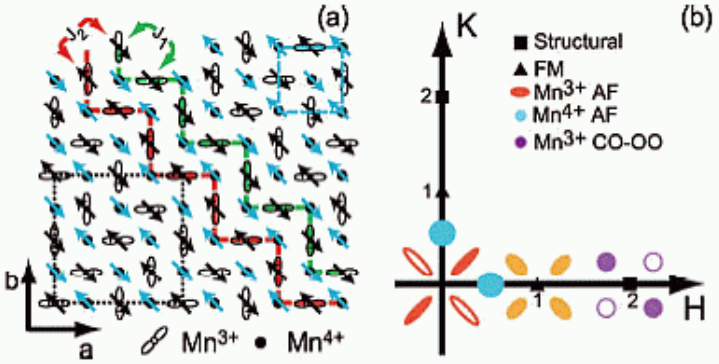


S. Mori et al., *Nature* **392**, 473 (1998)
M. Uehara et al., *Nature* **399**, 560 (1999)

AFM chromium



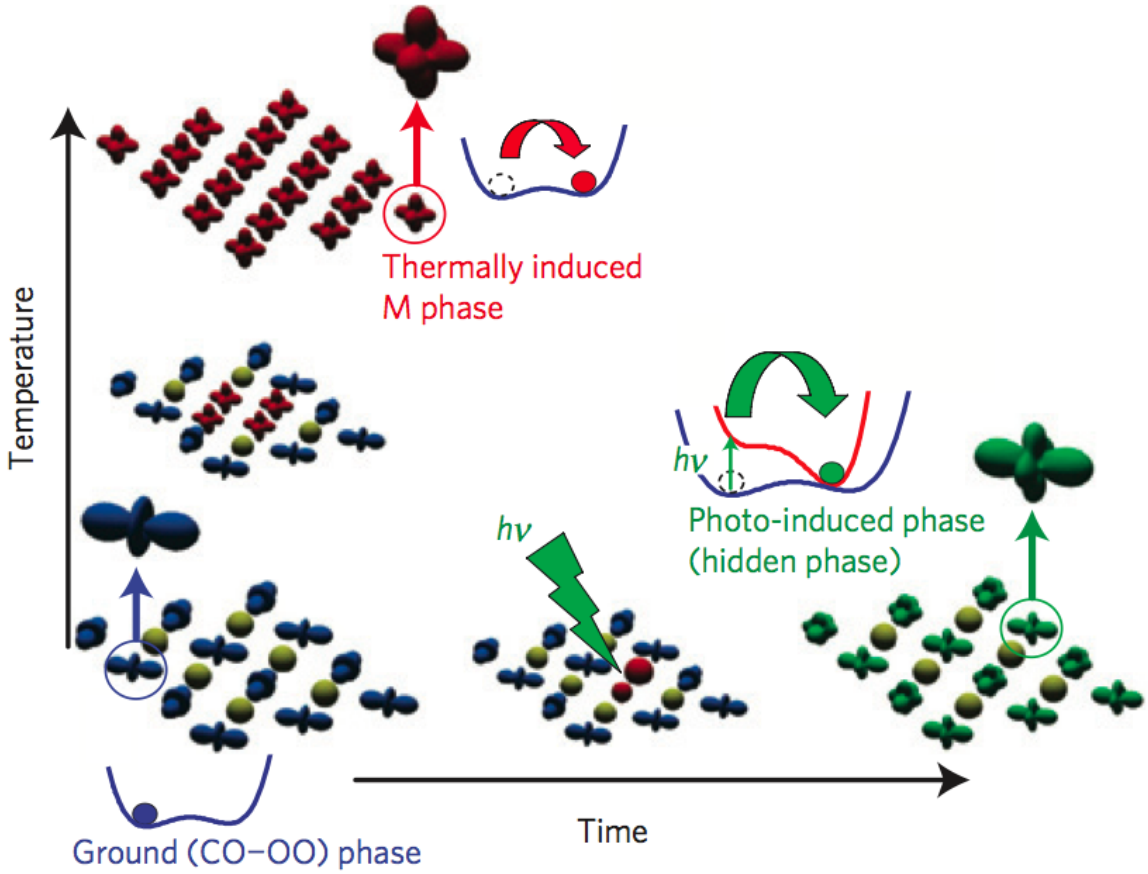
P. G. Evans et al., *Science* (2002)

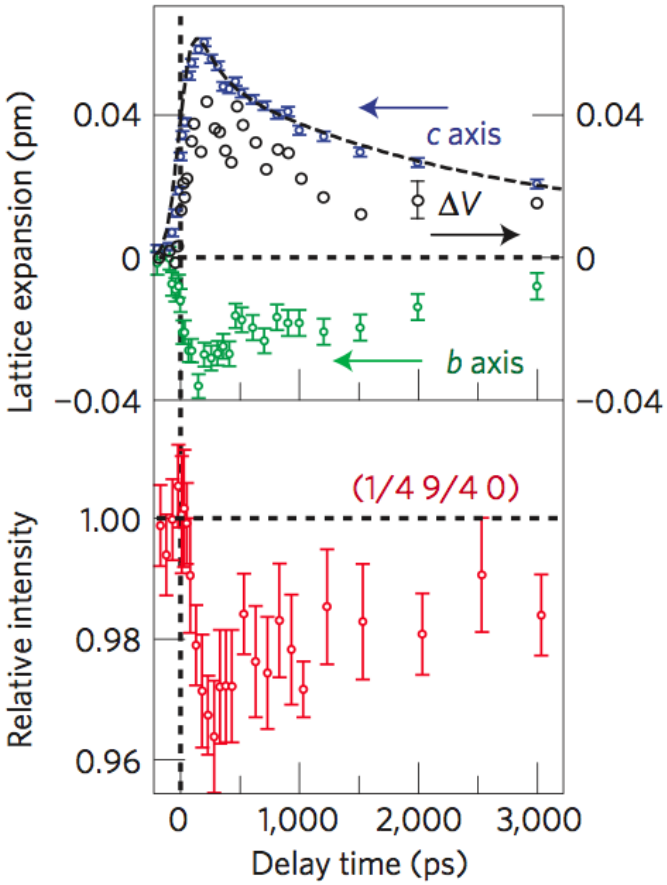


Transient "Hidden" Phase in Manganite

Hirohiko Ichikawa et al, Nature Materials **10** 101 (2011)

$Nd_{0.5}Sr_{0.5}MnO_3$ (NSMO) film on STO





"Femtosecond X-ray Laser induced transient electronic phase change observed in fullerene C_{60}"
B Abbey and H. Quiney, archive preprint (2013)