

Coherent Diffraction Imaging and other uses of Synchrotron X-ray Diffraction

Ian Robinson

Jesse Clark

Ross Harder

Xiaojing Huang

Moyu Watari

Gang Xiong

Xiaowen Shi

Maria Civita

London Centre for Nanotechnology
Research Complex at Harwell

Advanced Characterisation of
Materials CDT
February 2015

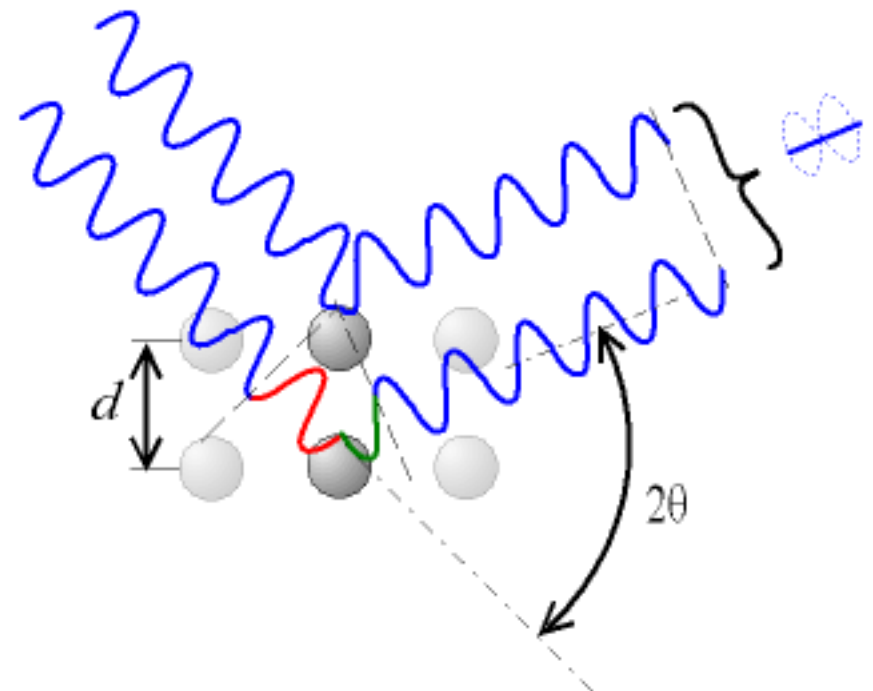
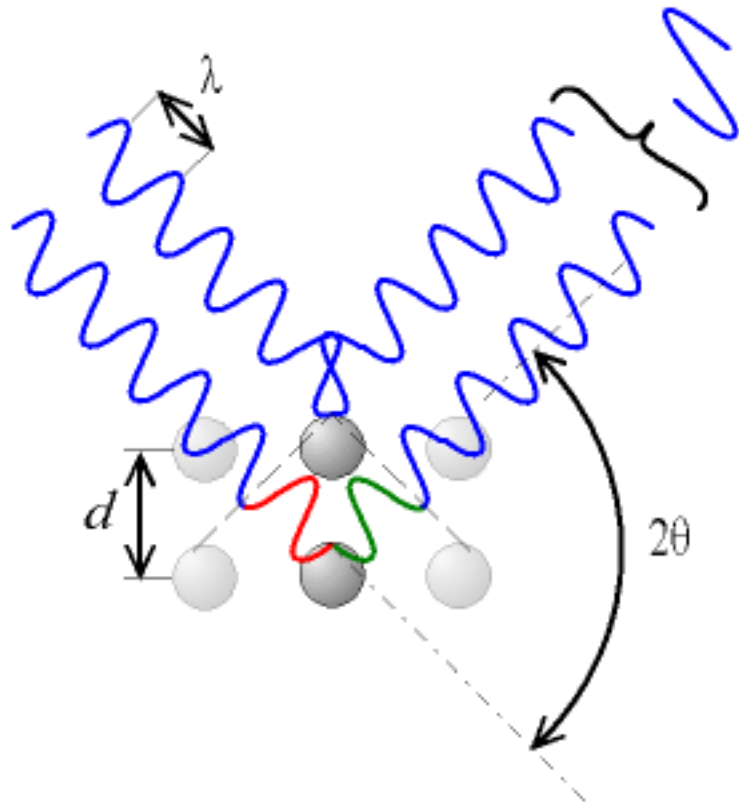
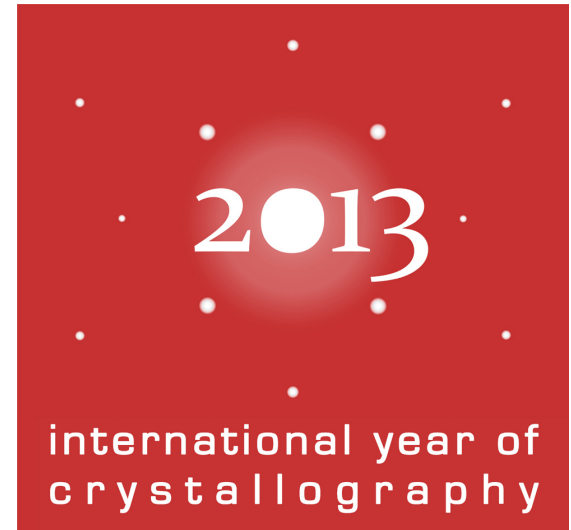
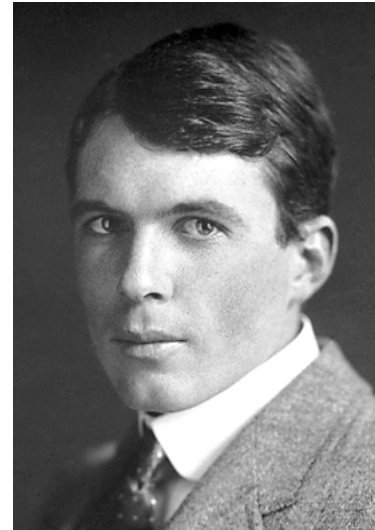
Outline

- X-ray Diffraction
- Diamond Light Source
- Nanocrystal structures
- Coherent X-ray diffraction
- Crystal strain as complex density
- Strain induced by surface reactions
- Ultrafast snapshots of moving matter

