

Ultrafast Mapping of Strain Using Coherent X-Ray Diffraction

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Jesse Clark

Ross Harder

Xiaoqing Huang

Moyu Watari

Loren Beitra

Gang Xiong

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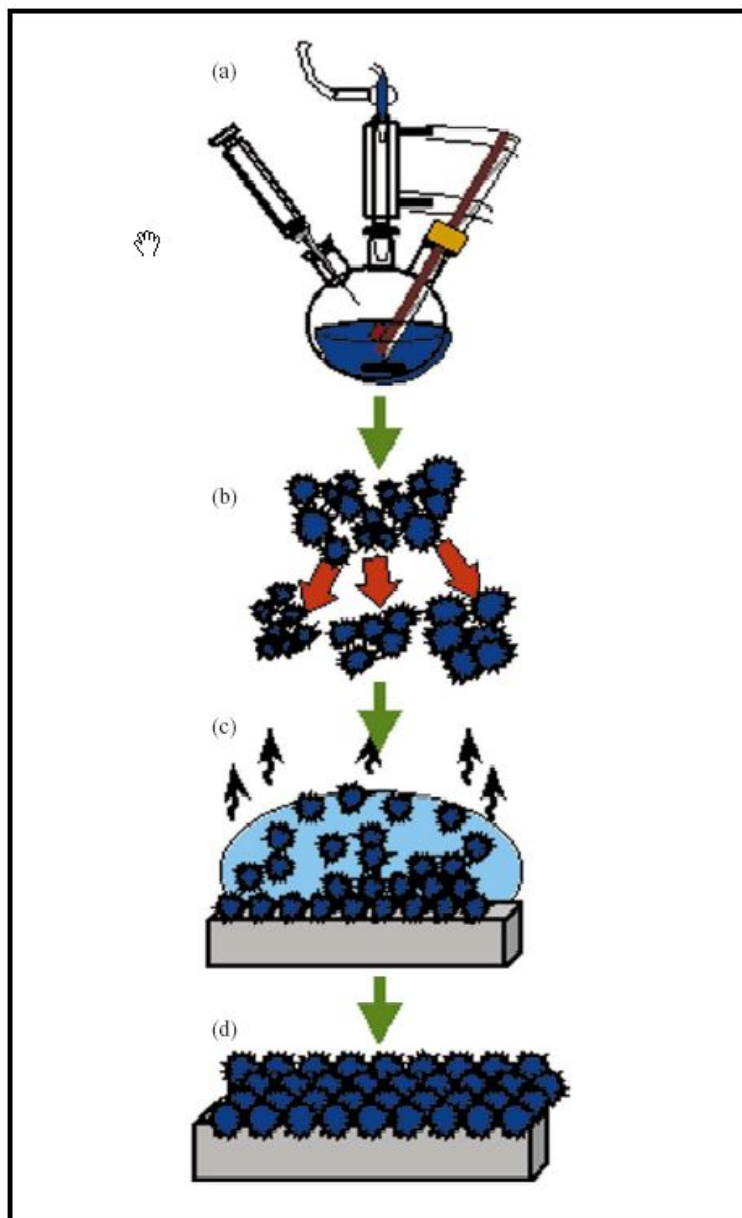
London Centre for Nanotechnology
Research Complex at Harwell

MSD Colloquium
Argonne
November 2012

Outline

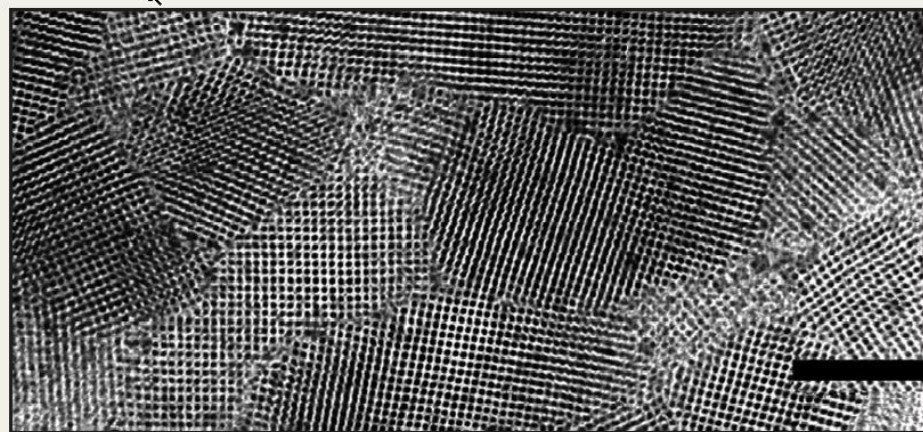
- Nanocrystal structures
- Coherent x-ray diffraction
- Crystal strain as complex density
- Strain induced by surface reactions
- Partial coherence correction
- Ultrafast CDI of nanocrystals
- Materials Science by XFEL?

Chemical Synthesis of Nanocrystals



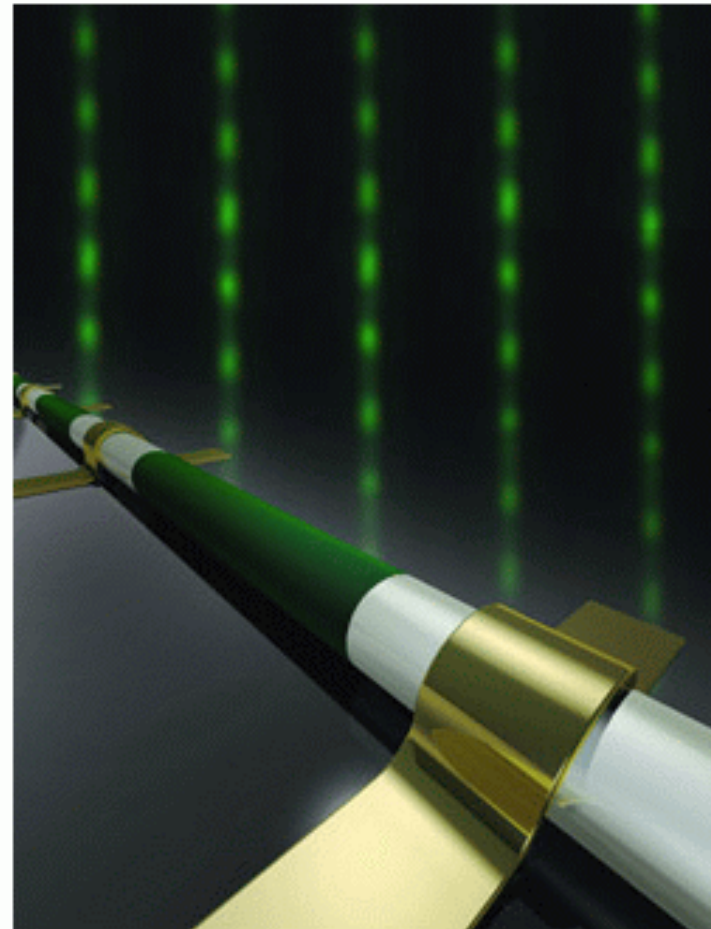
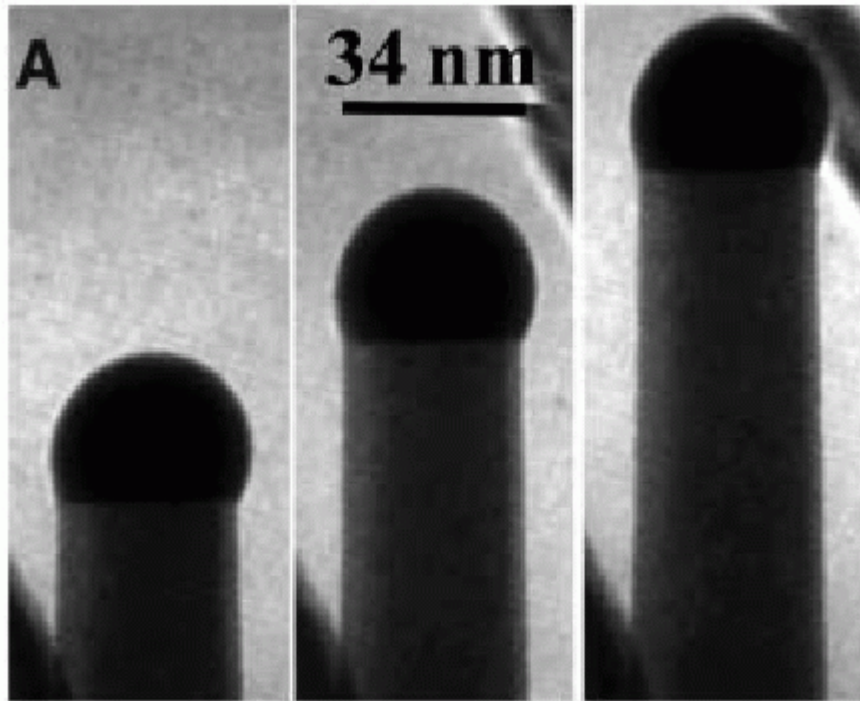
- Reactants introduced rapidly
- High temperature solvent
- Surfactant/organic capping agent
- Square superlattice (200nm scale)

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



VLS growth of nanowires

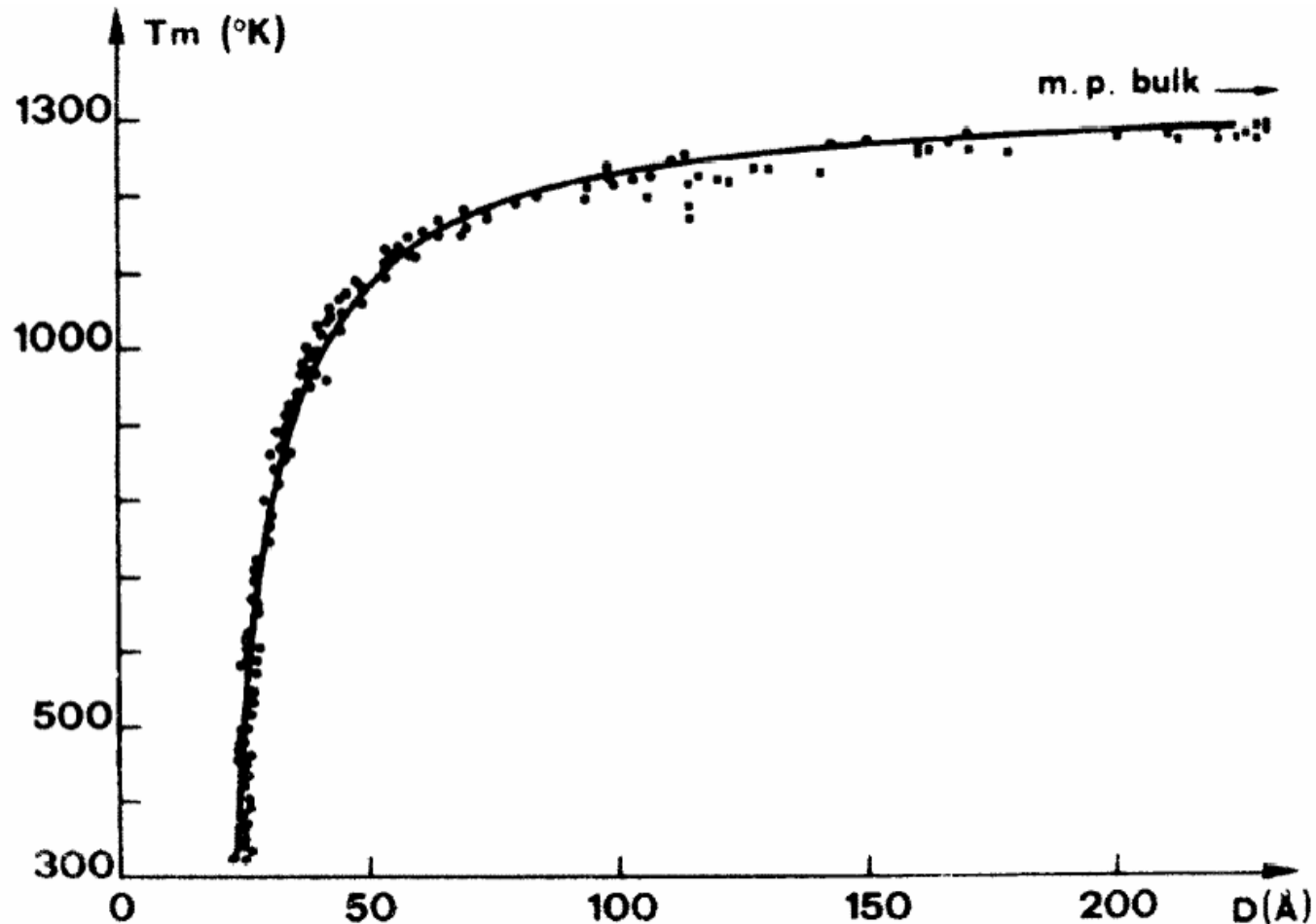
S. Kodambaka et al., *Science* 316 729 (2007)



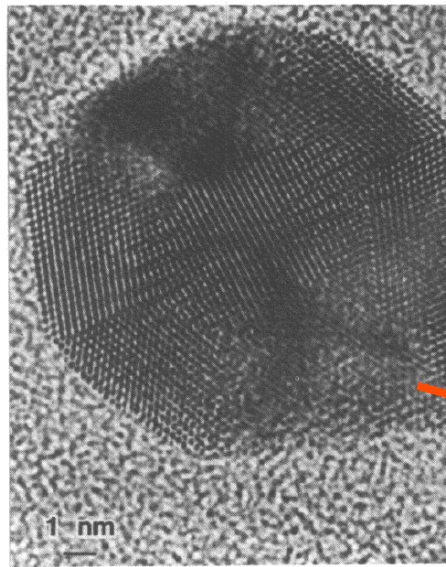
I. K. Robinson, ANL-M NiSi/Si nanowire heterostructure devices. *Nature* **430**, 61 (2004).

Size-dependent Melting of Au Particles

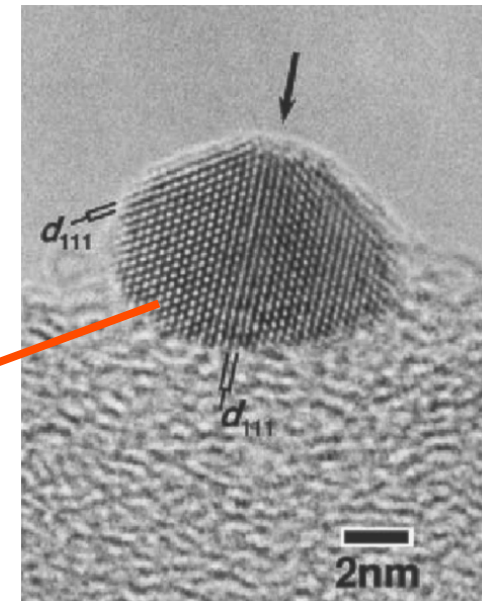
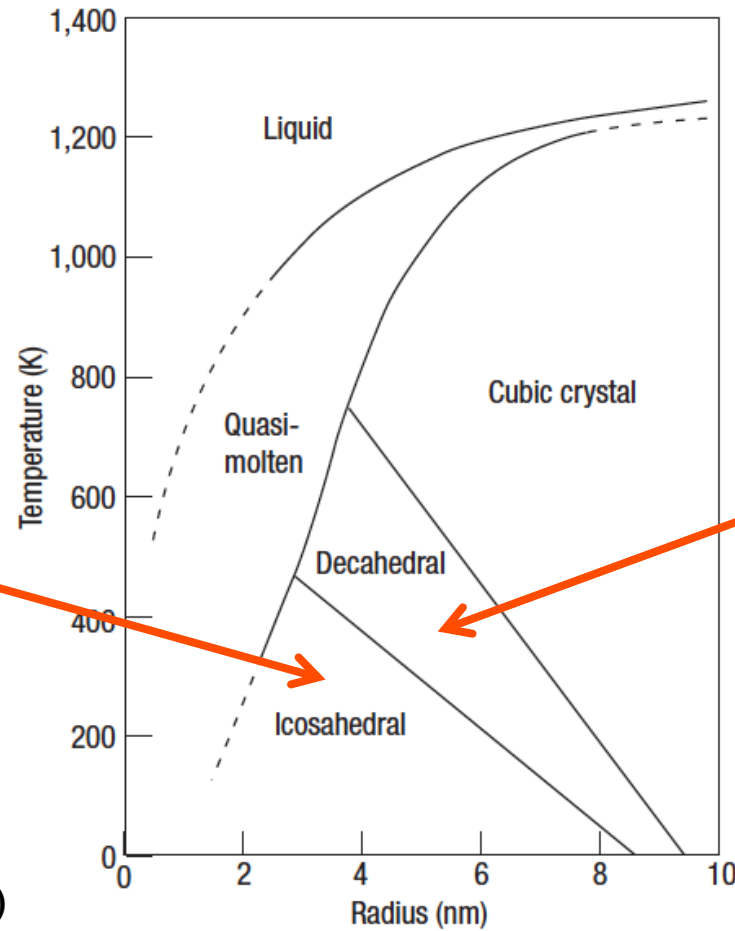
P. Buffat and J-P. Borel, Phys. Rev. A 2287-97 (1975)



Structure of Gold vs Size



L. D. Marks, RPP (1994)

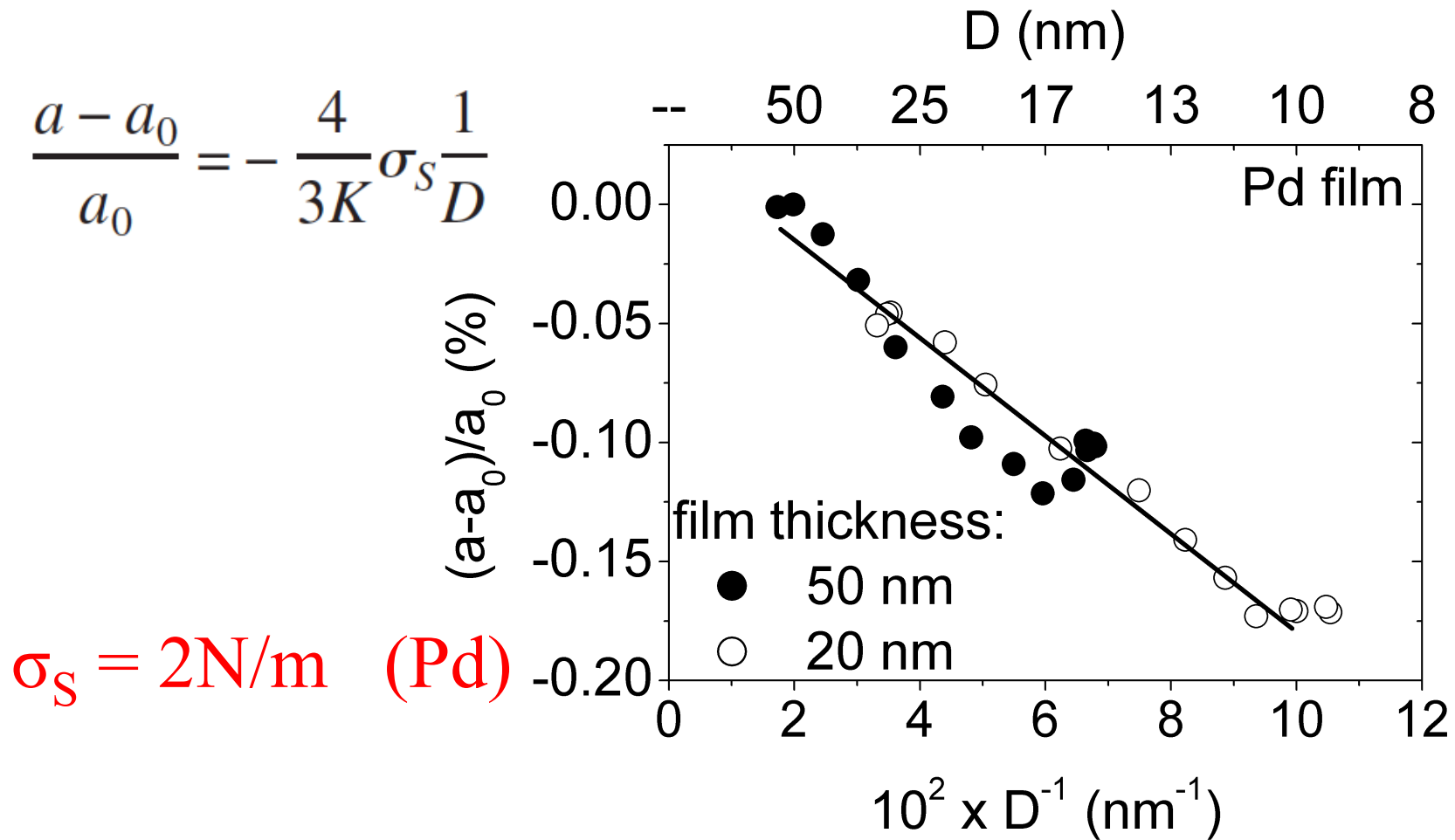


Koga and Sugawara (2003)

Contraction of Small Particles

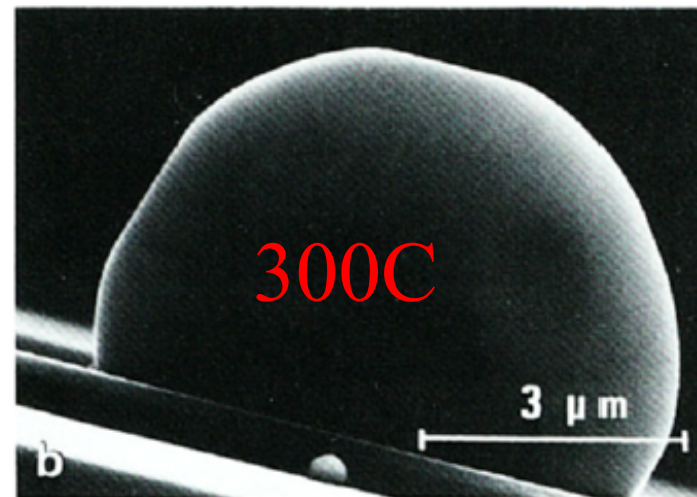
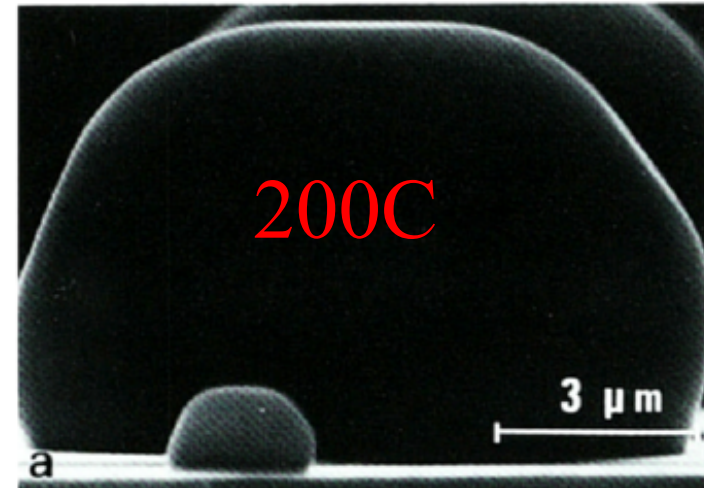
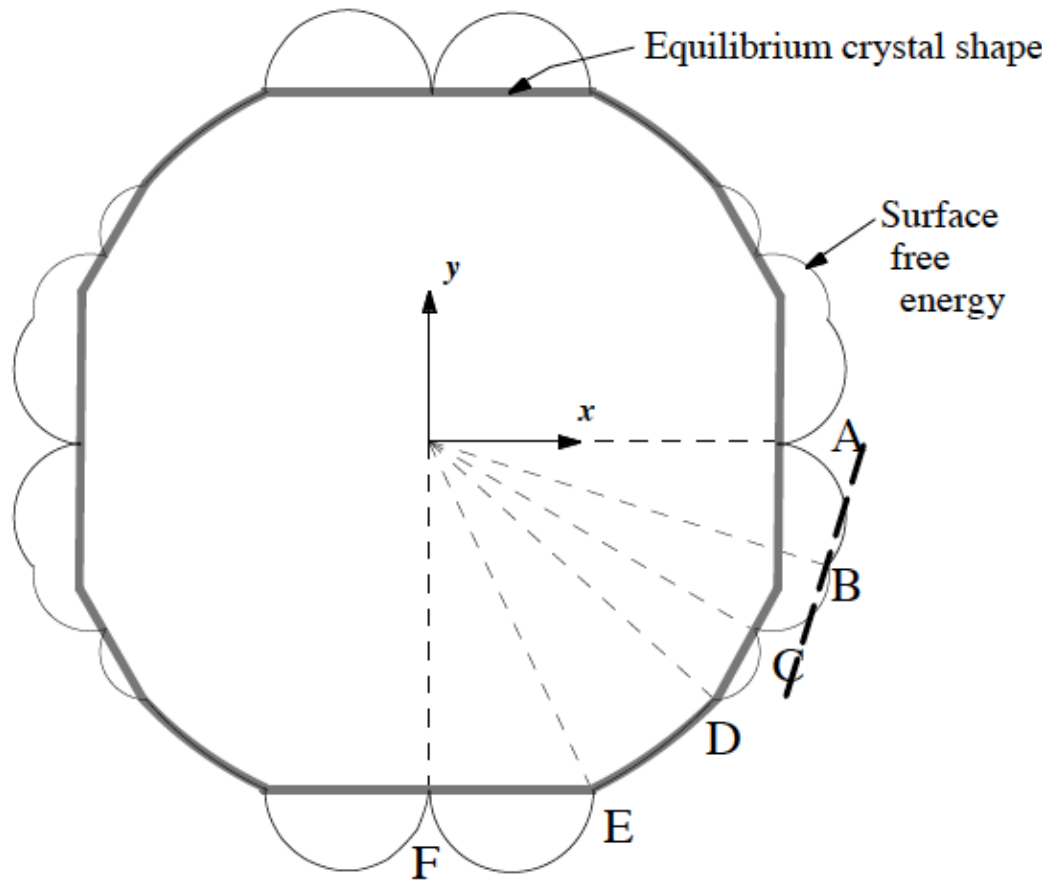
Gibbs Thomson pressure + Bulk modulus

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



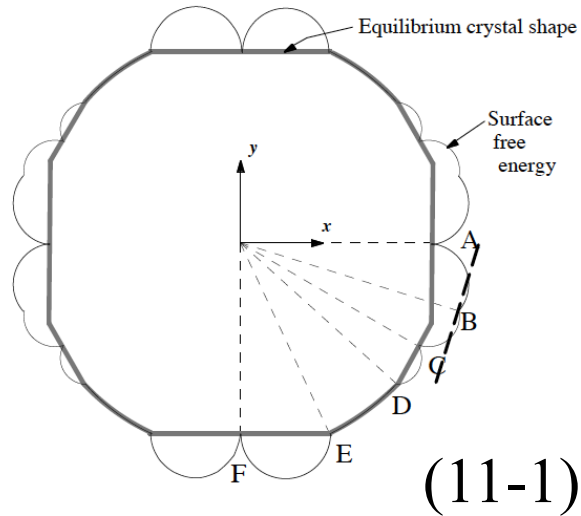
Equilibrium crystal shape

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

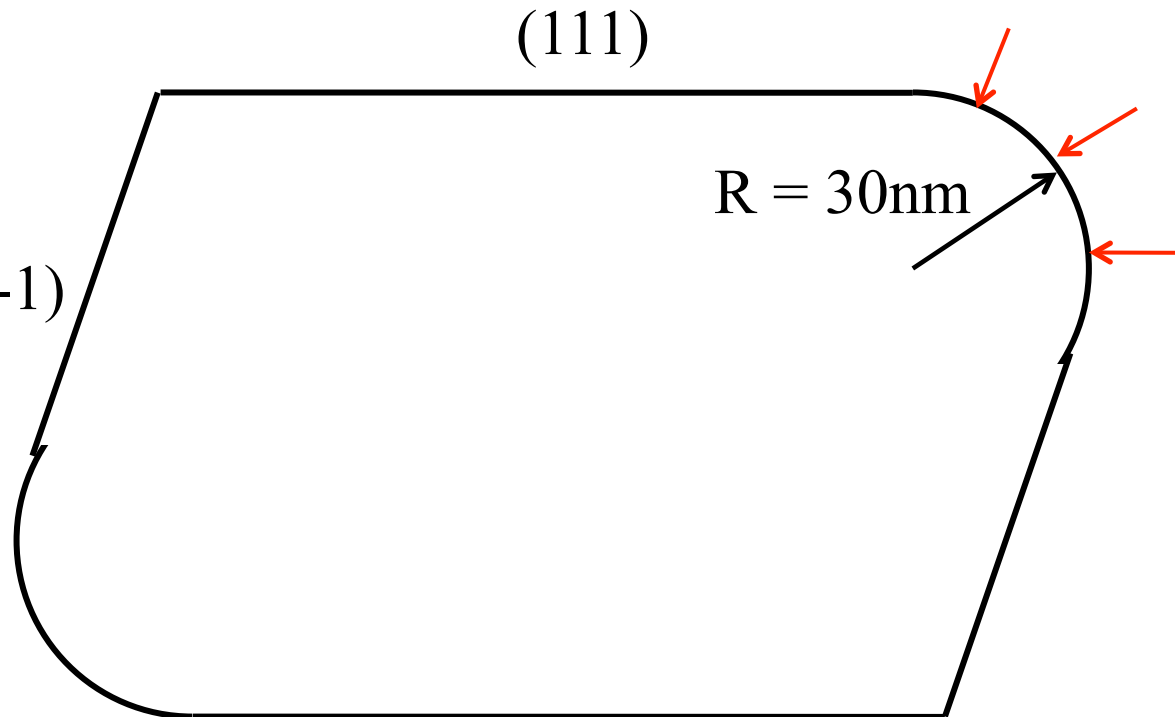


Equilibrium crystal shape

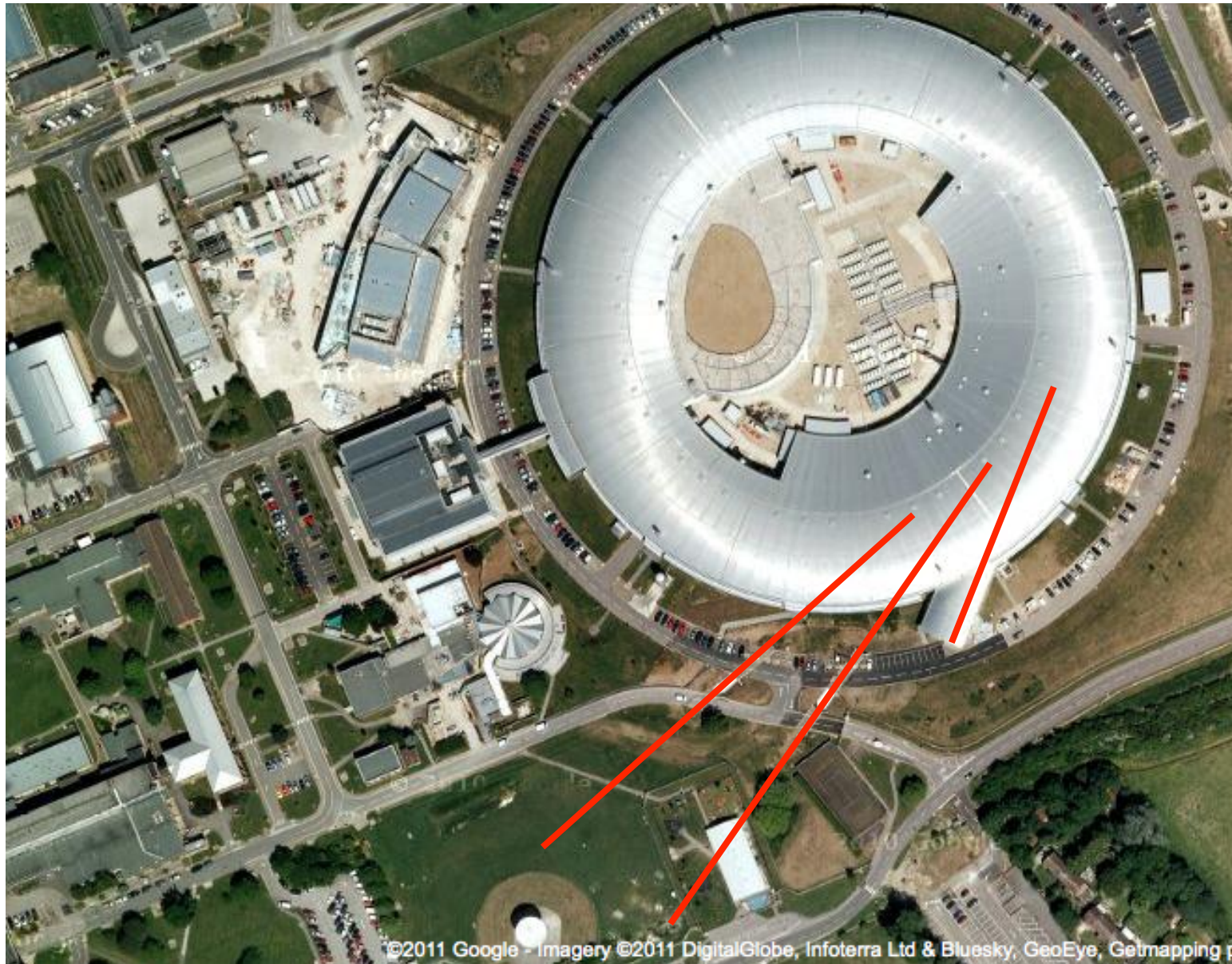
Wulff construction + Gibbs Thomson (Young-Laplace) pressure



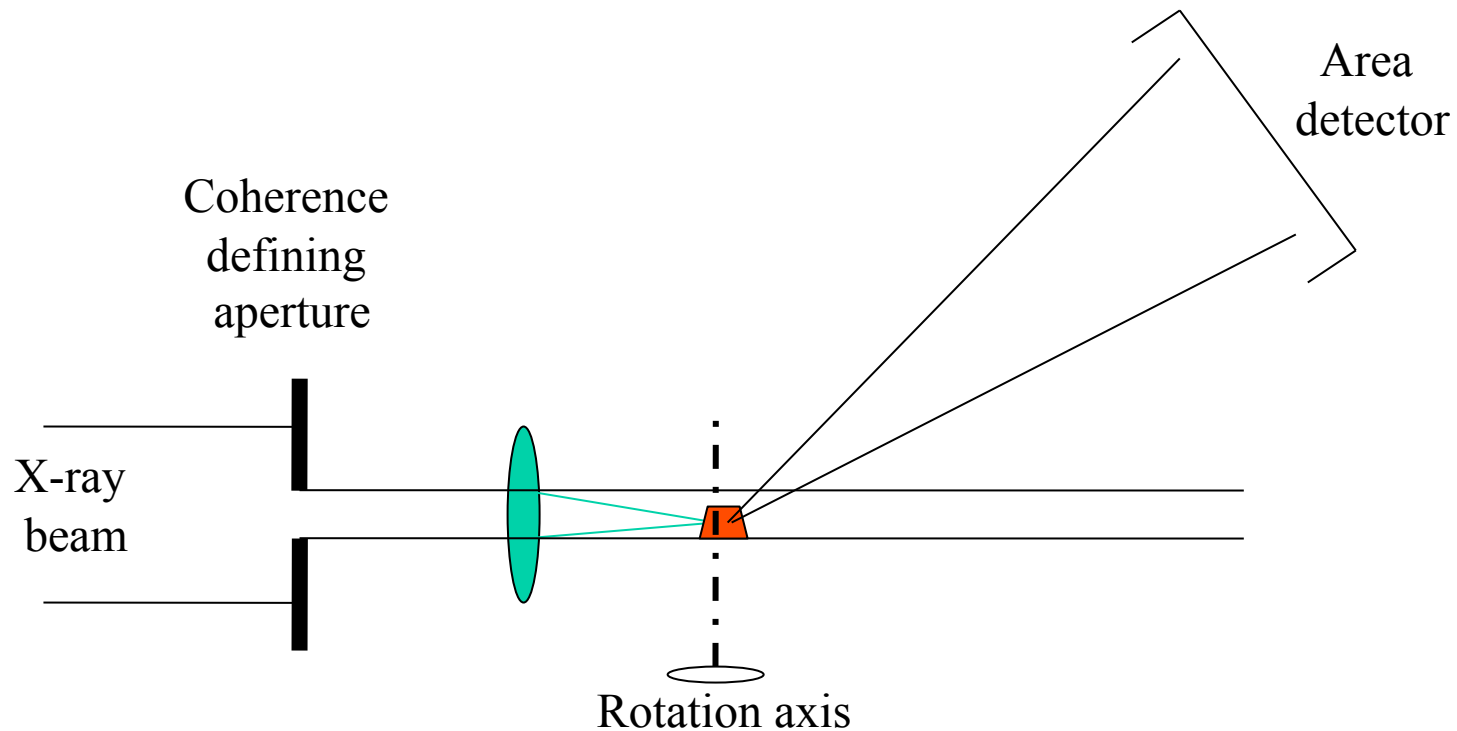
$$\gamma = 2\text{N/m}$$
$$P = 2\gamma/R = 70\text{MPa}$$
$$\varepsilon = 0.1\%, \quad \Phi = 0.6 \text{ rad}$$

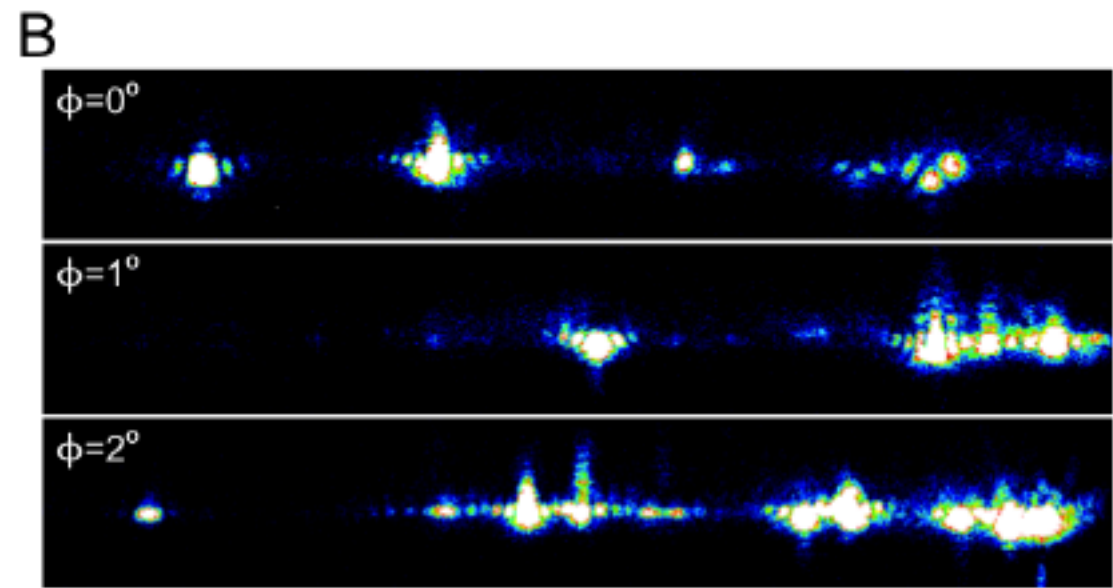
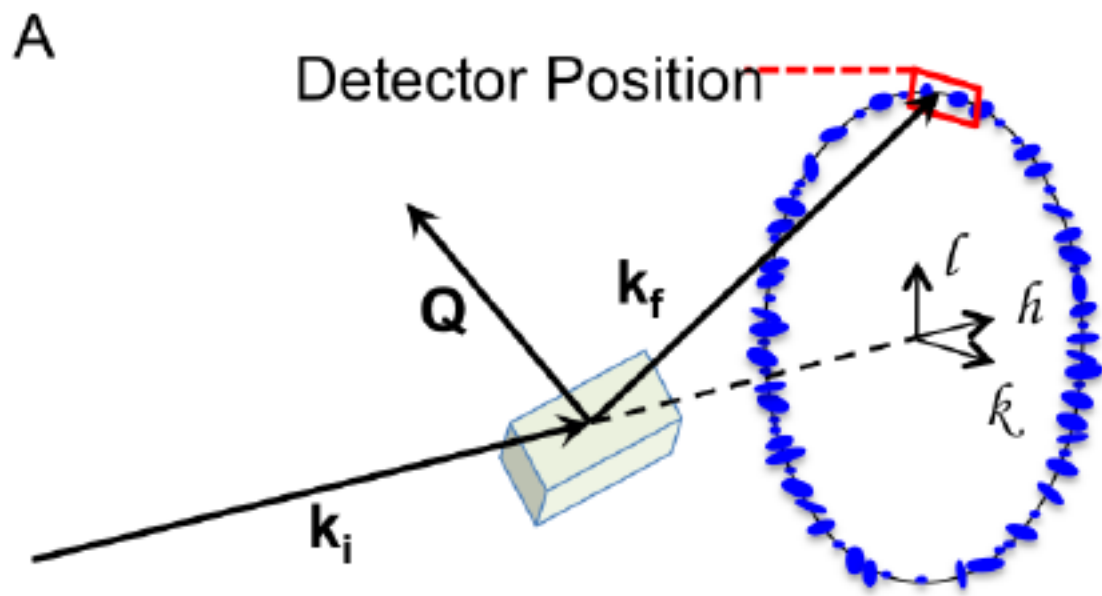