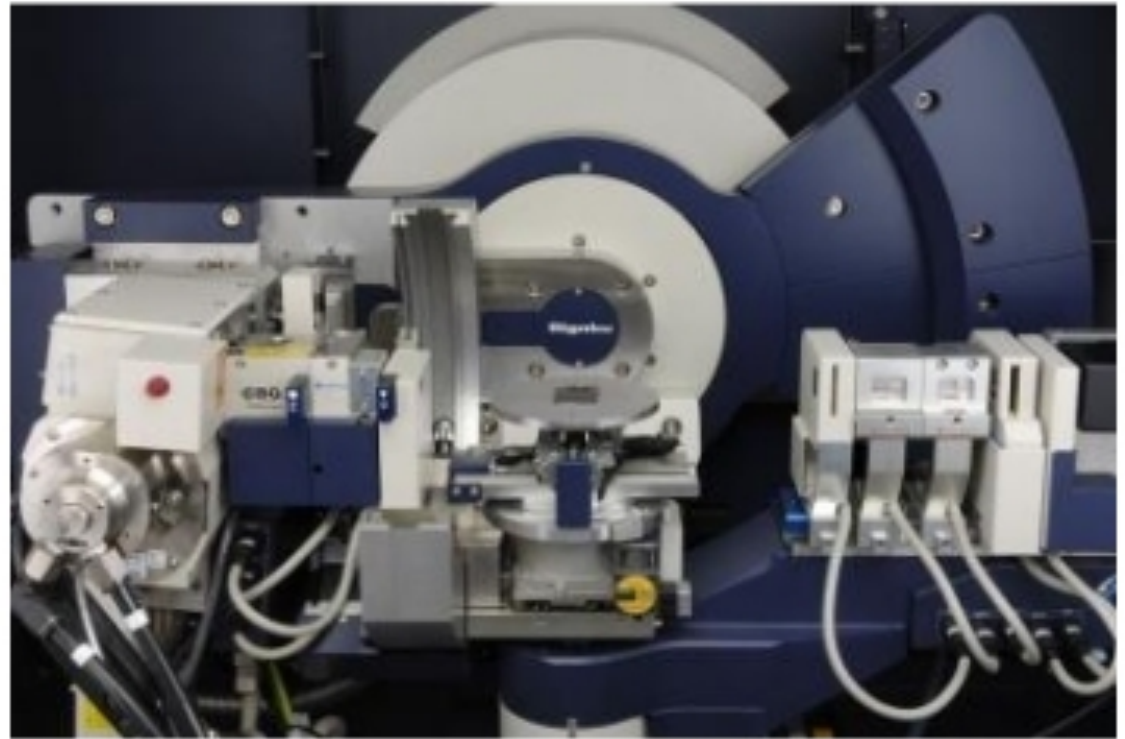
Bragg Law 1913

$$2d\sin\theta = \lambda$$

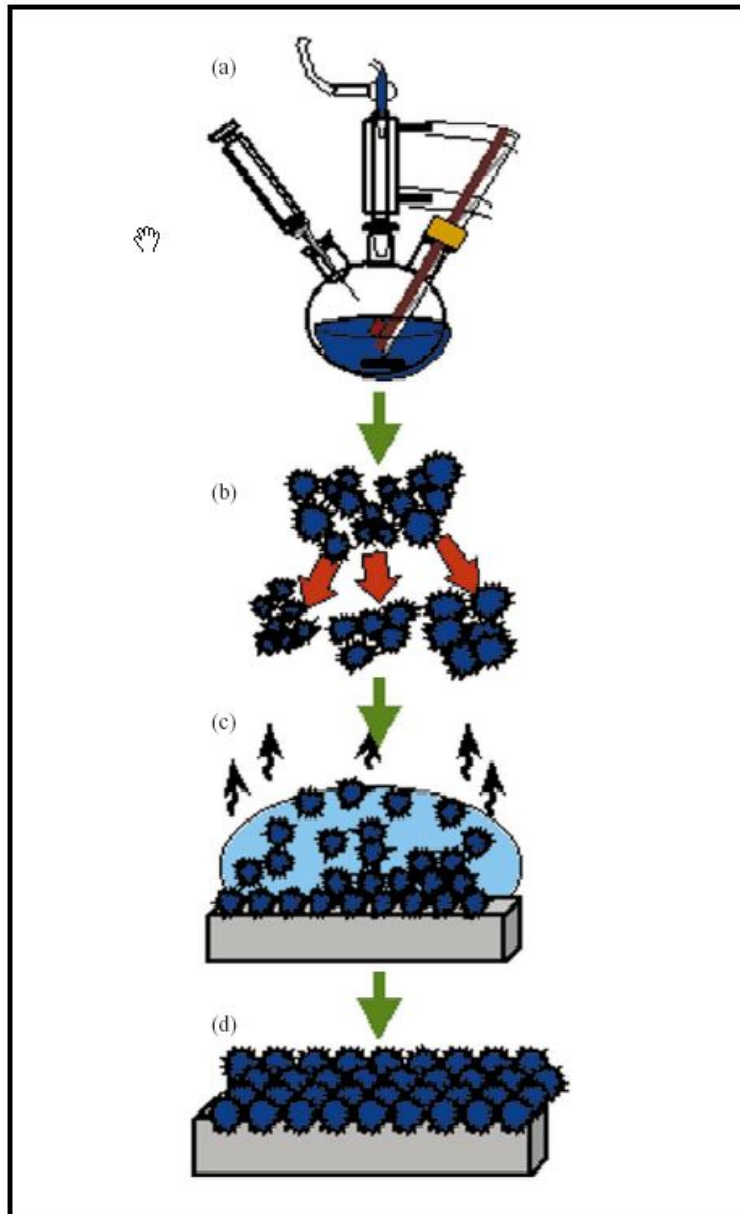
C D Ngoc Chan, Wikipedia



Rigaku Smart-Lab Diffractometer F-10 Physics

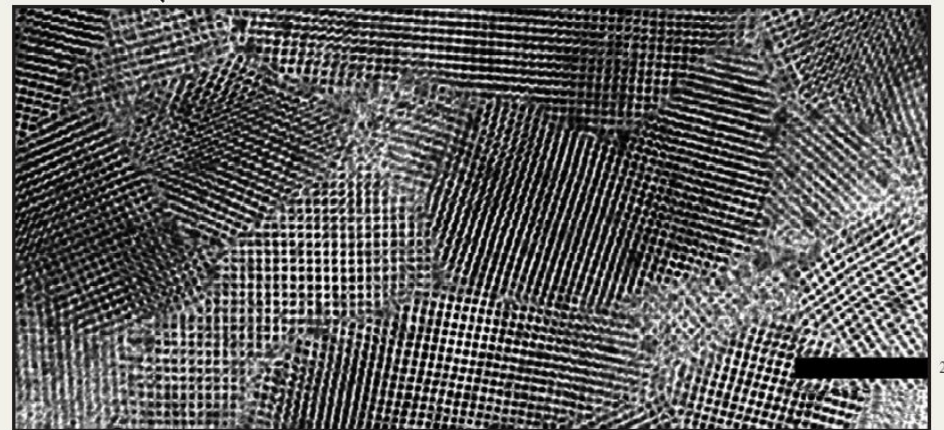