Diamond Light Source, 2009

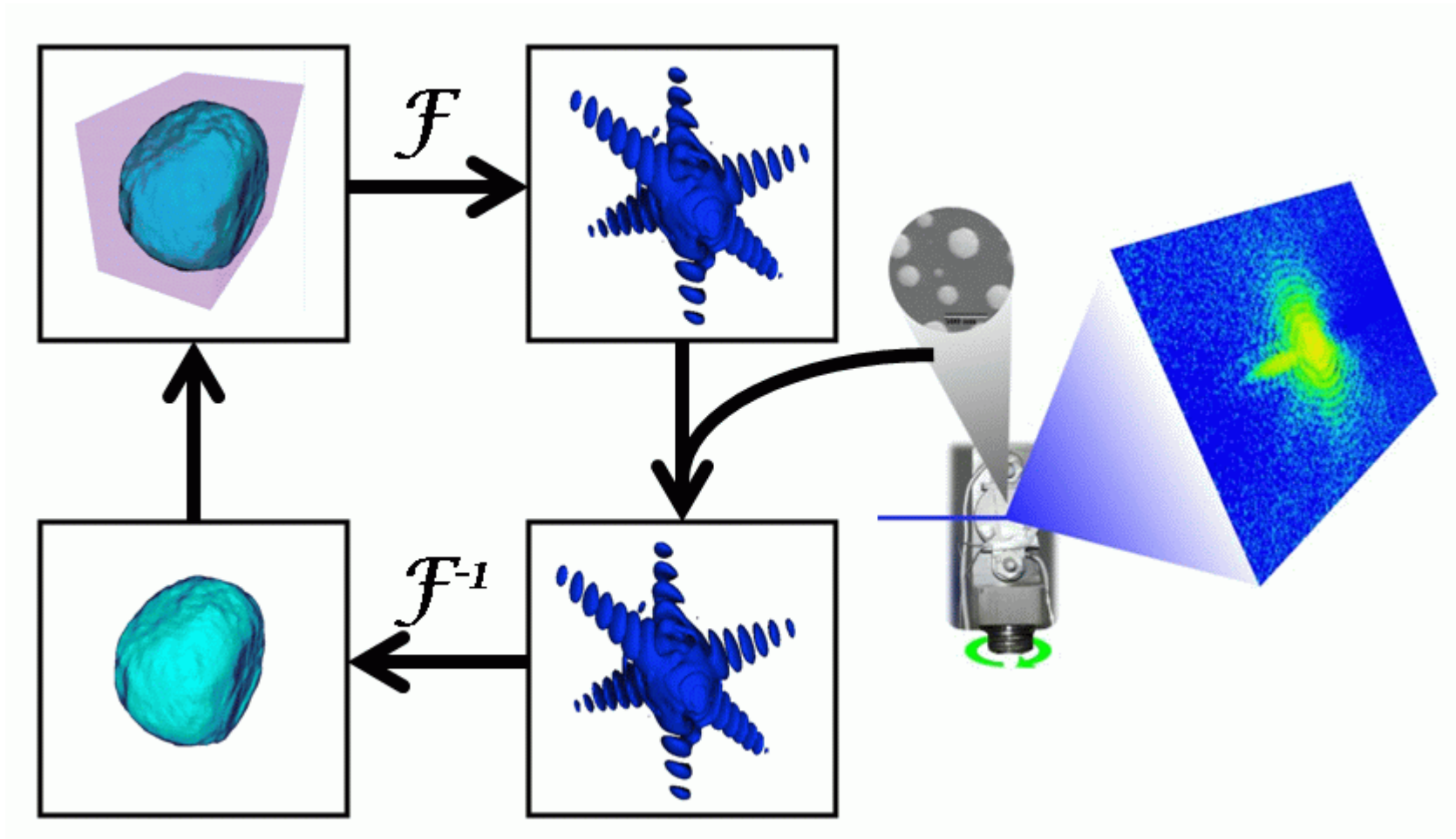


Lensless X-ray Microscope, 2003





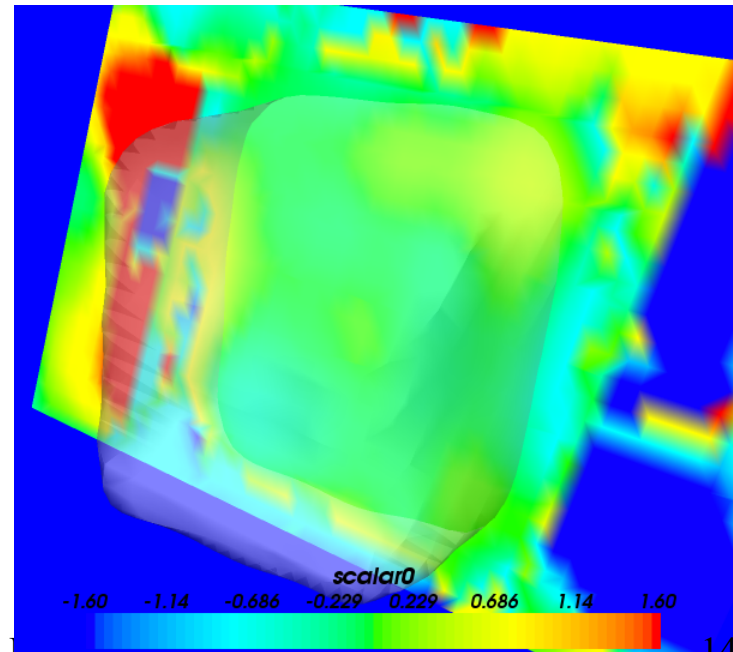
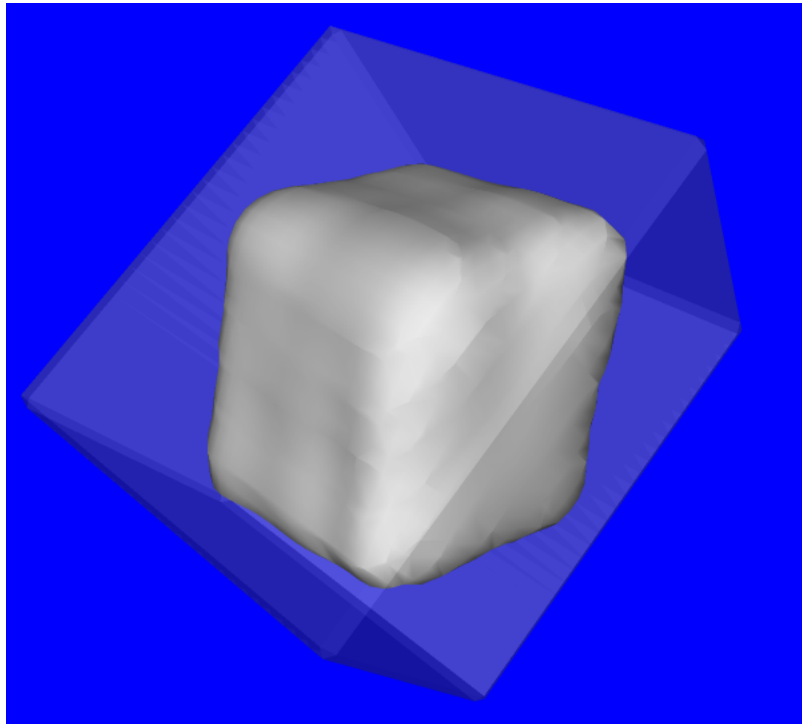
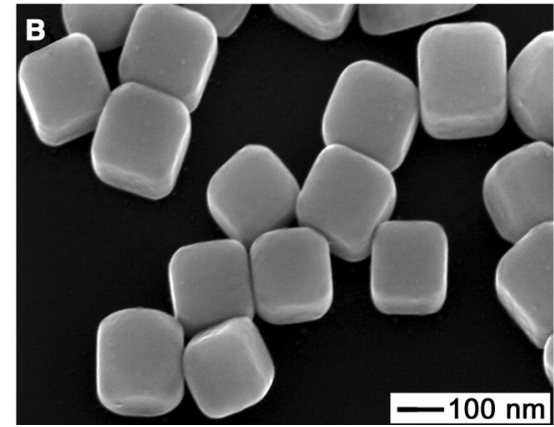
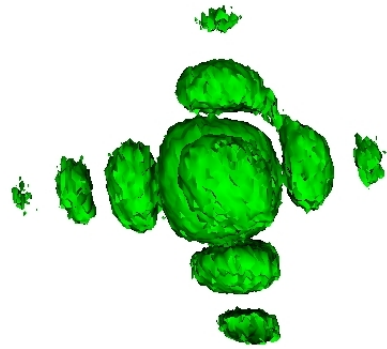
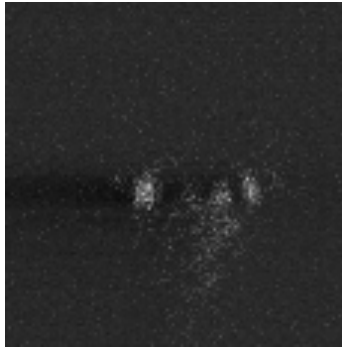
Generic “Error Reduction” method



J. R. Fienup *Appl. Opt.* 21 2758 (1982)

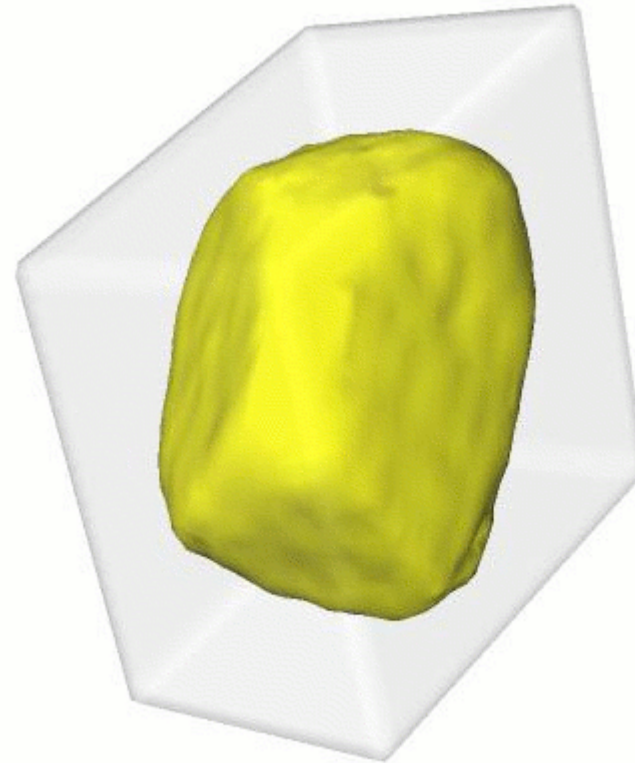
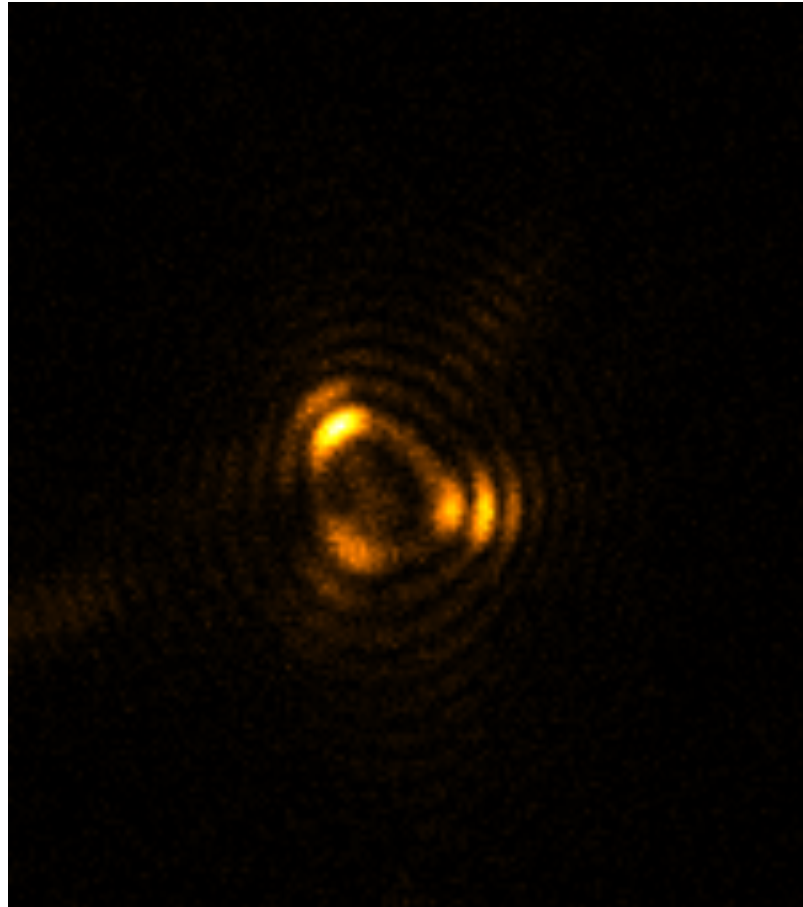
R. W. Gerchberg and W. O. Saxton *Optik* 35 237 (1972)

I. K. Robinson, ANL-MSD 2012

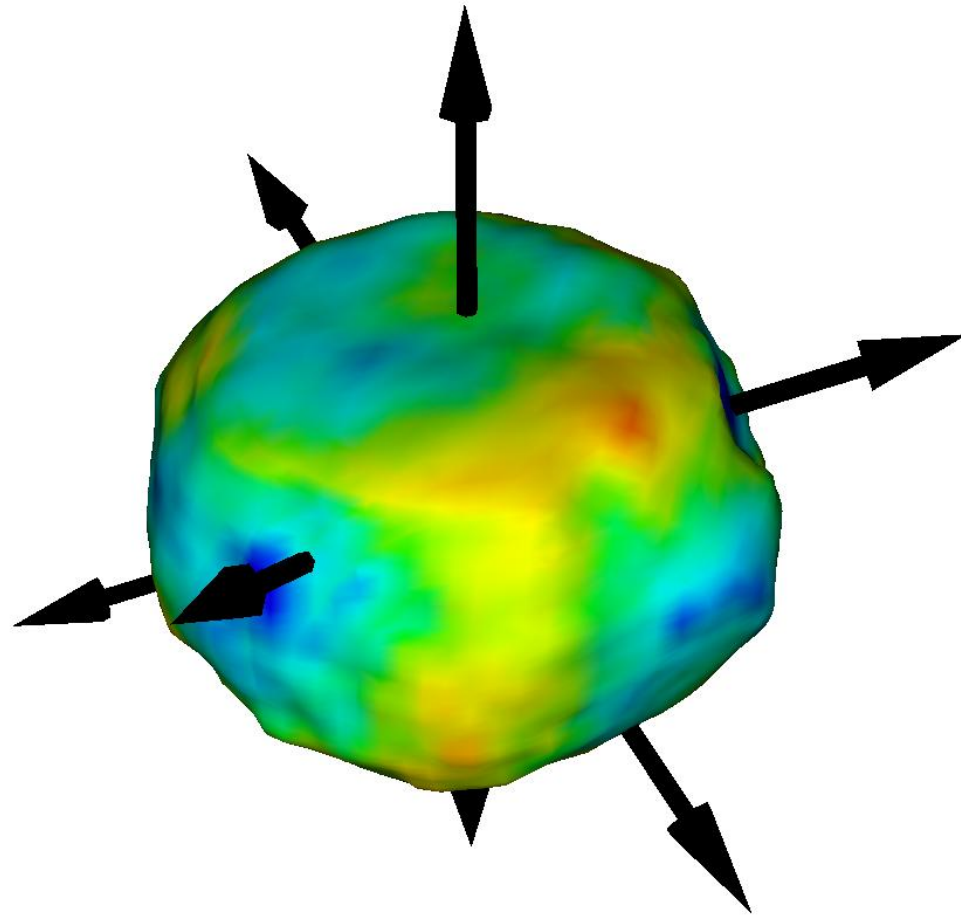


Gold nanocrystal reconstruction

showing support used for 20 HIO followed by 10 ER

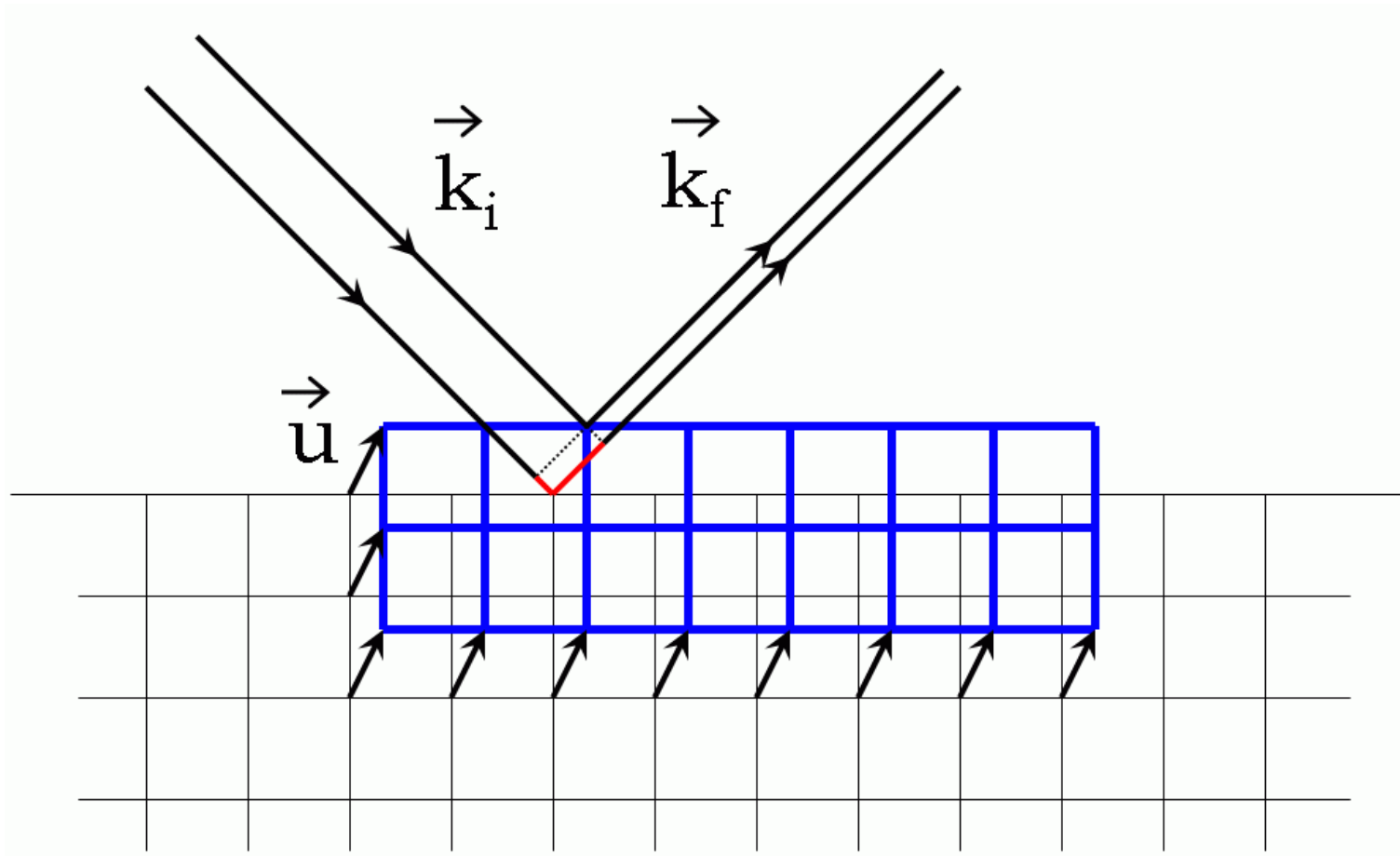


Phase isosurface of residual strain



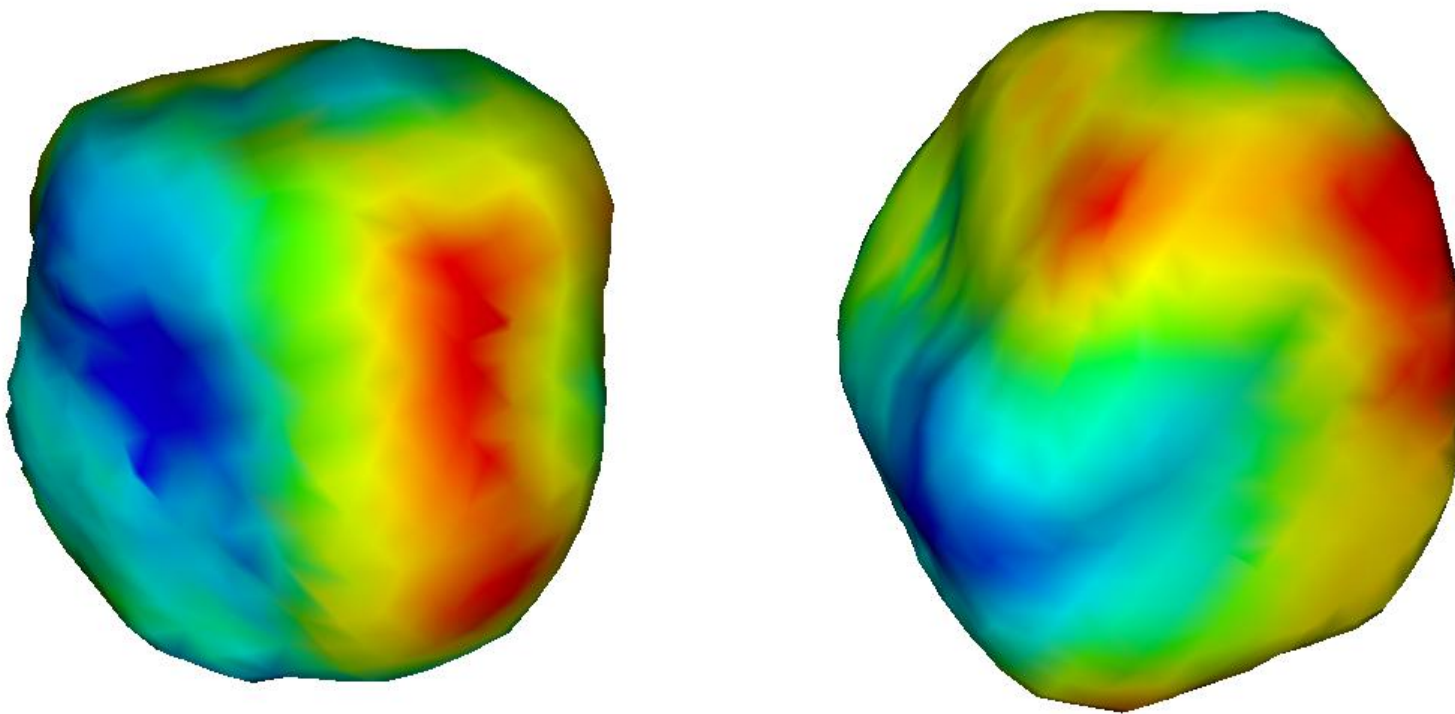
Sensitivity to strain

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



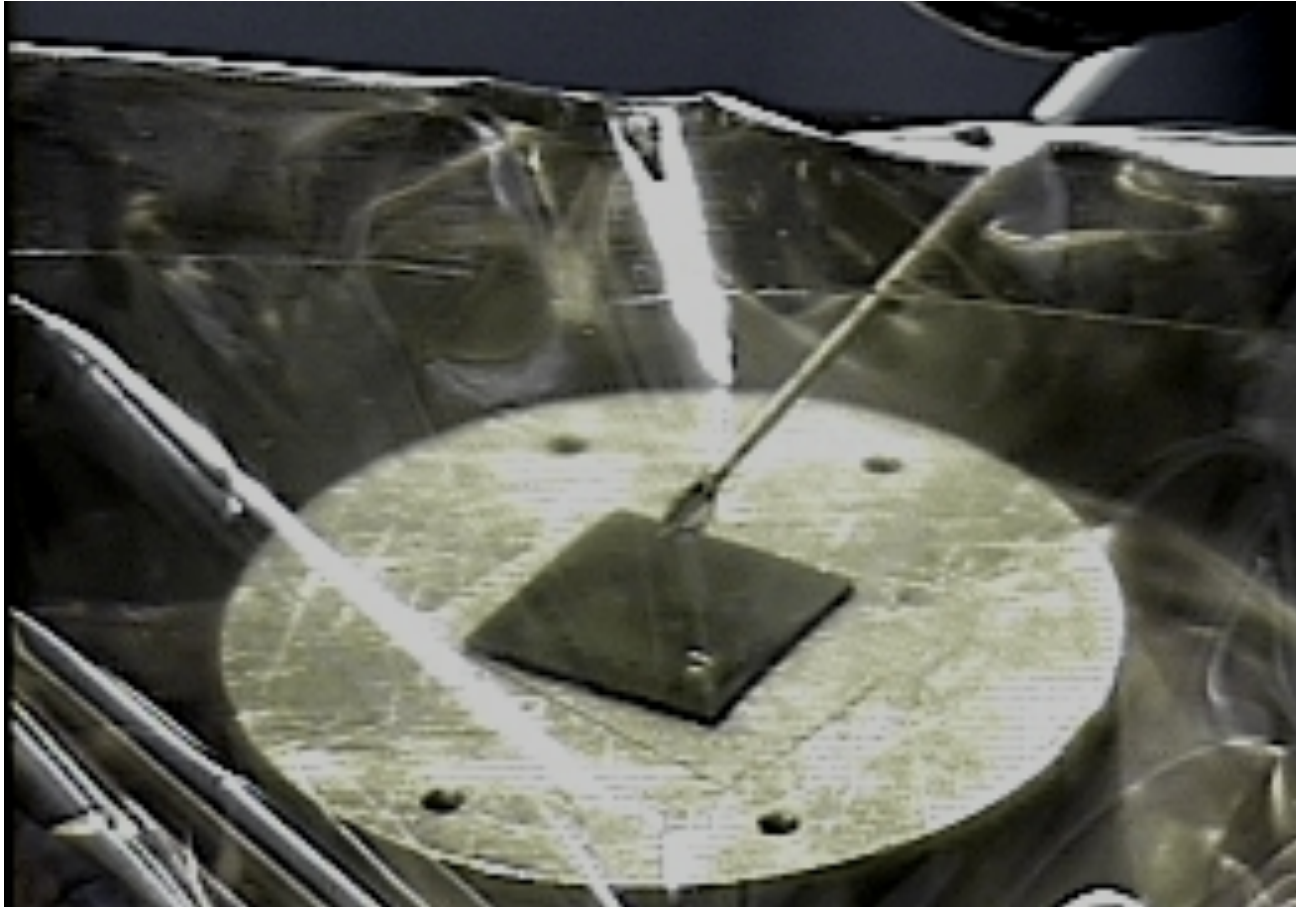
Phase isosurface of residual strain

200nm Barium Titanate (BTO) crystals



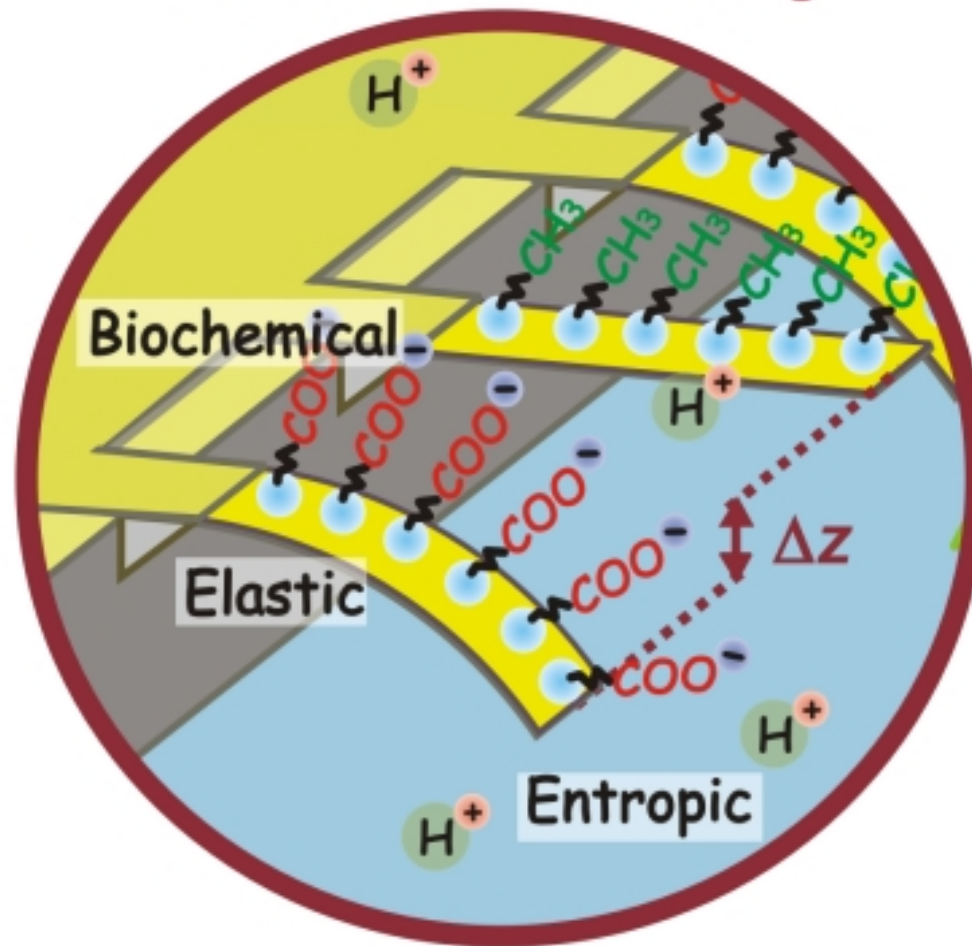
Dosing with C_3H_7SH in ethanol

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)



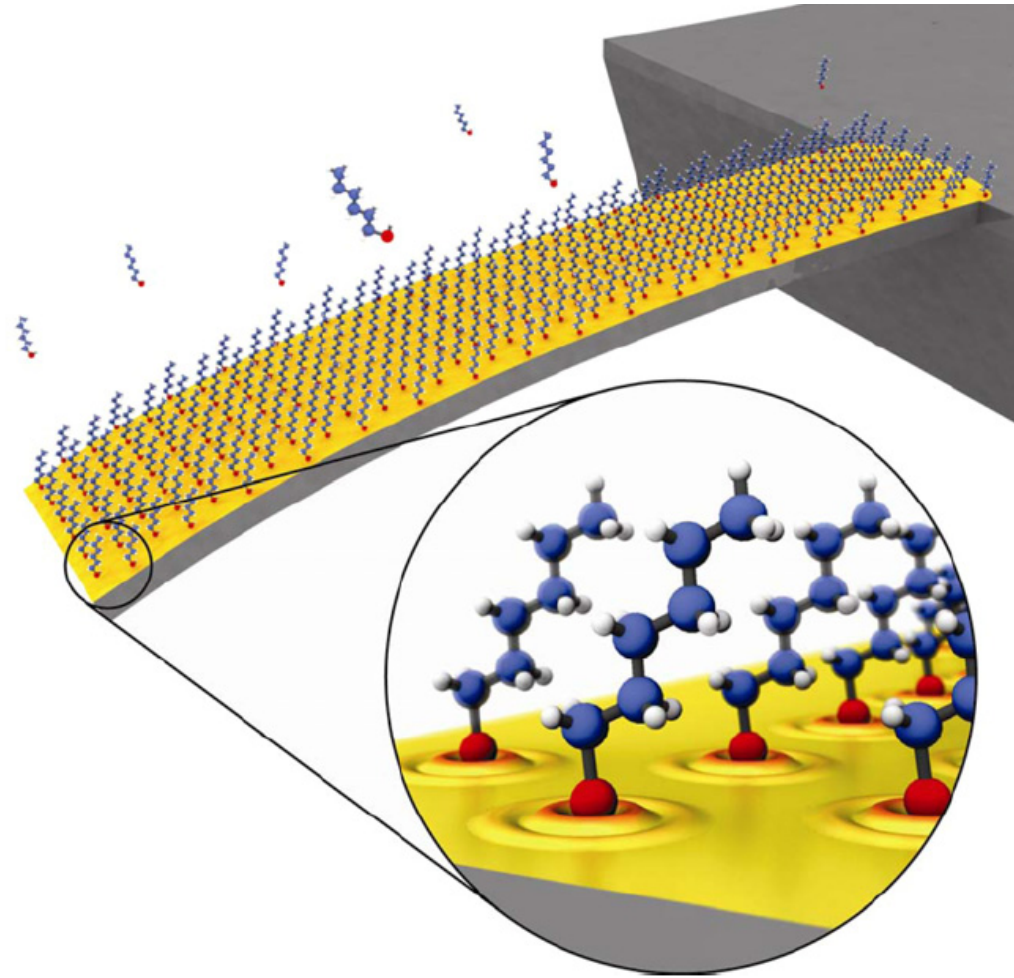
Nanocantilevers

Dr Rachel McKendrie



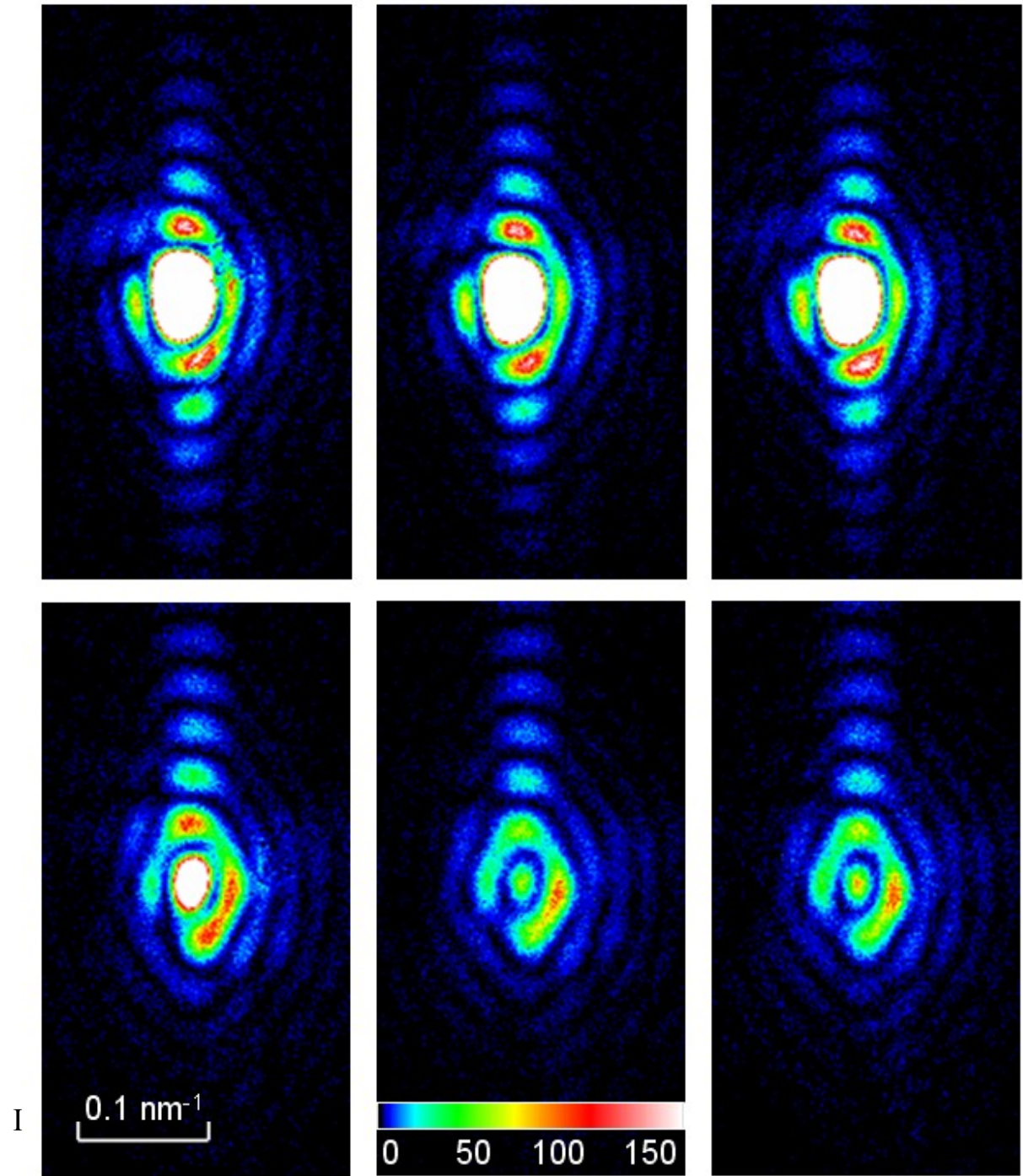
Formation of SAM on cantilever

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



Rocking curve of Au crystal

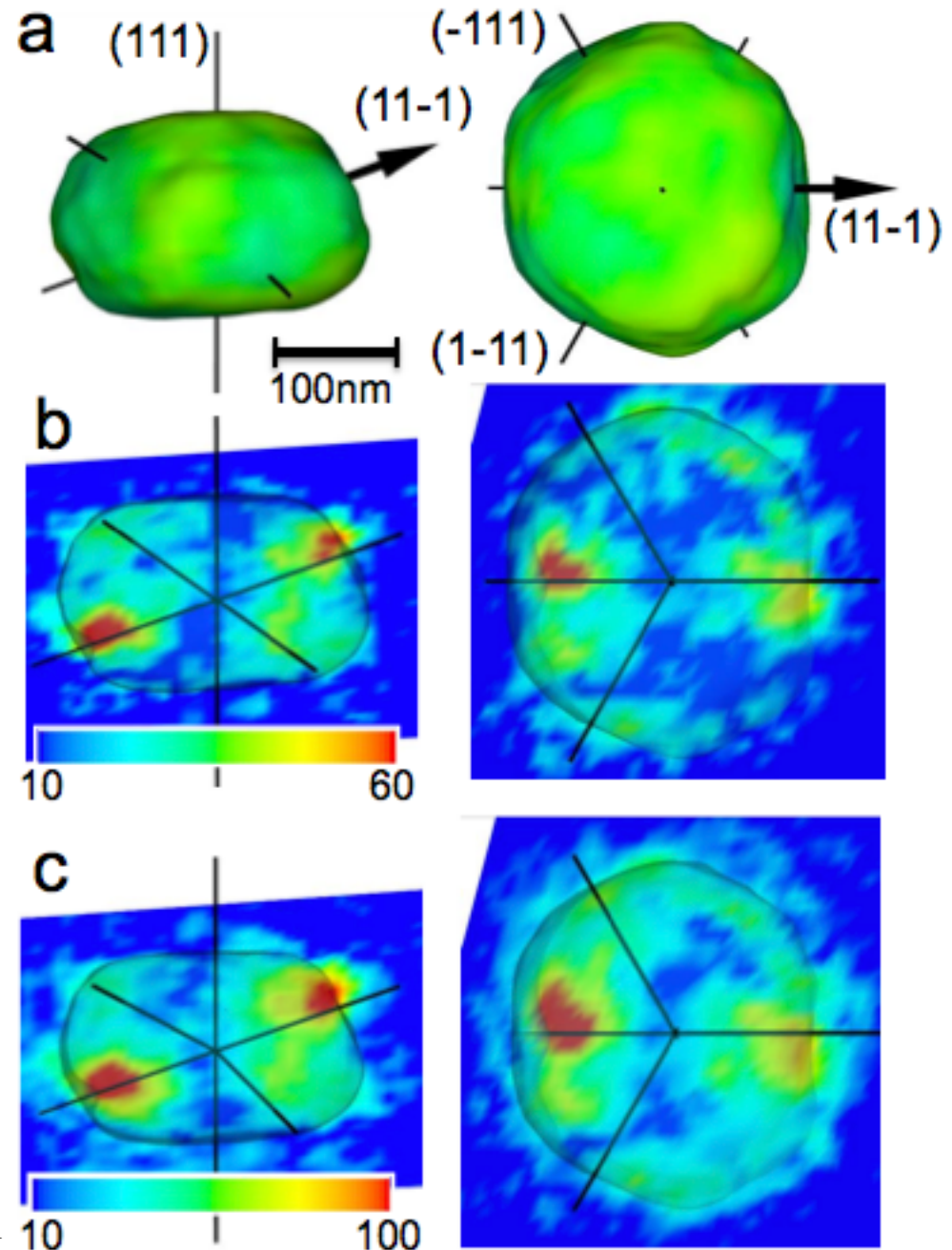
Before dosing /
dose 1 / dose 2



Difference Fourier:

Magnitude of phase
change caused by
dosing

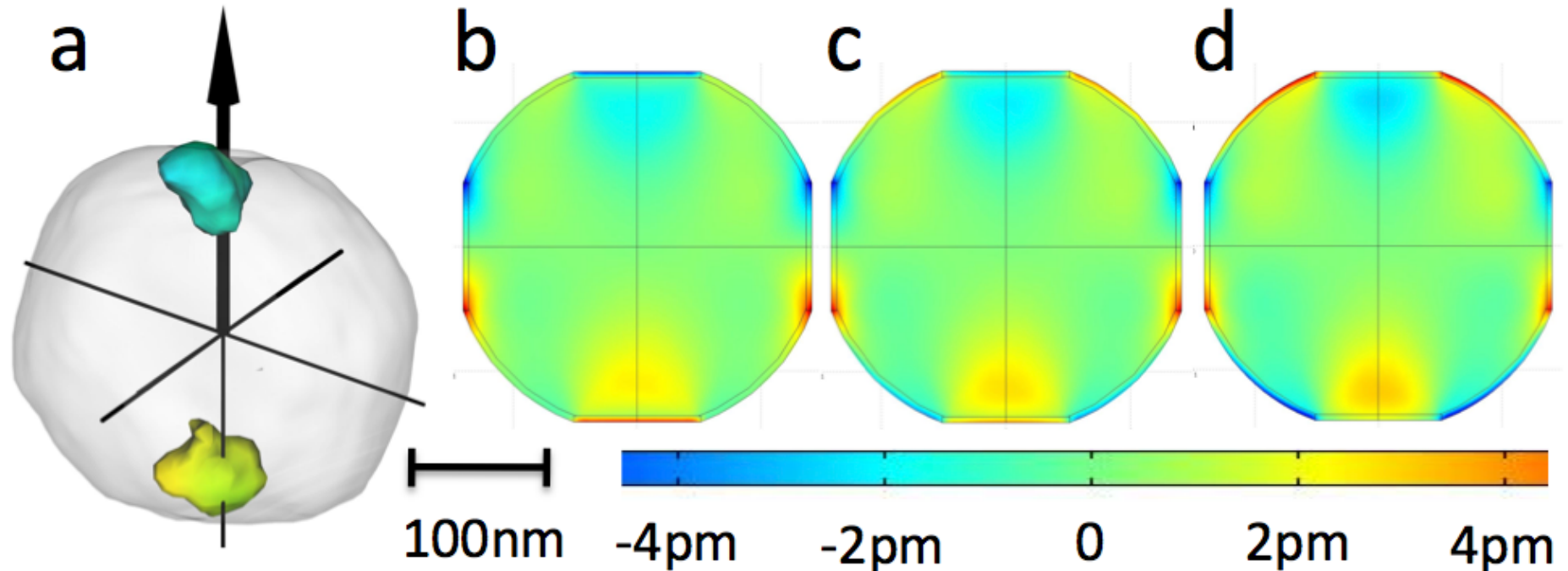
Moyu Watari et al,
Nature Materials (2011)