Chemical Synthesis of Nanocrystals



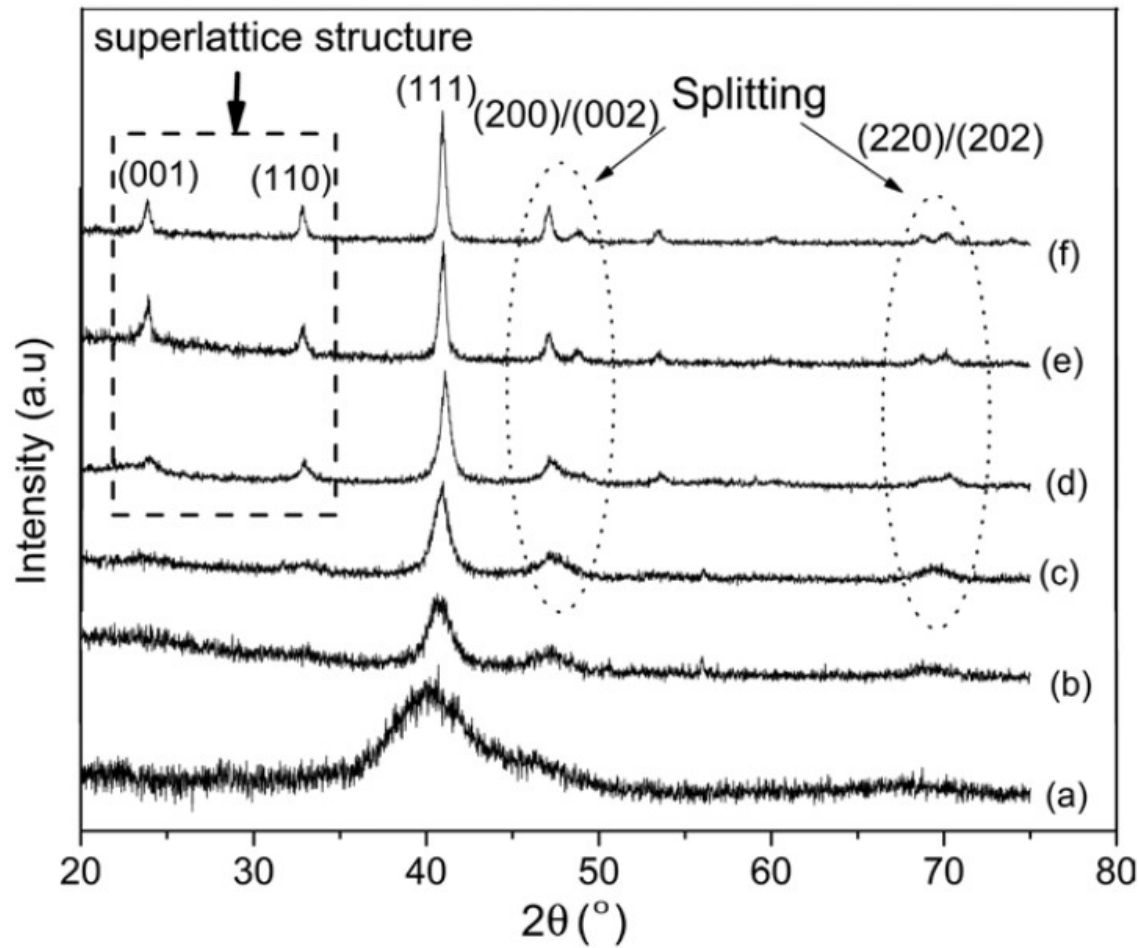
- Reactants introduced rapidly
- High temperature solvent
- Surfactant/organic capping agent
- Square superlattice (200nm scale)

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



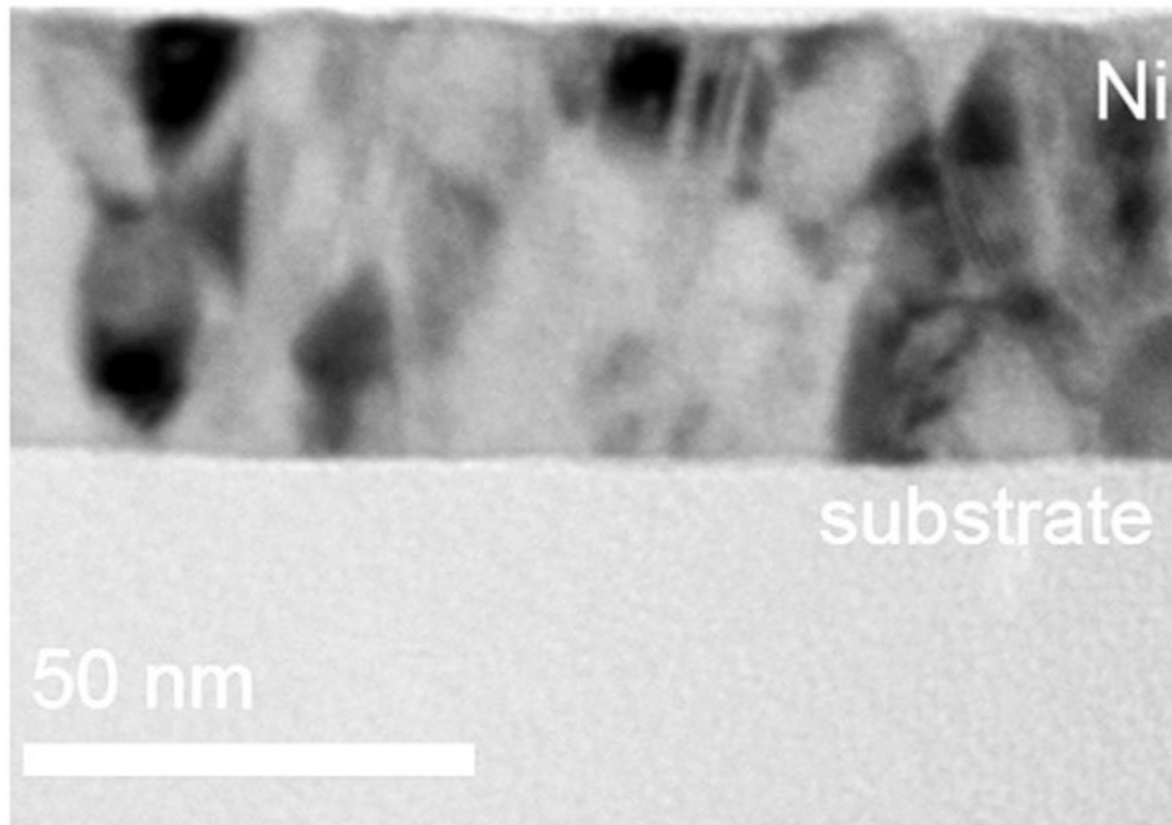
Nanostructured FePt alloy

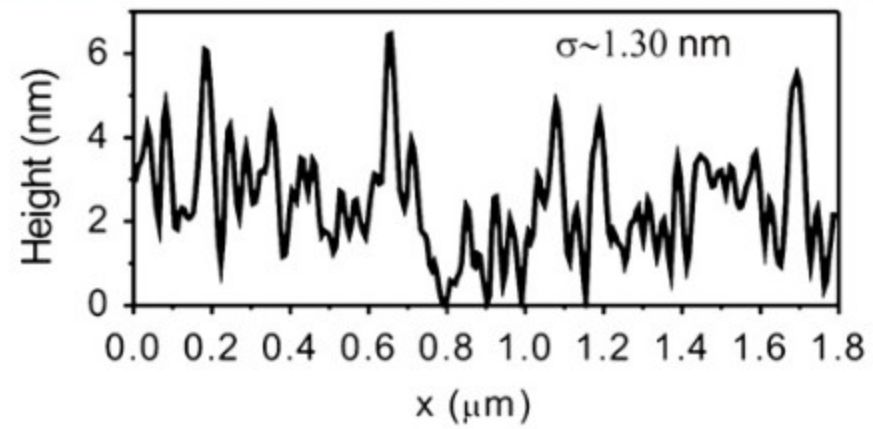
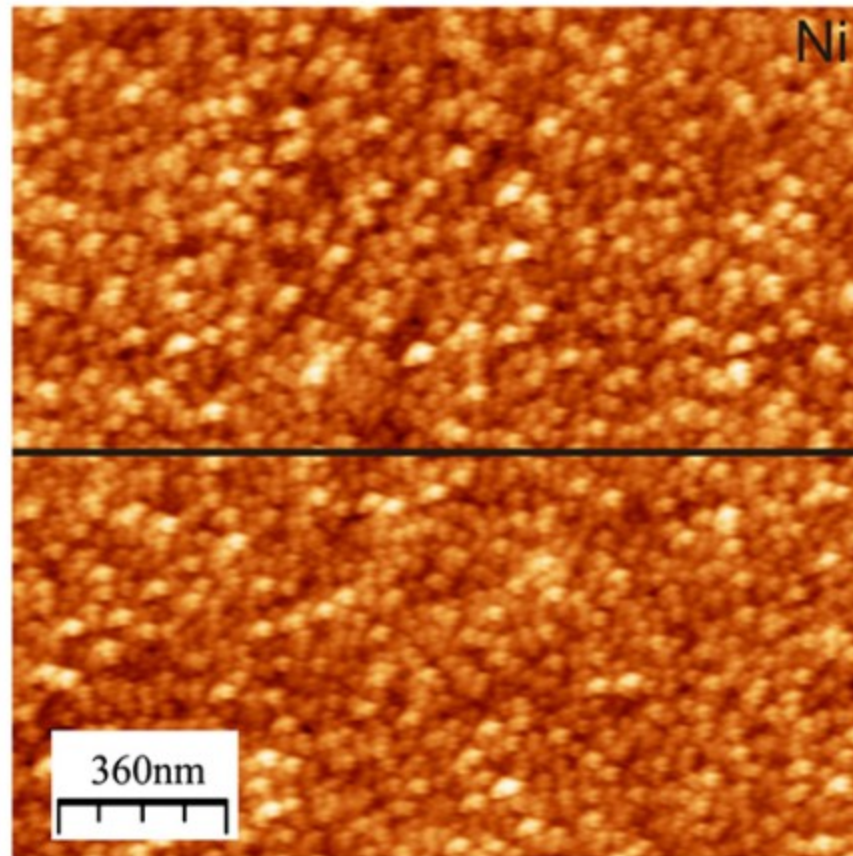
R. Medwal et al, J. Phys. D 45 055001 (2012)