I. K. Robin

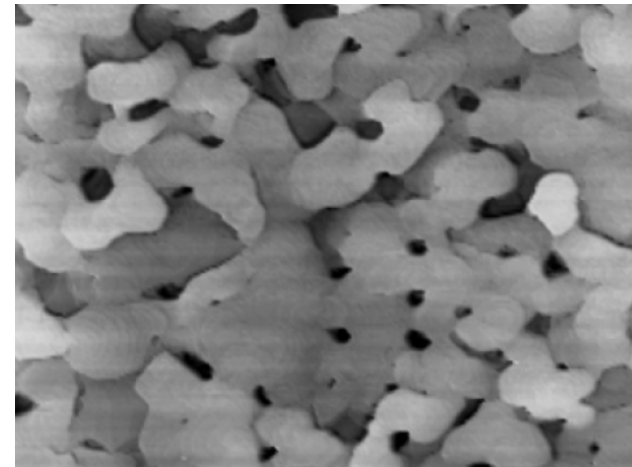
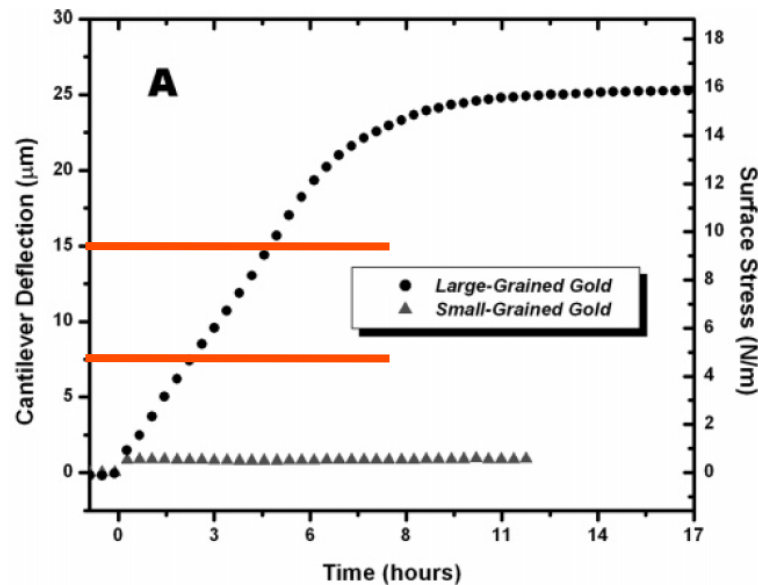
Finite-element Analysis

Differential stress introduced in “skin”
 $\pm 3 \times 10^8$ Pa in 5nm thickness = 1.5N/m



Cantilever strain experiment

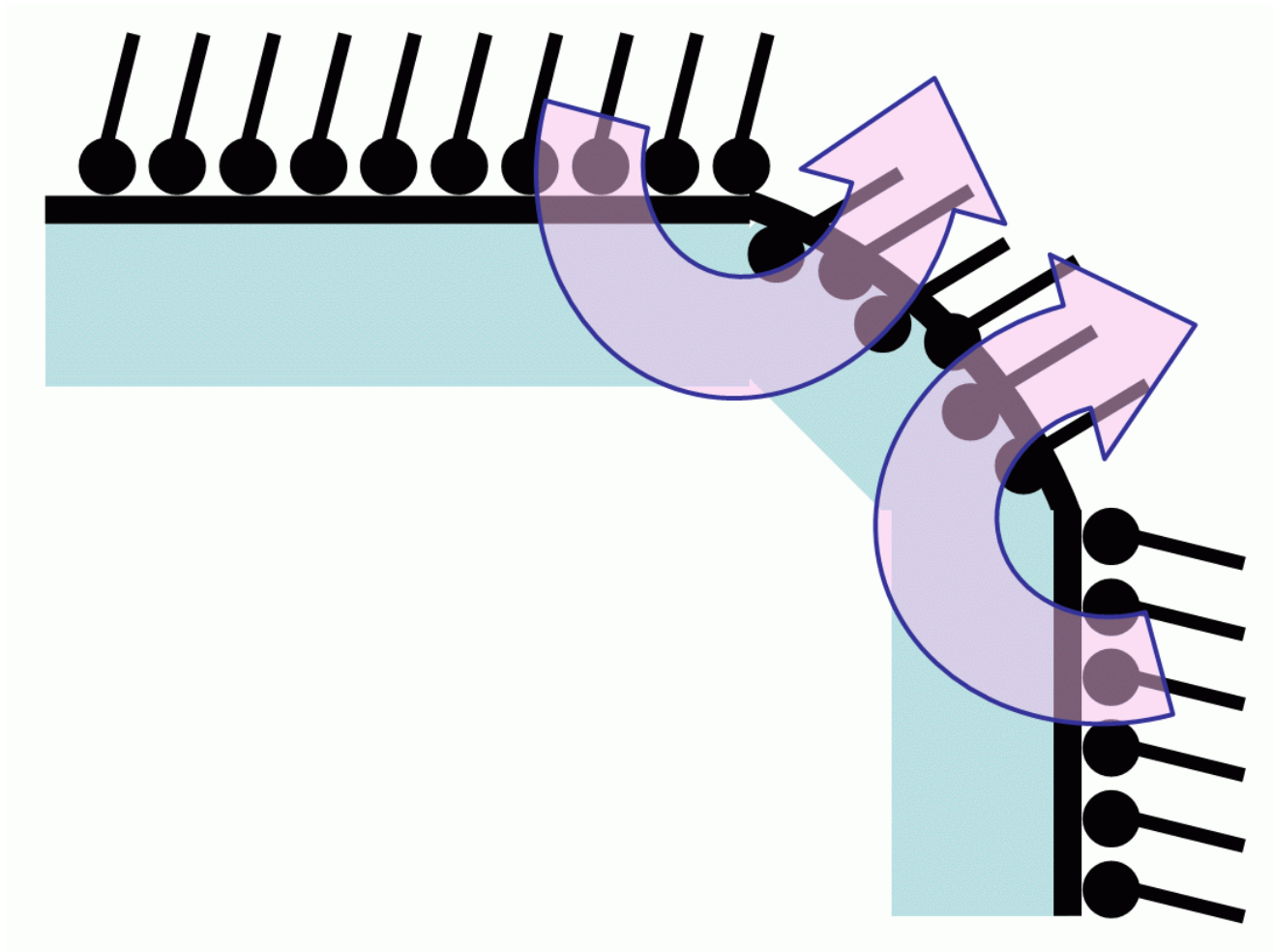
M. Godin et al Langmuir **20** 7090 (2004)



$3 \times 2 \mu\text{m}$ STM image

Calculated surface stress from FEA analysis:
 $4.5 \pm 2 \text{N/m}$ after 1hr, $9.5 \pm 3 \text{N/m}$ after 2hrs

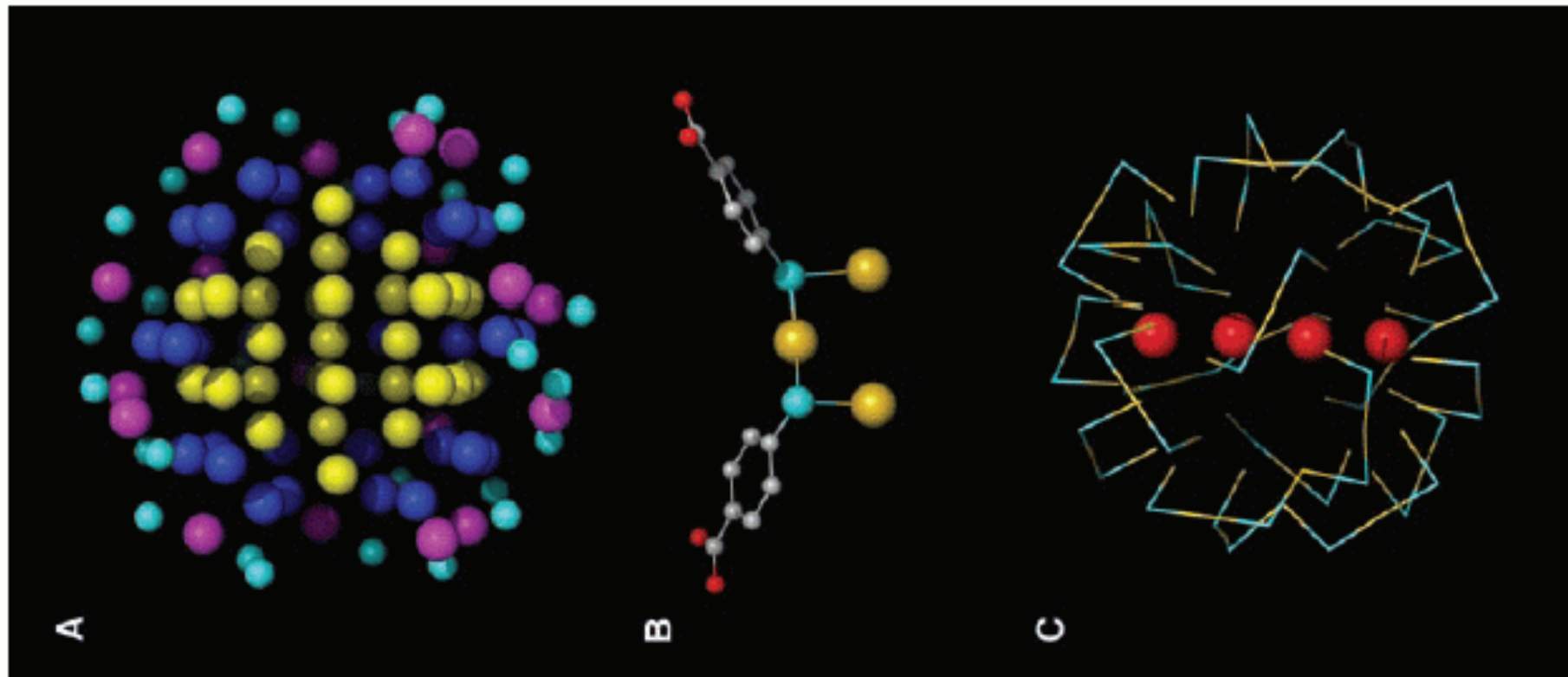
Differential adsorption, thiol/Au

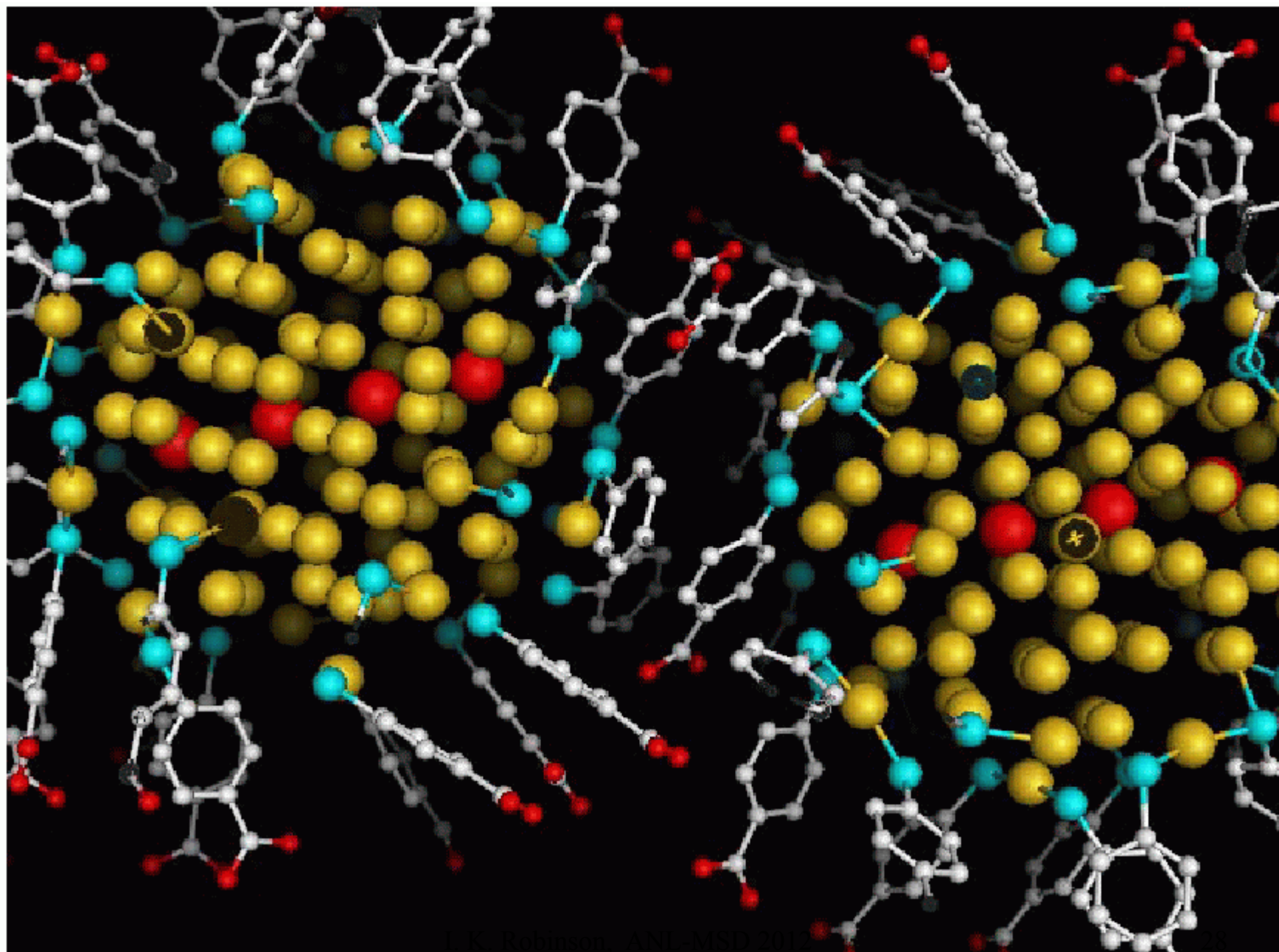


Gold-sulphur network in 1.6nm NC

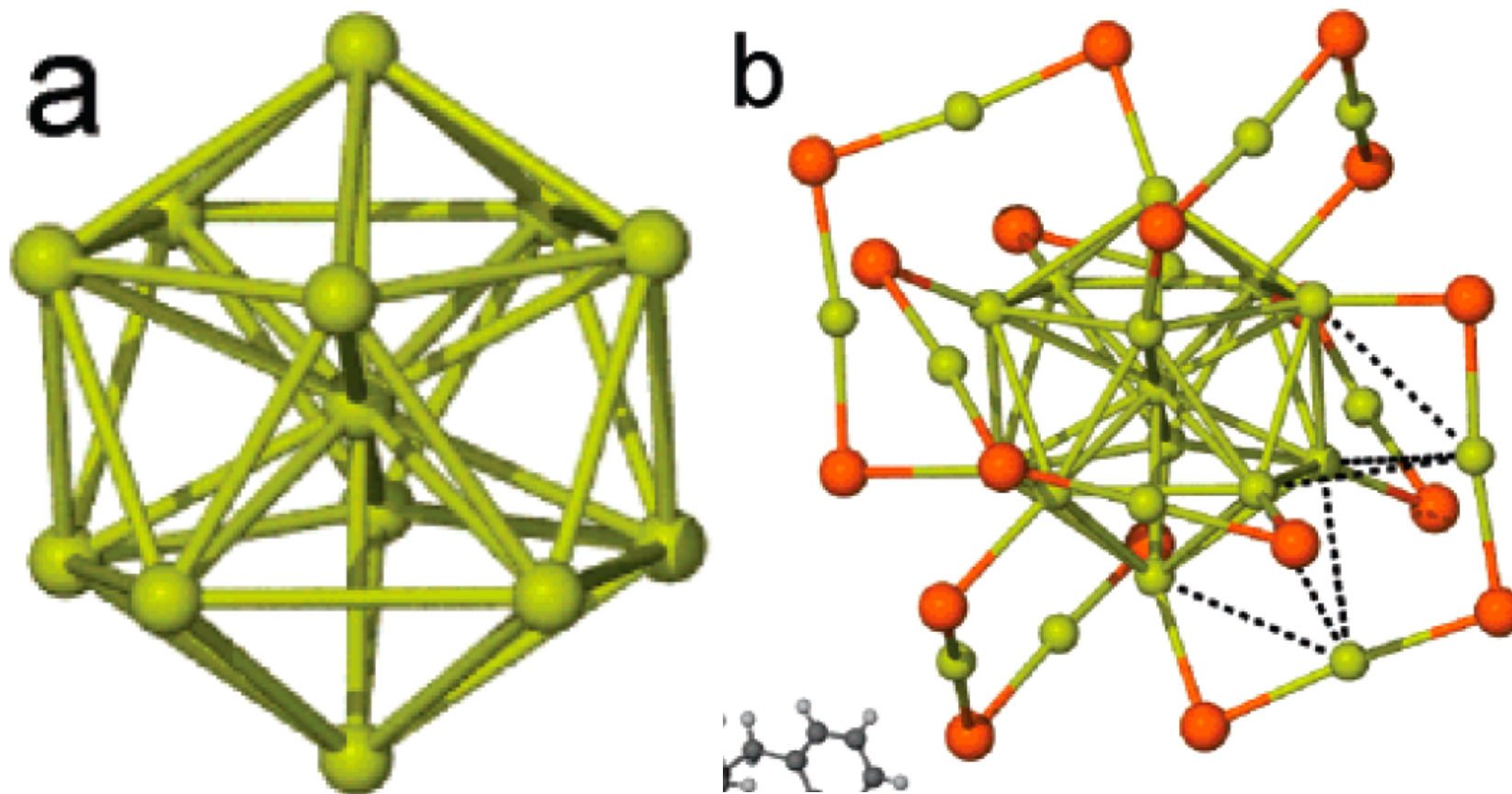
p-mercaptobenzoic acid (p-MBA)

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

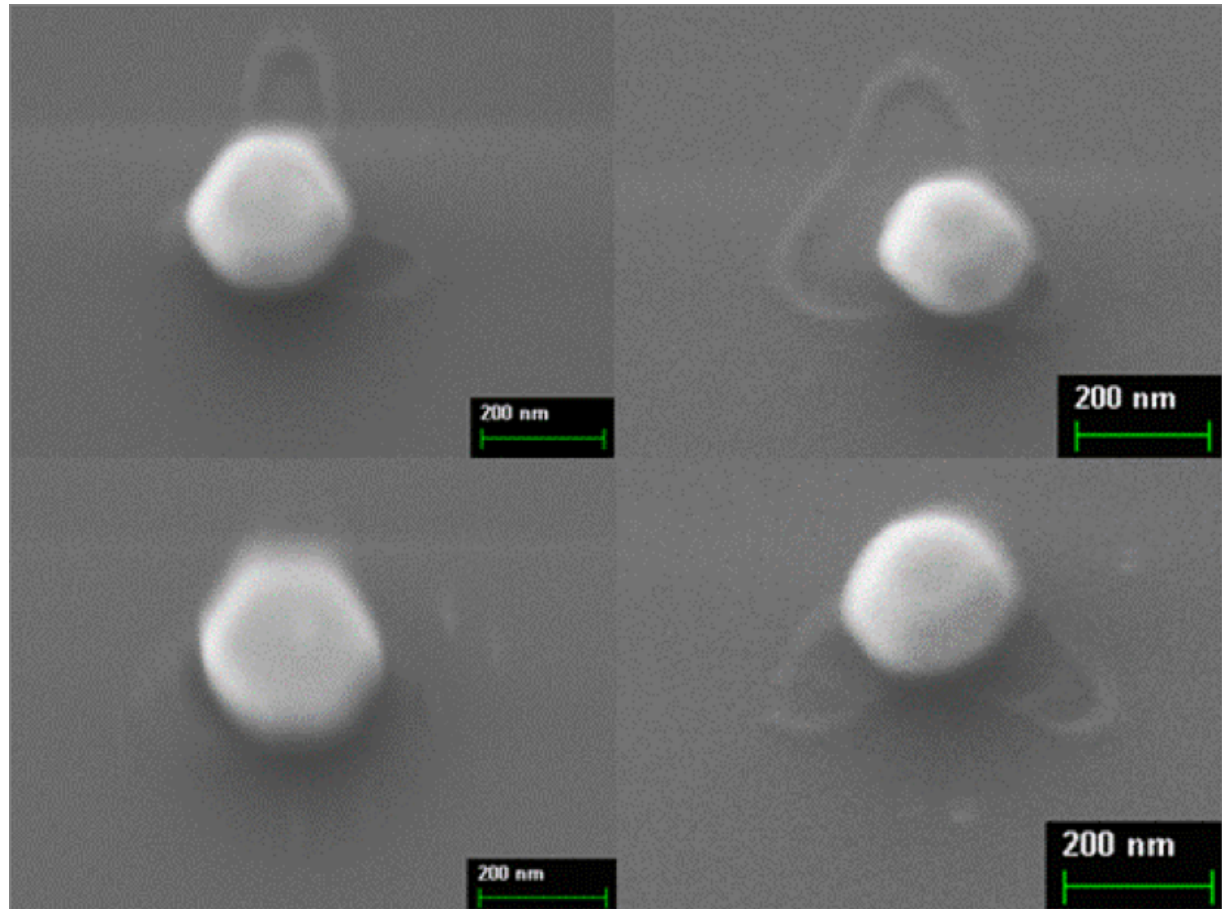
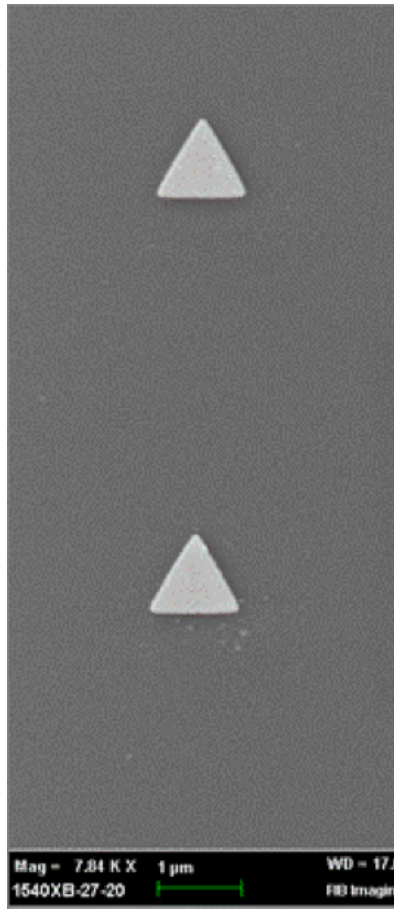




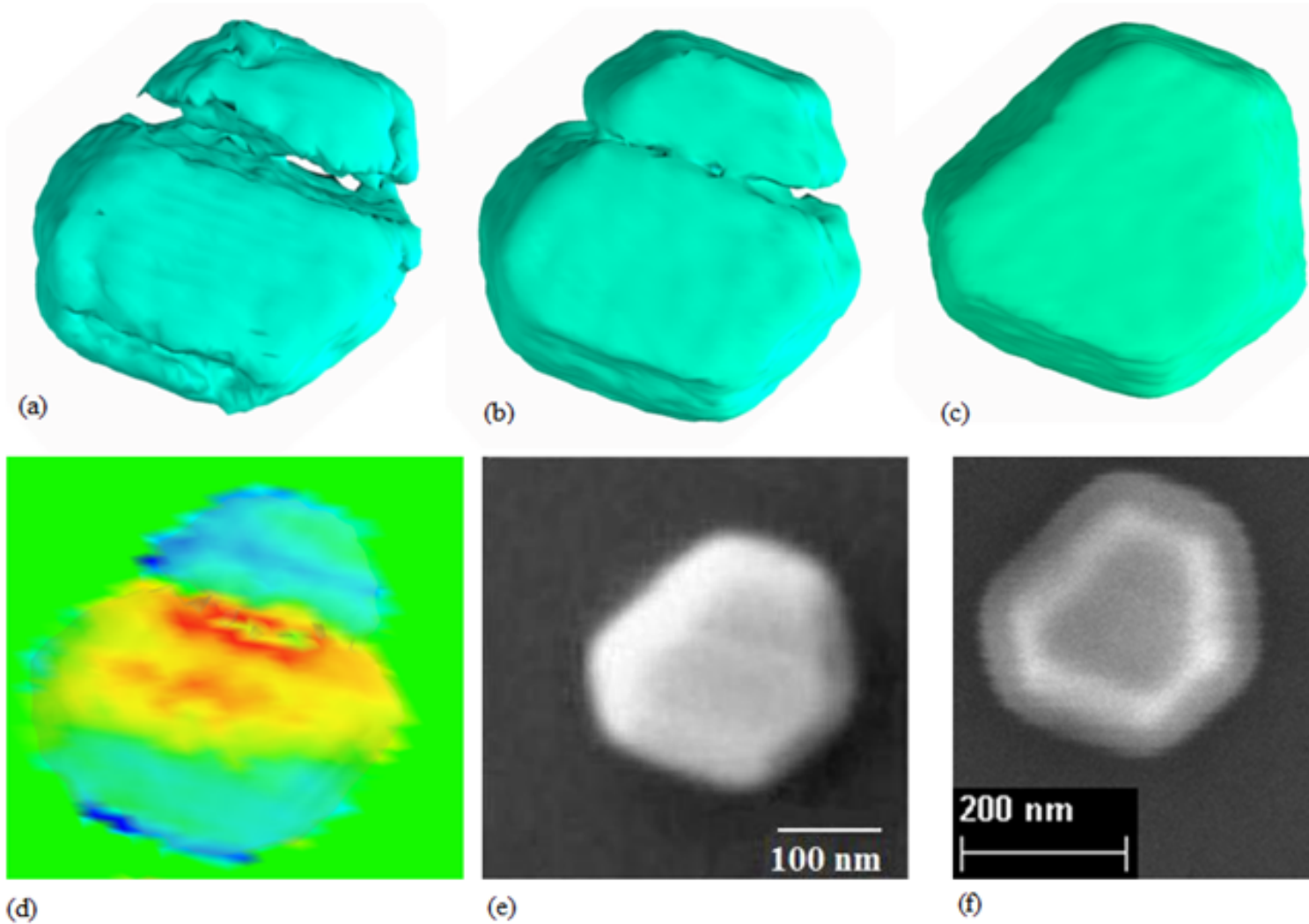
Gold-sulphur structure $\text{Au}_{13}(\text{Au}_2\text{RS}_3)_6$ $=\text{Au}_{25}\text{RS}_{18}$ Heaven et al, JACS 130 3755 (2008)



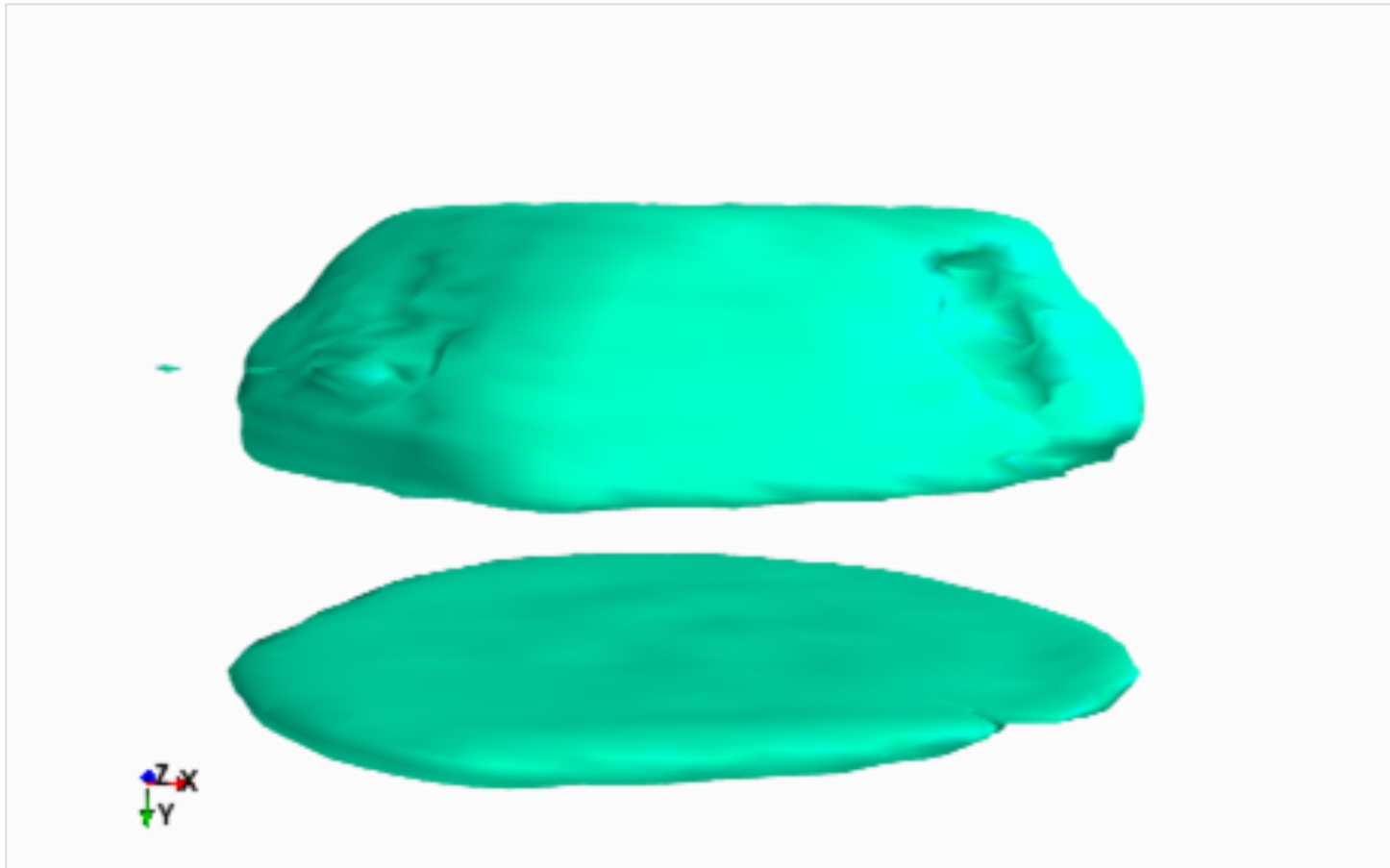
Single Au nanocrystal synthesis



Different Bragg Peaks

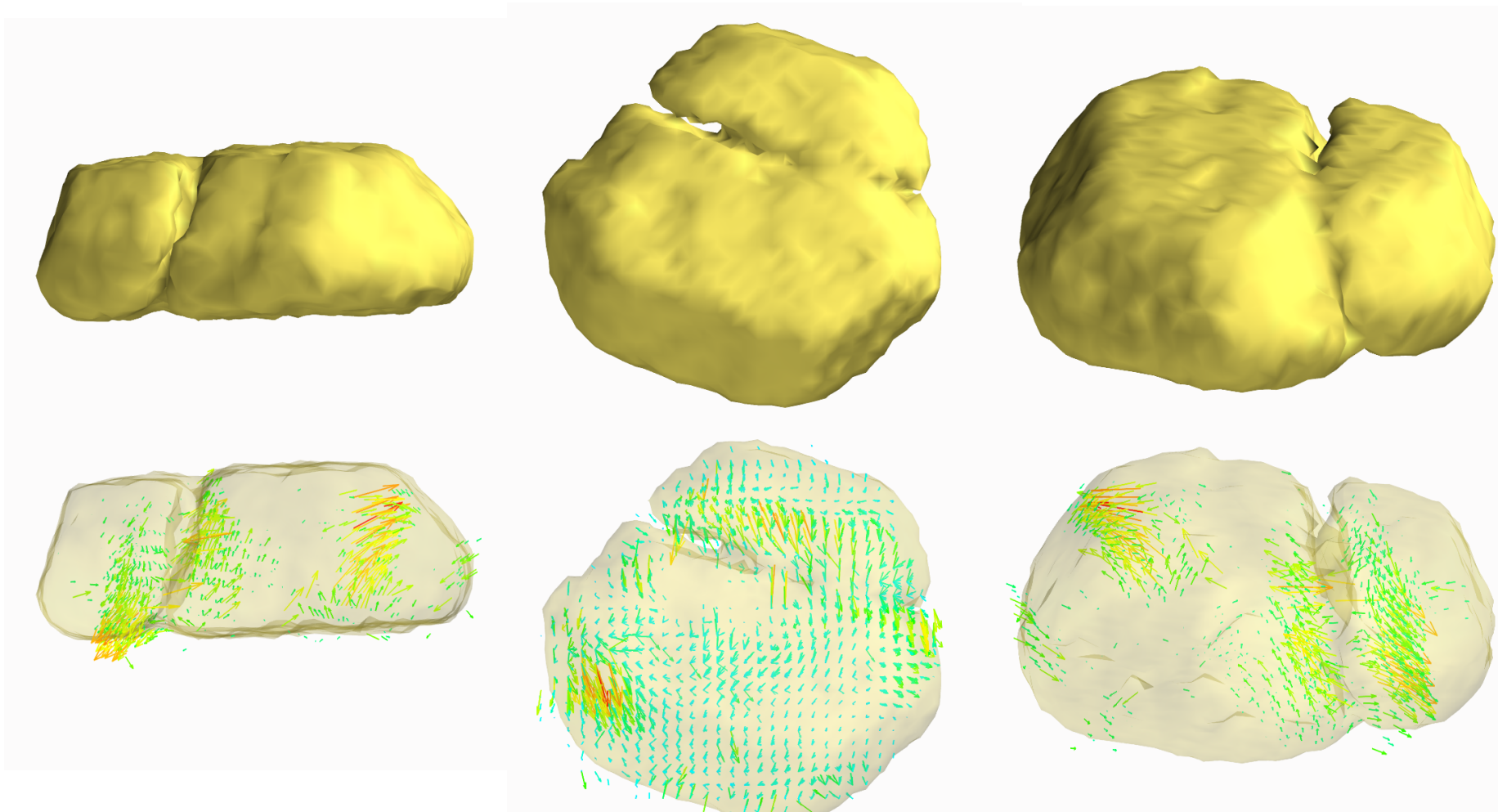


Horizontally Faulted Slab



Vector displacement field

Merged reconstructions from (11-1) (020) and (-111)



Vary transverse coherence

using horizontal entrance slit settings

$12\mu m$

High coherence



Assuming perfect
coherence

$50\mu m$

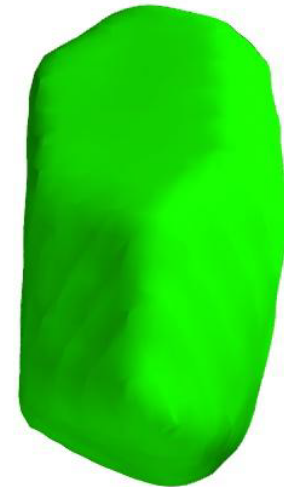
Low coherence



Assuming perfect
coherence

$50\mu m$

Low coherence



Correcting for partial
coherence

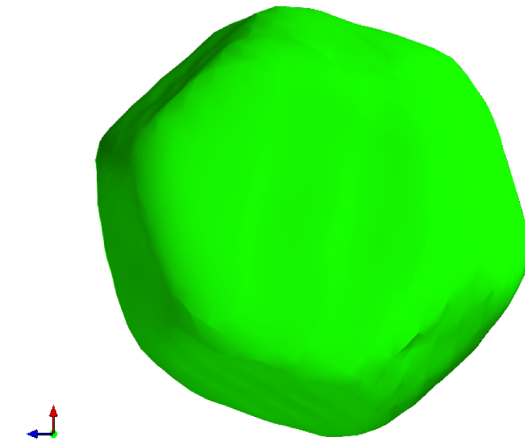
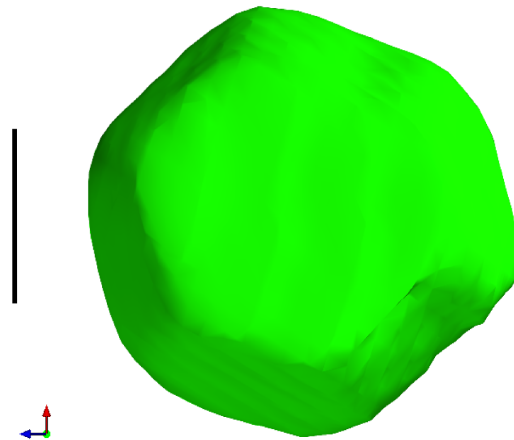
Top view of crystal

Jesse Clark, unpublished

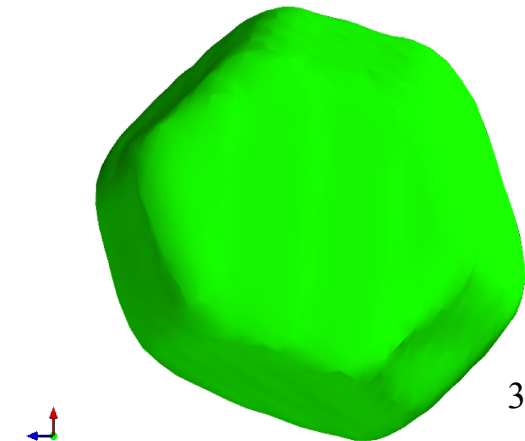
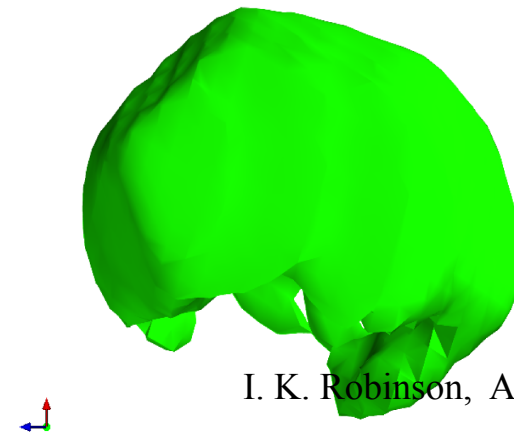
Assuming full
coherence

Correcting for partial
coherence

‘High’ coherence



‘Low’ coherence



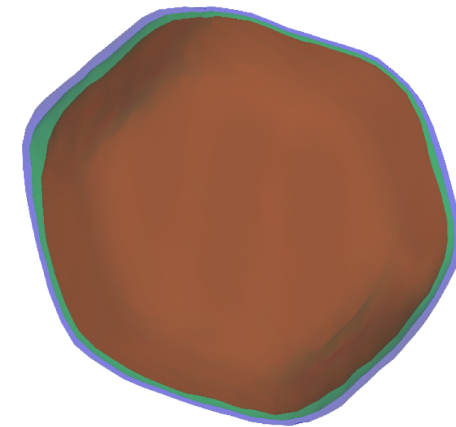
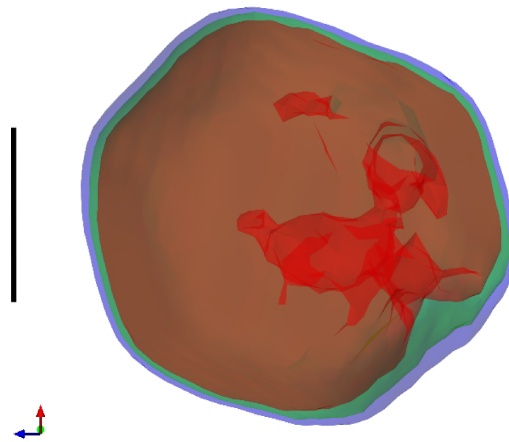
Top view of crystal

Jesse Clark, unpublished

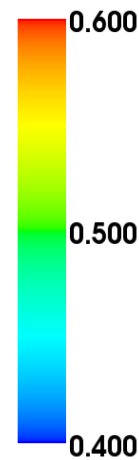
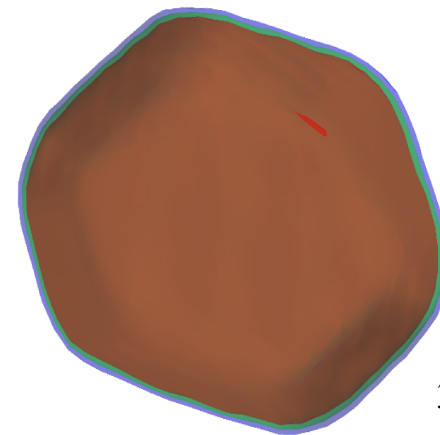
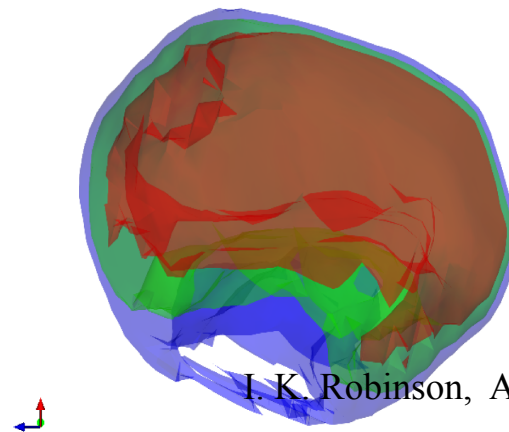
Assuming full
coherence