Lattice parameter of nanocrystalline Ni

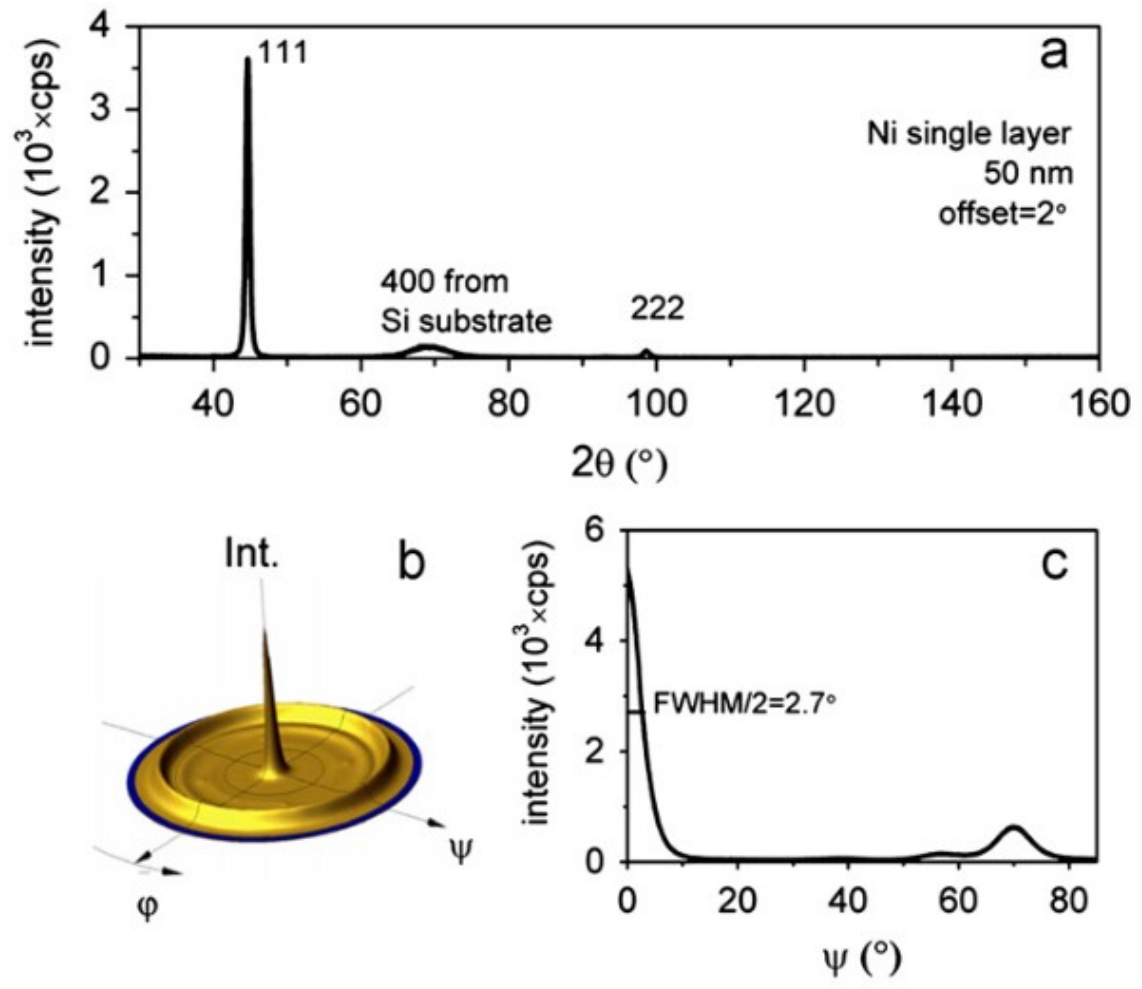
J. Sheng et al, Physica E 43 1155 (2011)