Correcting for partial
coherence

‘High’ coherence

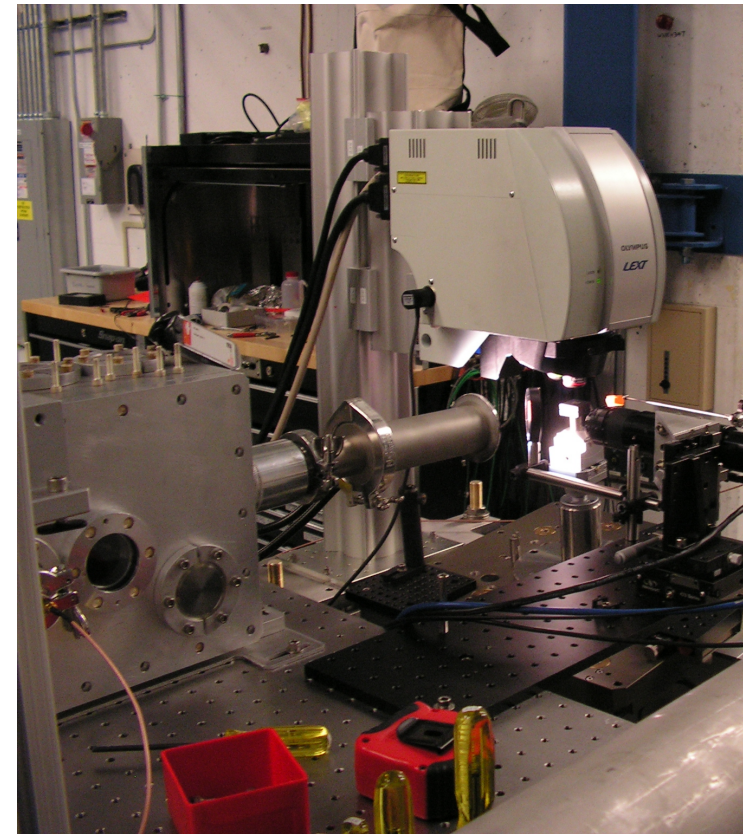
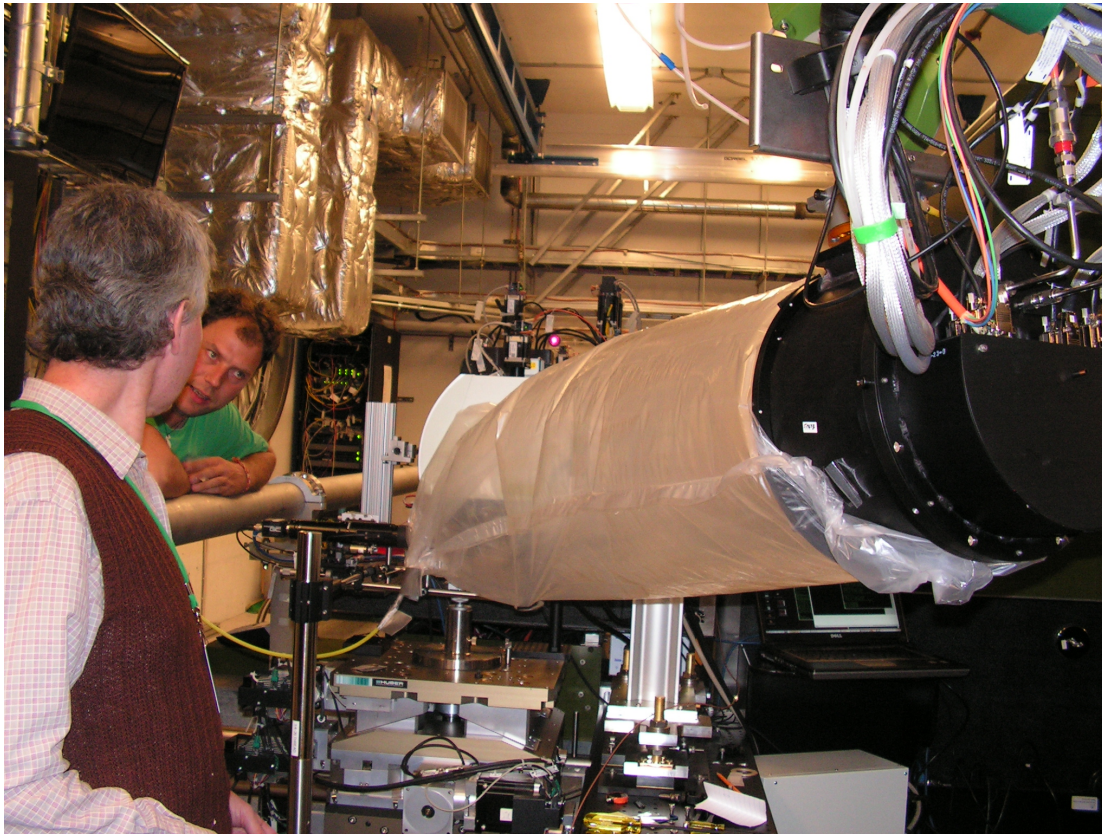


‘Low’ coherence



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

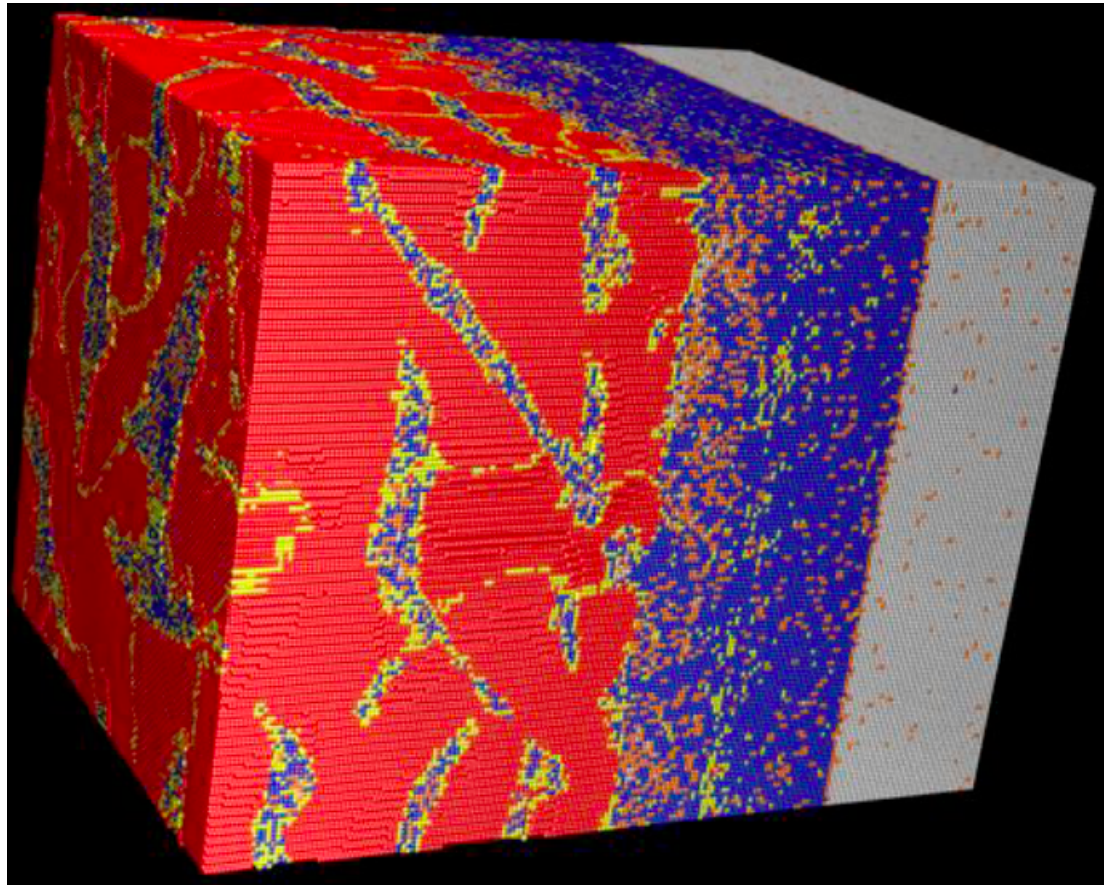


MD simulation of Shock Wave

Damage in Fe along (001) direction

K Kadau, TC Germann, PS Lomdahl, and BL Holian.

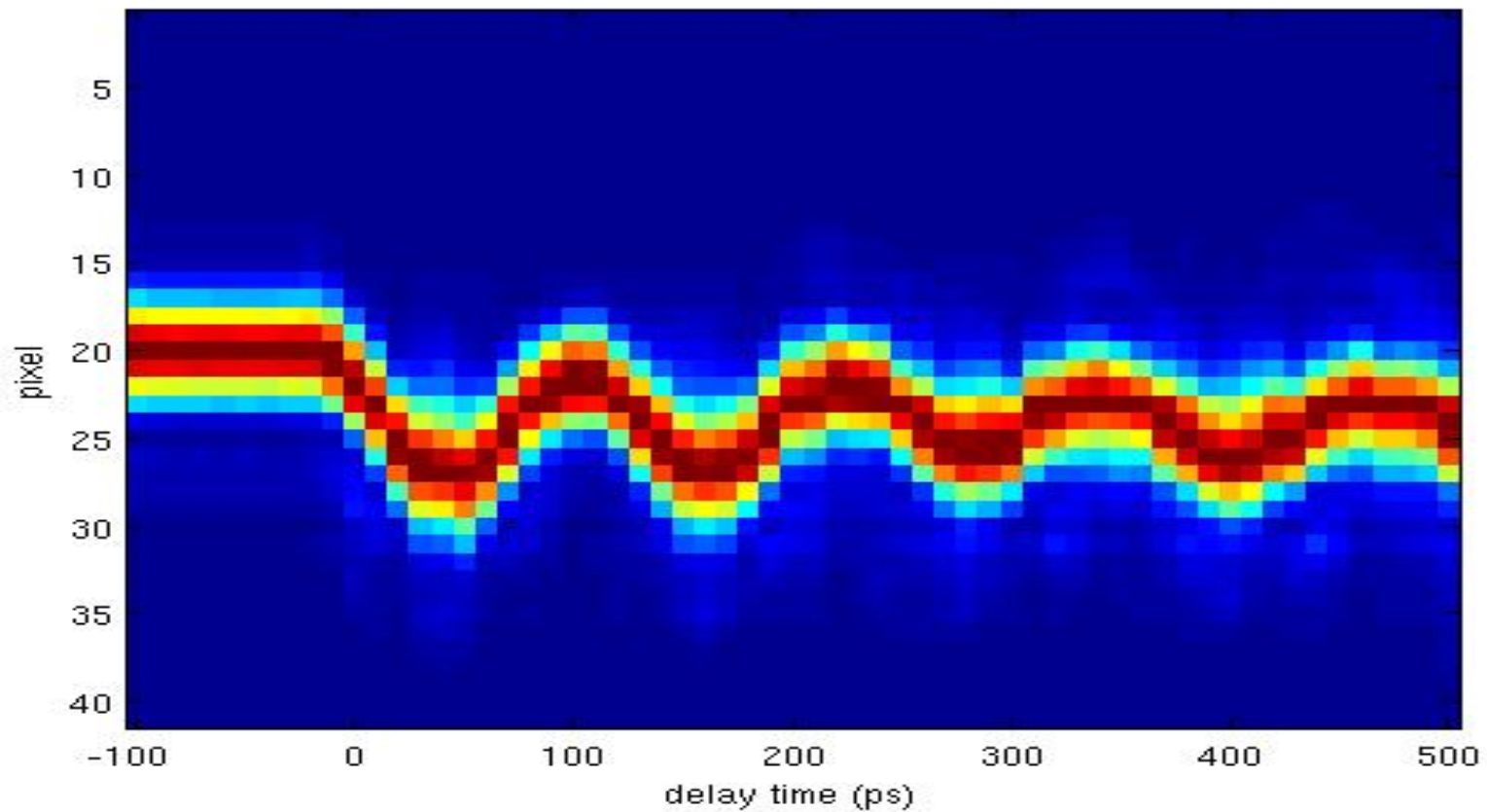
Science, 296 1681 2002



I. K. Robinson, Annecy 2012

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



“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)

(a) Two-temperature model

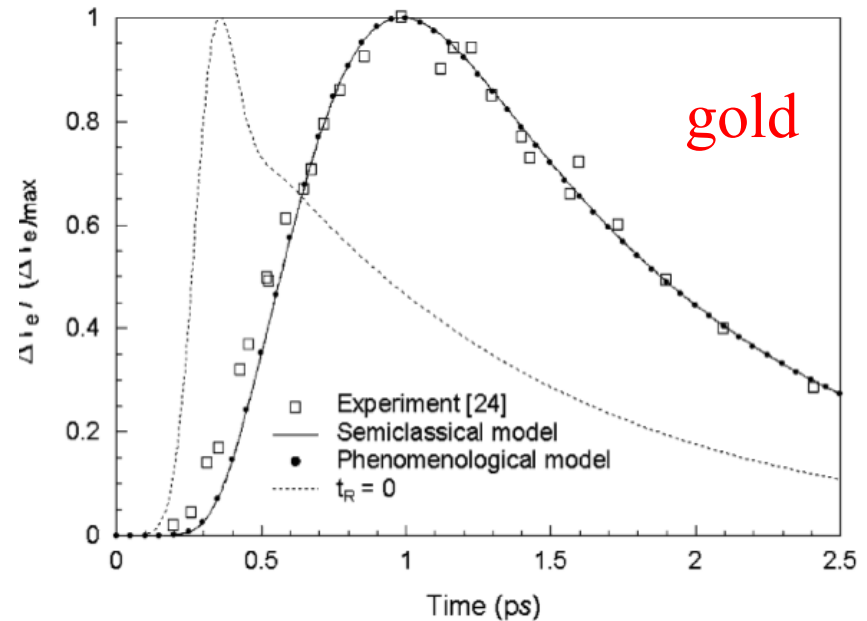
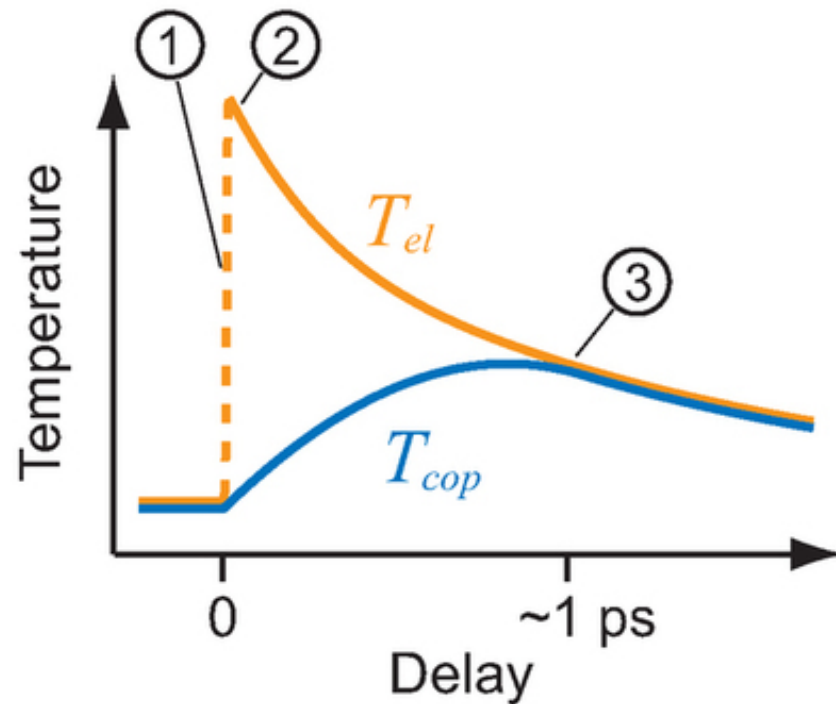
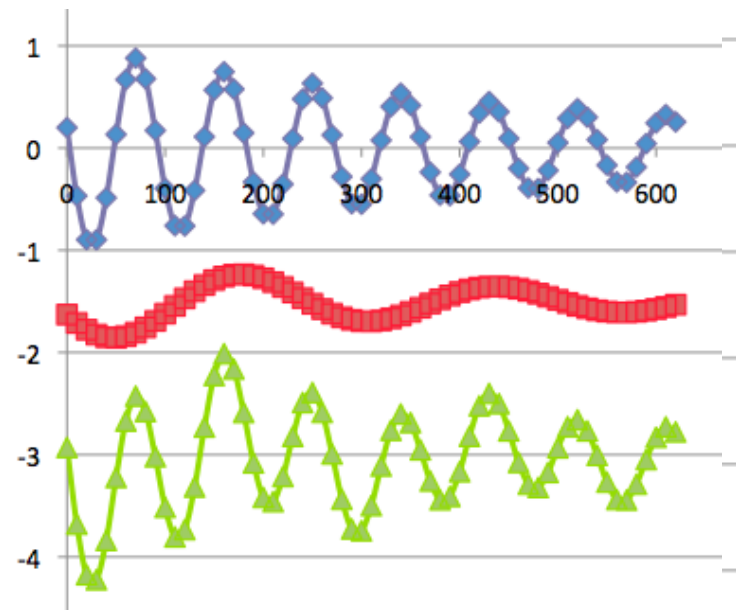
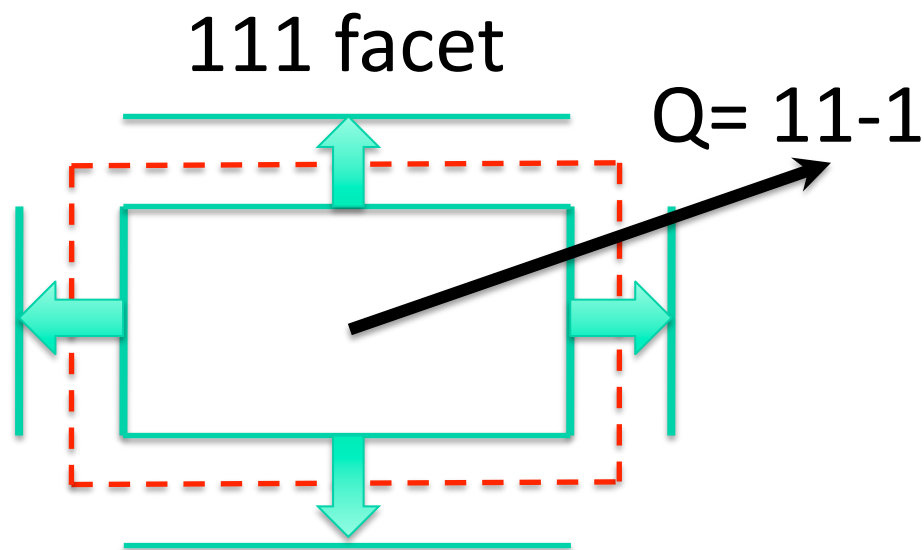


fig. 2. Comparison of the change in electron temperature at the front surface of an 80-nm gold film irradiated by a 2.8 mJ/n², 800 nm, 150-fs laser pulse.