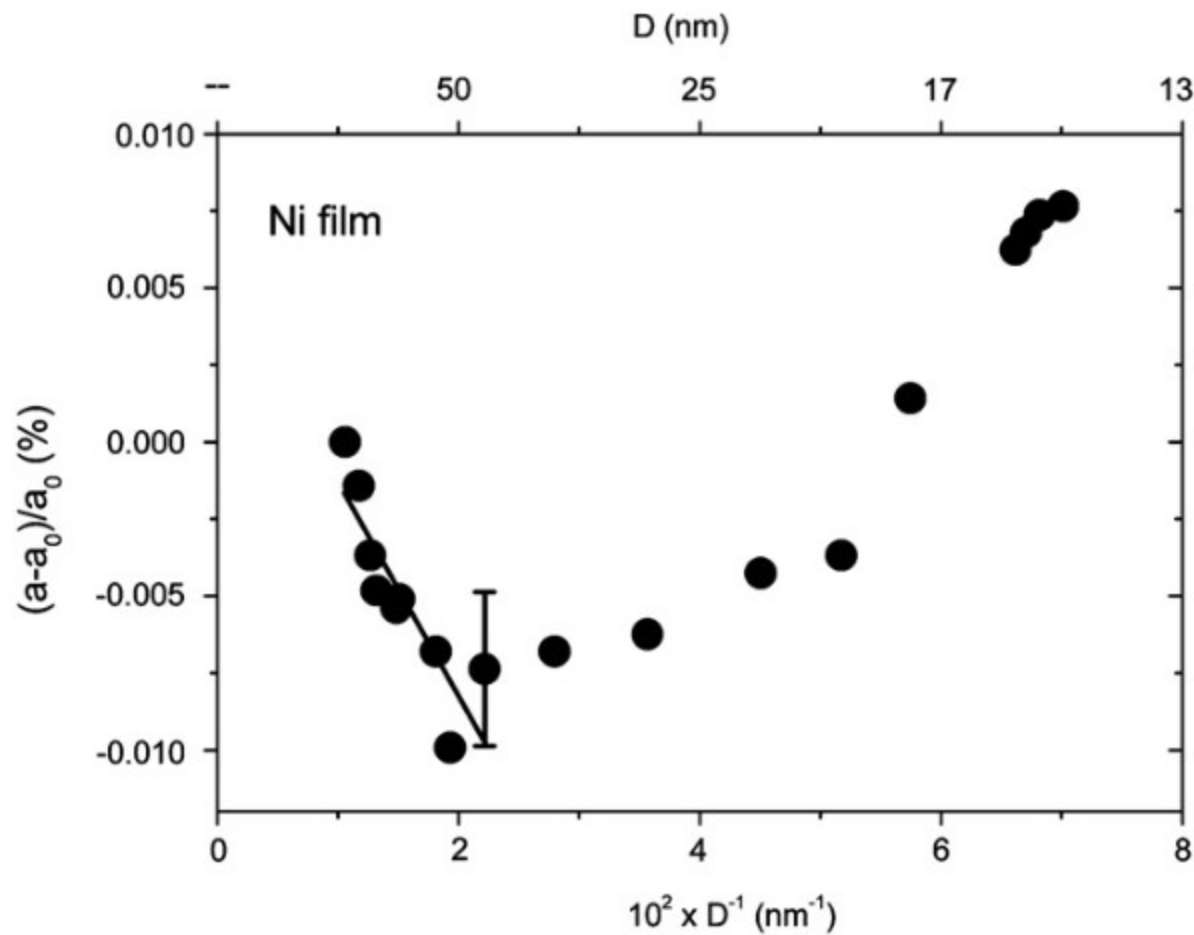
Lattice parameter of nanocrystalline Ni

J. Sheng et al, Physica E 43 1155 (2011)



Lattice parameter of nanocrystalline Ni

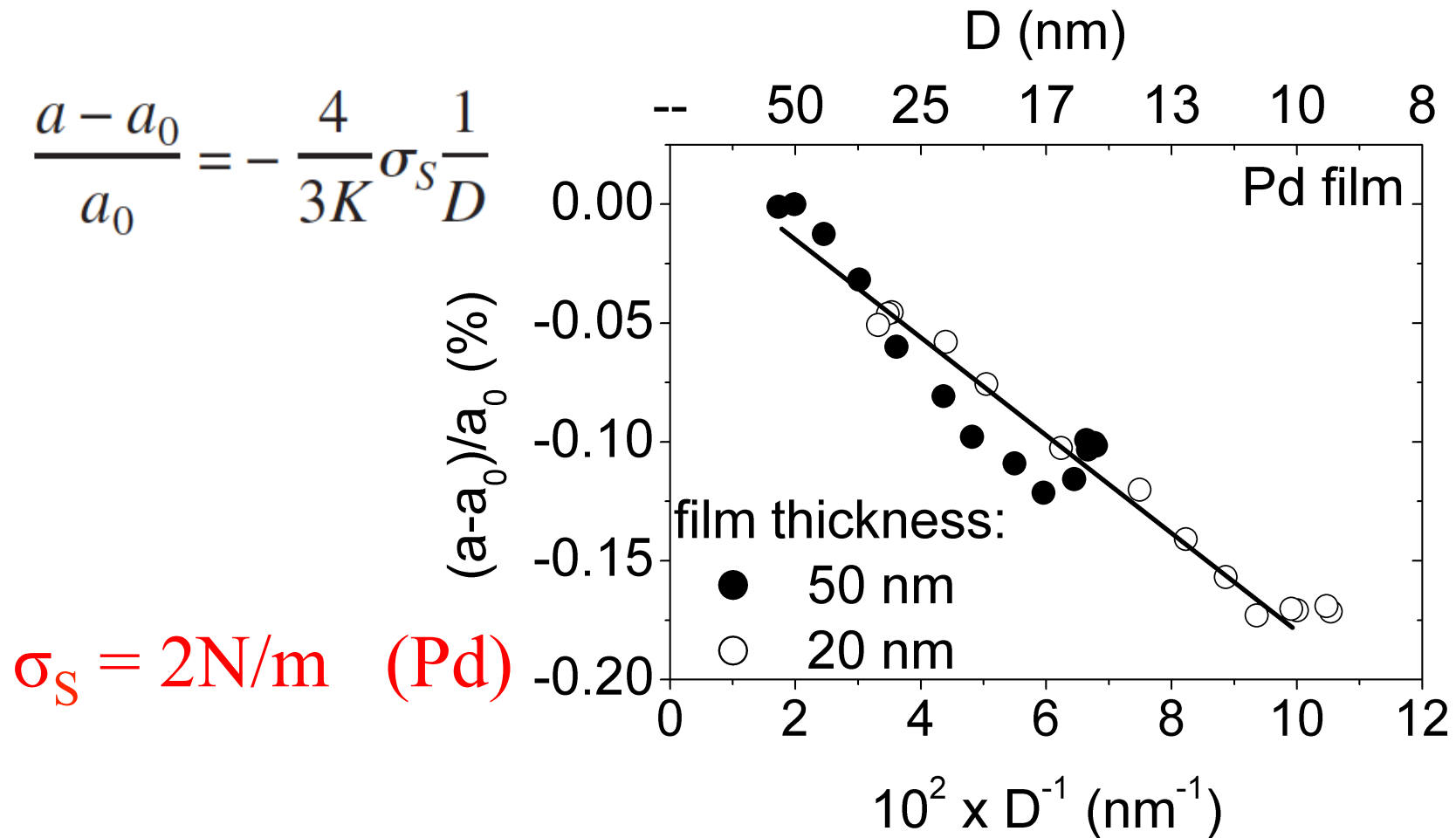
J. Sheng et al, Physica E 43 1155 (2011)



Contraction of Small Particles

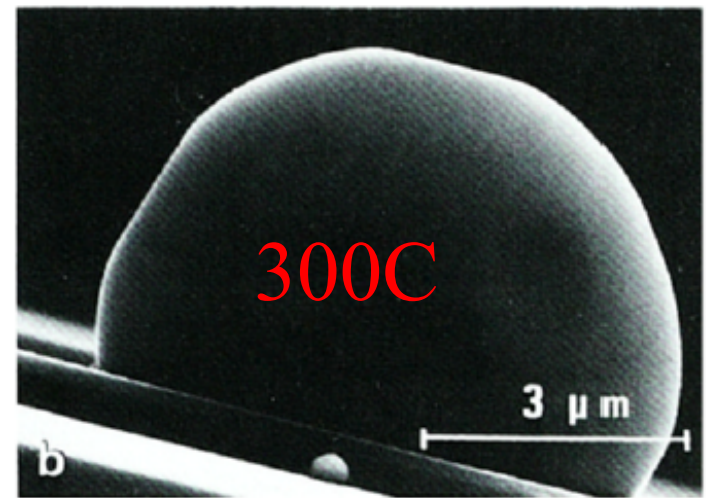
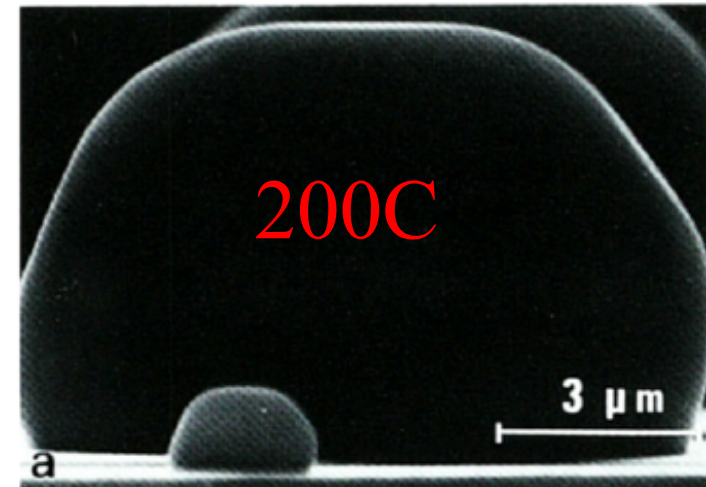
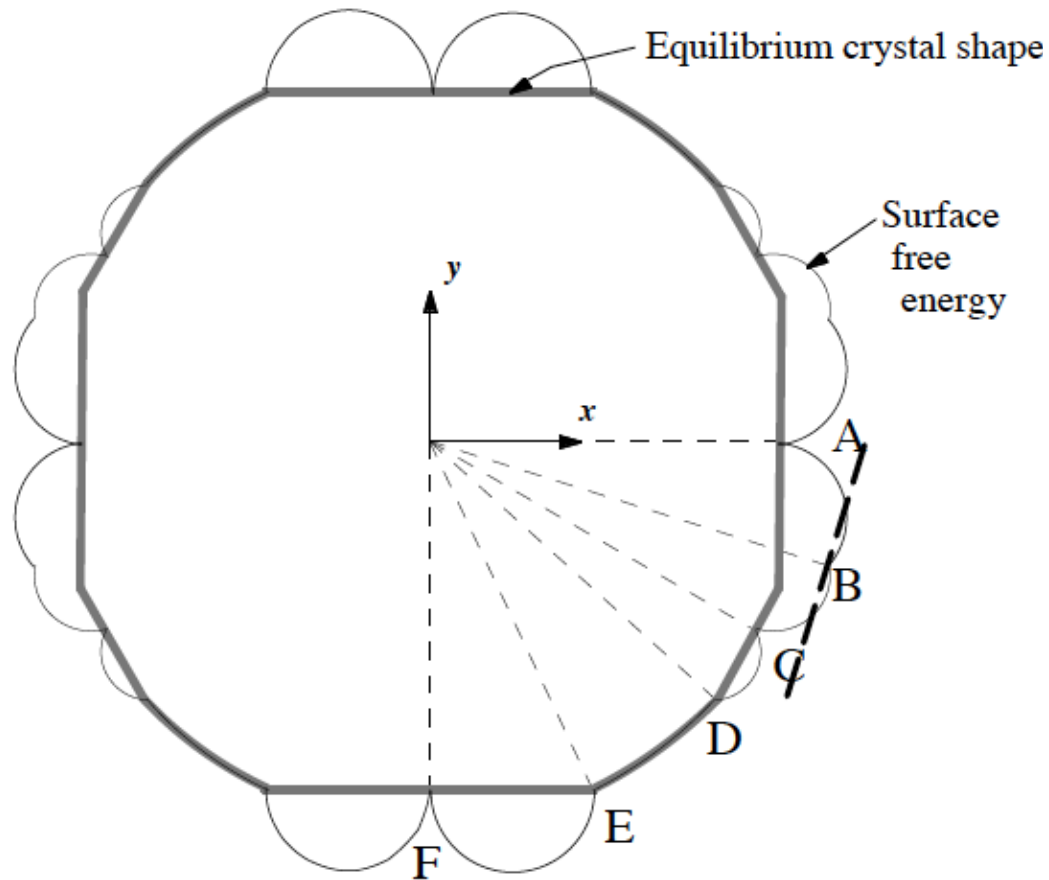
Gibbs Thomson pressure + Bulk modulus

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



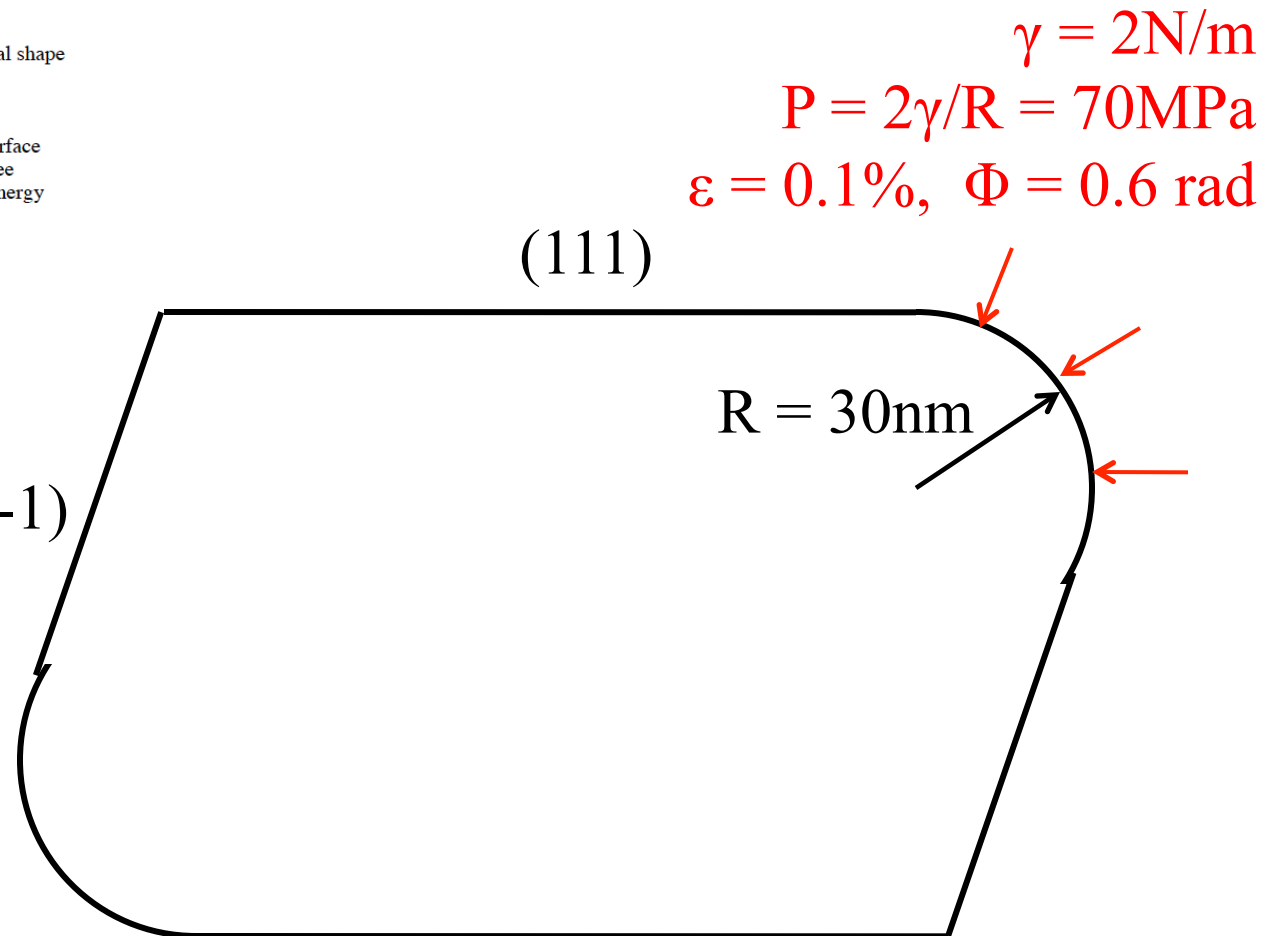
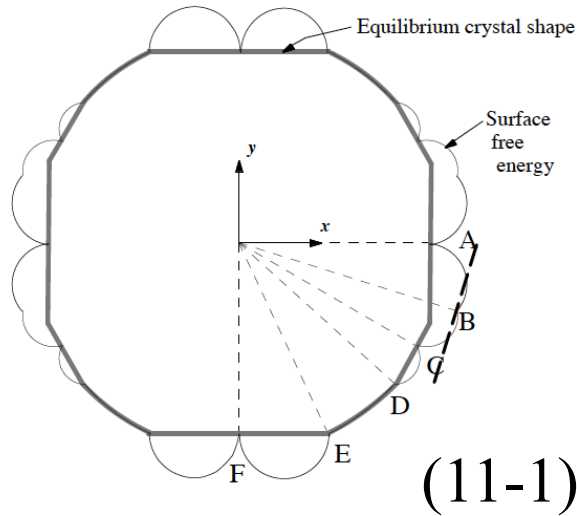
Equilibrium crystal shape