Two Normal Modes of Vibration

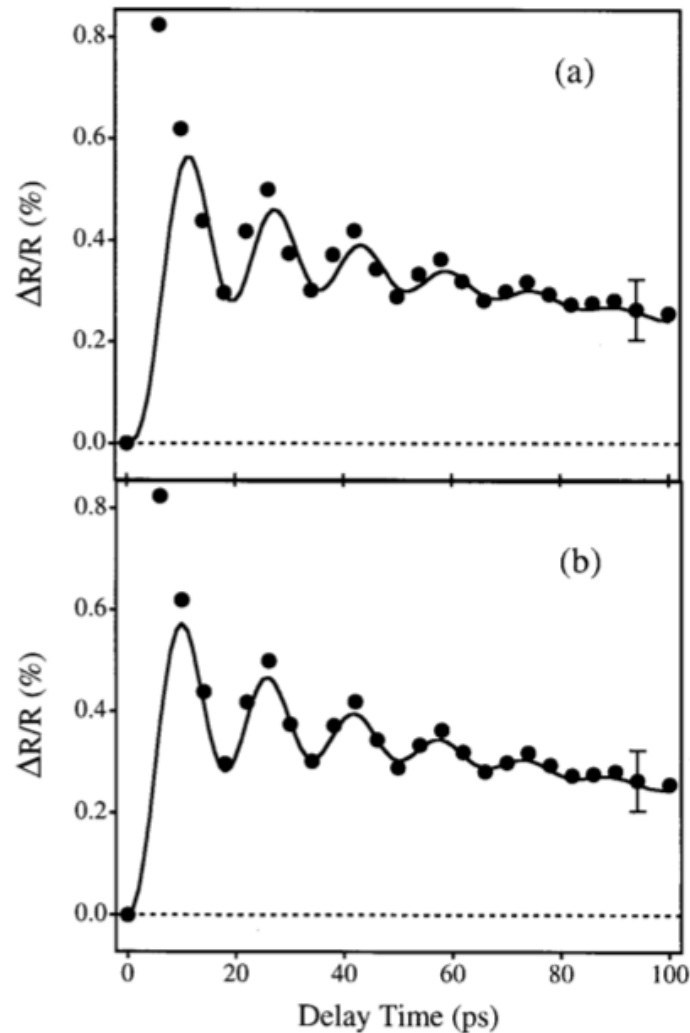
$$S(\tau) = \sum_{n=1}^N A_n \exp[-(\tau/\tau_{d,n})^2] \cos(\omega_n \tau + \varphi_{0,n})$$



$$\begin{aligned} T_1 &= 90\text{ps} & h_1 &= 145\text{nm} & c_s &= 3240\text{ m/s} \\ T_2 &= 259\text{ps} & h_2 &= 420\text{nm} \end{aligned}$$

Ultrafast Absorption Spectroscopy

24nm Au nanoparticles in H₂O, converted to radius
G. V. Hartland, J. Chem. Phys. 116, 8048 (2002)

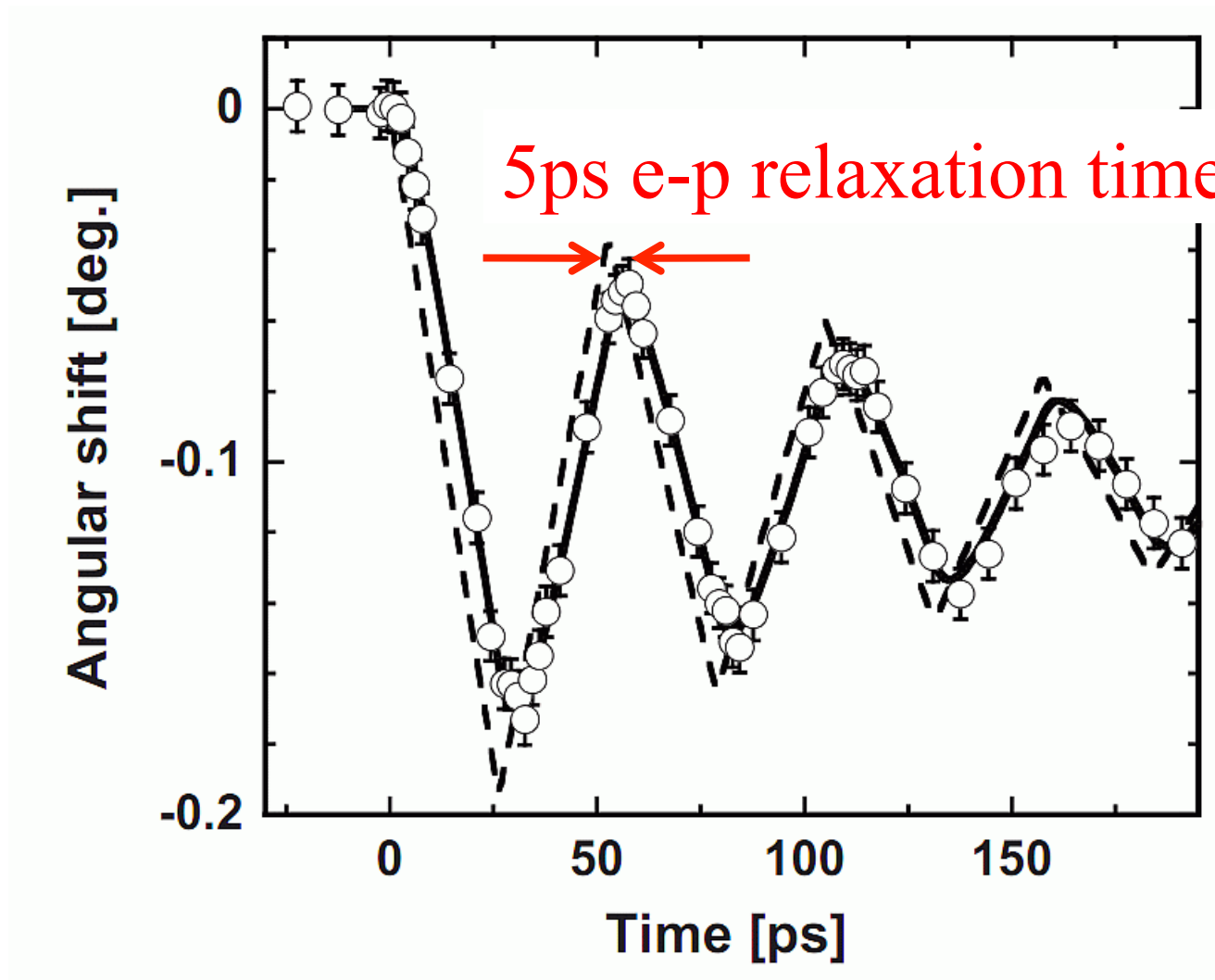


(a) Simple normal mode analysis with impulse heating.

(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)

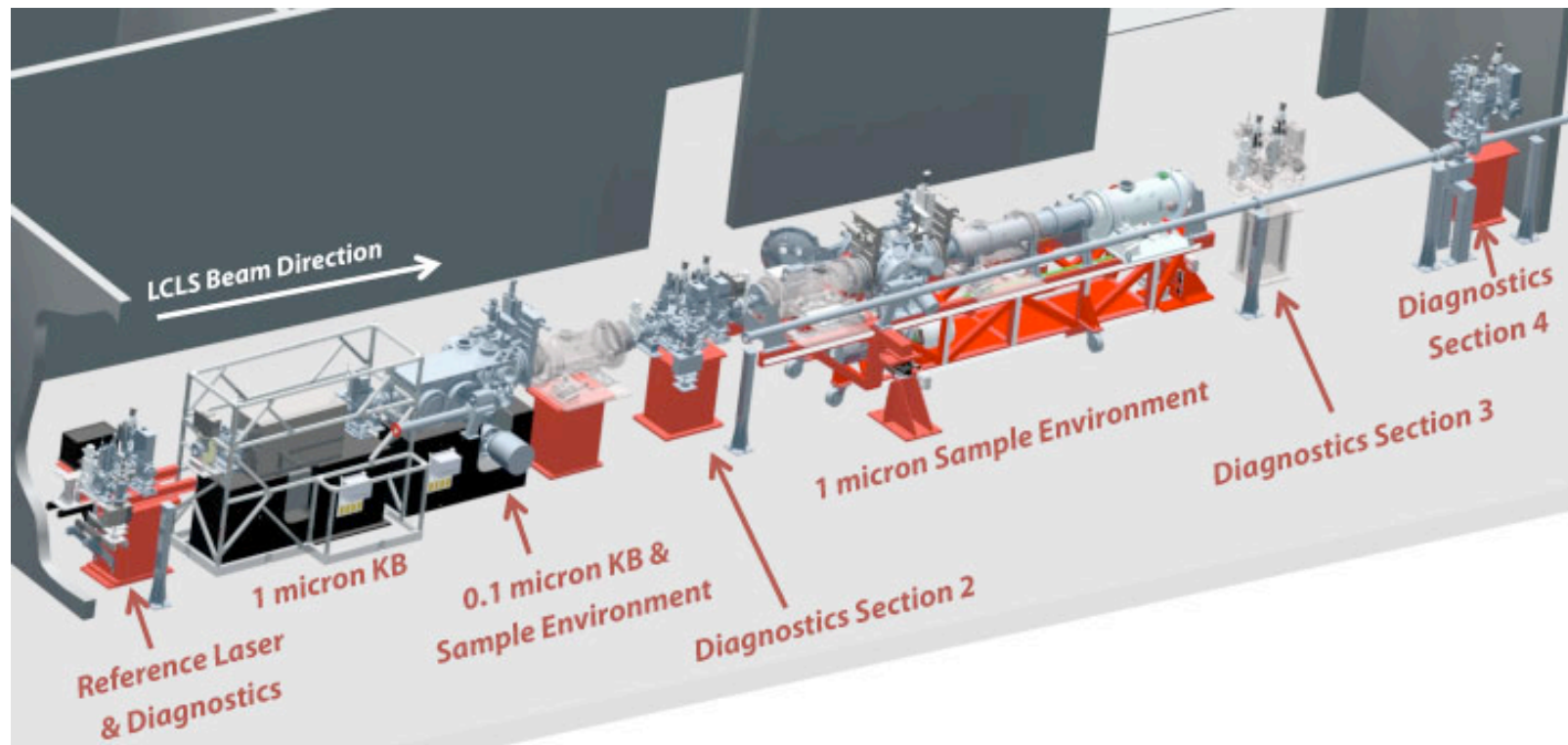


Materials Science using XFELs

- Ground rule #1: \$15,000 per **hour**
- Ground rule #2: fully automated experiment
- Ground rule #3: non-toxic, safe samples
- Ground rule #4: nanoparticles by the kg
- 30,000 pulses/sec at E-XFEL, 3000x10
- up to 2000 frames/sec data rate = TB/hr

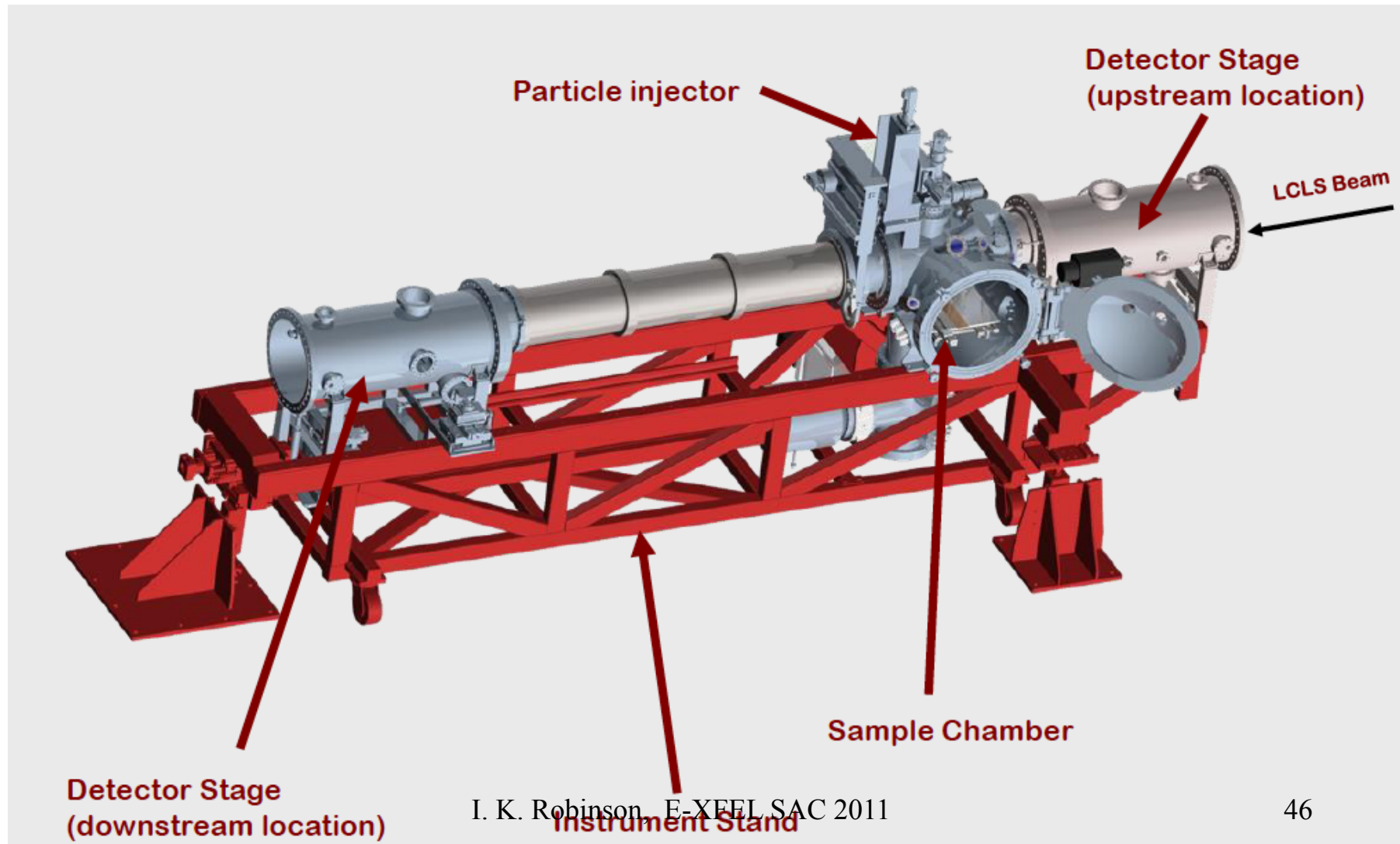
CXI instrument at LCLS

Sebastien Boutet and Garth Williams



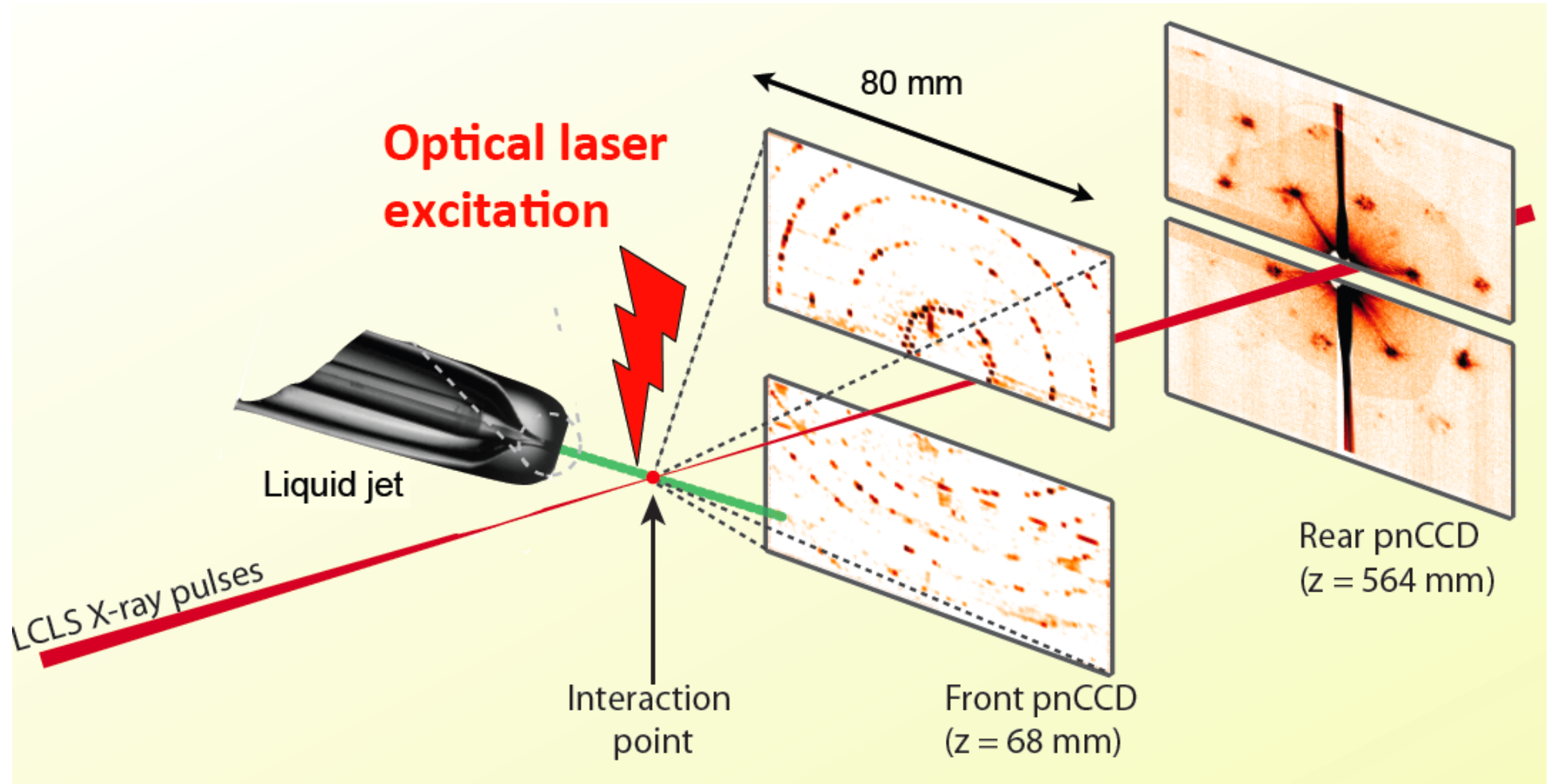
CXI “1 μ m” instrument at LCLS

Sebastien Boutet and Garth Williams



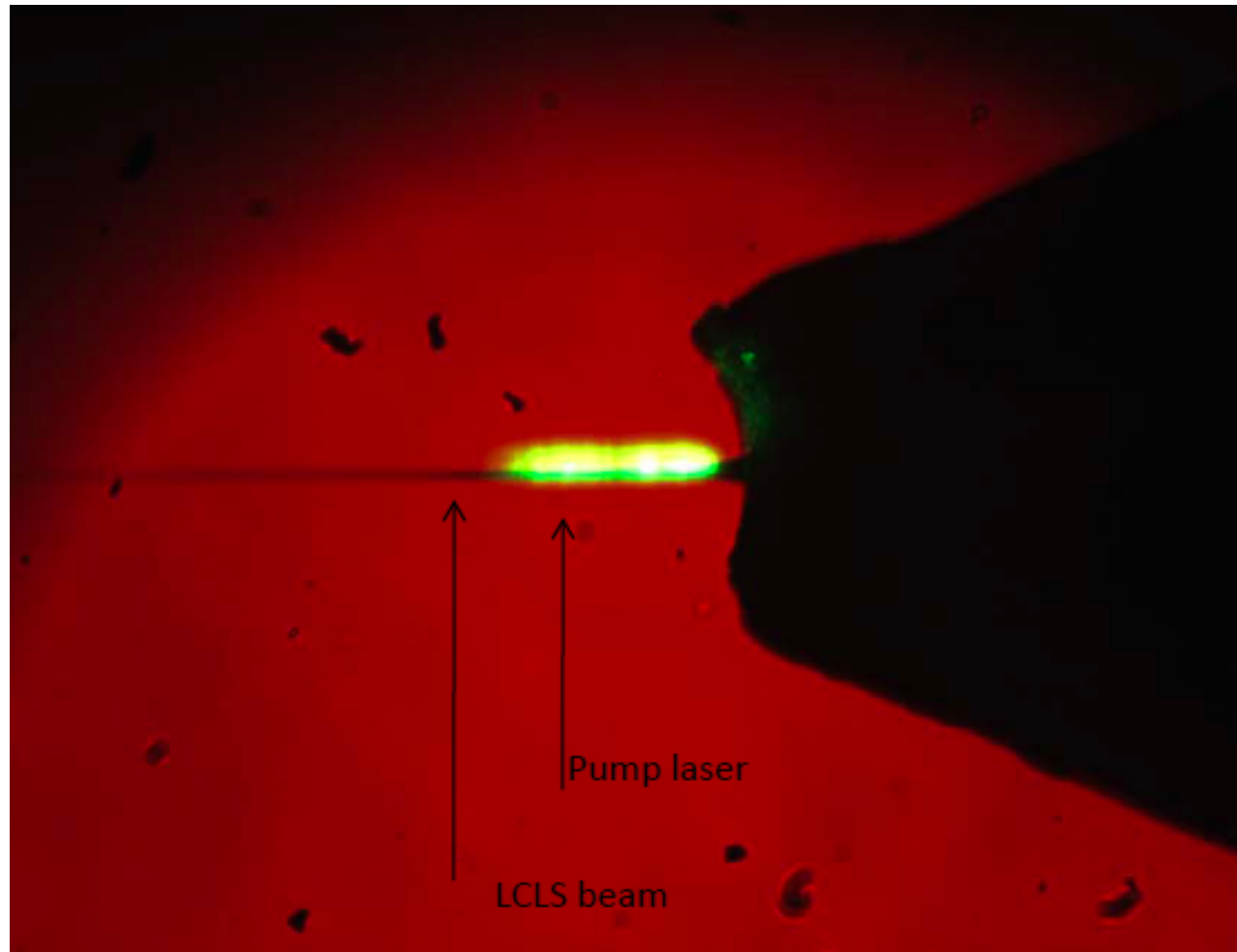
Pump-probe schematic

Henry Chapman, Bio-XFEL workshop Jan 2011



Pump-probe demonstration

John Spence, Bio-XFEL workshop Jan 2011



- Ferredoxin nanocrystals
- Time delay 0-10 us
- 532nm 7ns 50uJ laser
- 100um focal spot (fibre)

Materials Science using XFELs

- Nanoparticles injected into X-ray beam
- Veto frames where no “hit” or diffraction
- Pump-Probe has two state variables
 - Optical fluence = sample temperature
 - Delay time after ‘instantaneous’ heating
- Pressure accessible using “tamper”
- Explore phase diagrams eg spinodals
- Nanoparticle sample format = synthesis

Coherent x-ray diffraction (CXD)

- Complex density can image strain
- Strain associated with nano-shape
- Differential strain under thiols
- Partial coherence correction
- Time resolved strain patterns
- Materials Science by XFEL?