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

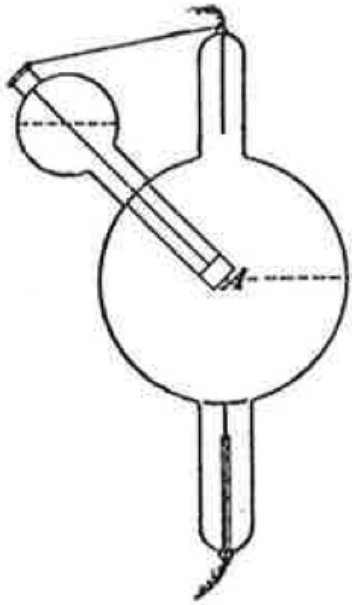


Equilibrium crystal shape

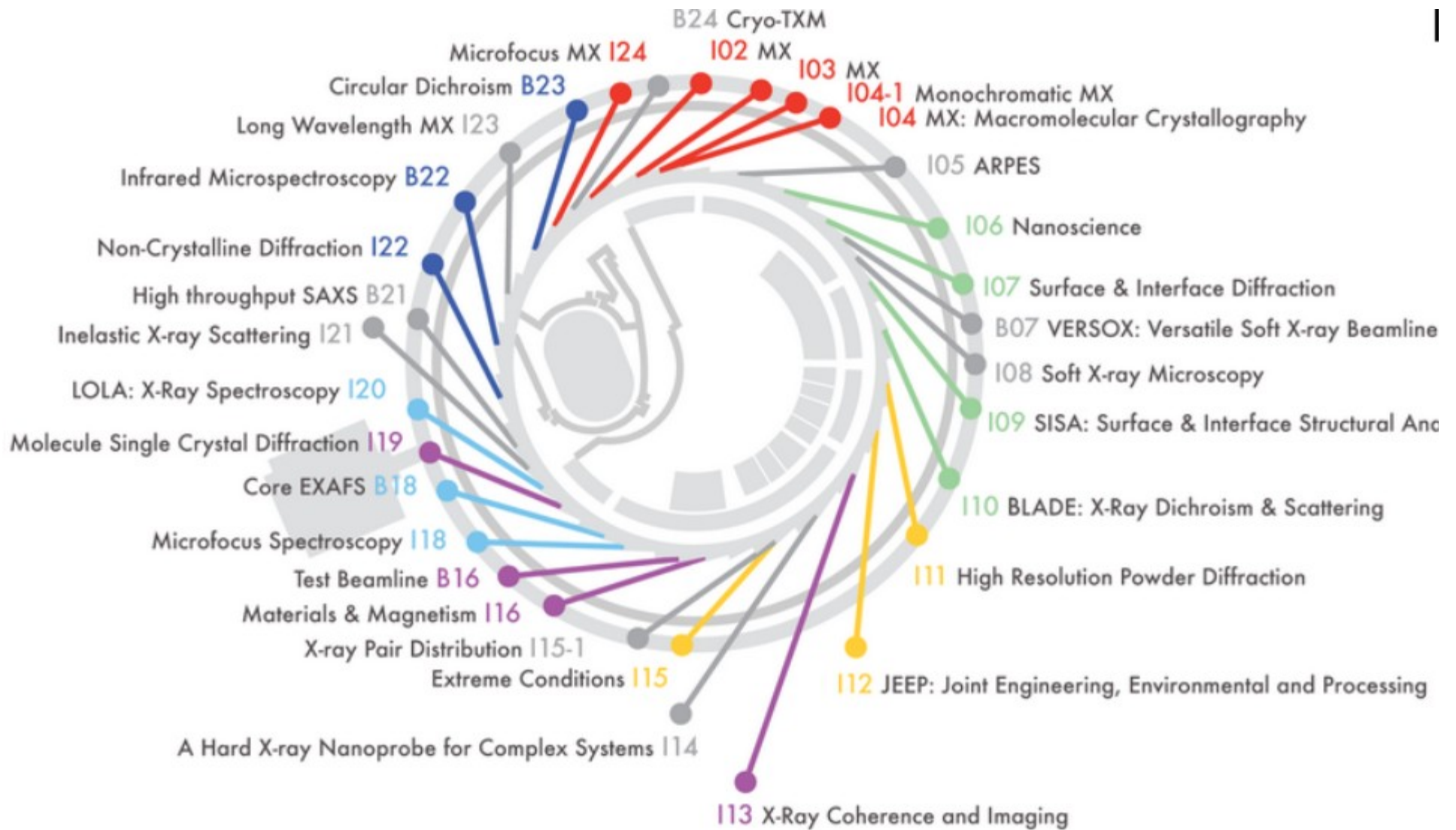
Wulff construction + Gibbs Thomson (Young-Laplace) pressure



Diamond Light Source (RAL Harwell)



Diamond Beamline Plan



X-ray Diffraction at Diamond

Phase I (January 2007)

I-02 to 04 Macromolecular crystallography beamlines

I-15 Extreme conditions beamline

I-16 Materials and magnetism beamline

Phase II (2010)

I-07 Surface and interface diffraction beamline

I-10 Beamline for Advanced Dichroism Experiments (BLADE)

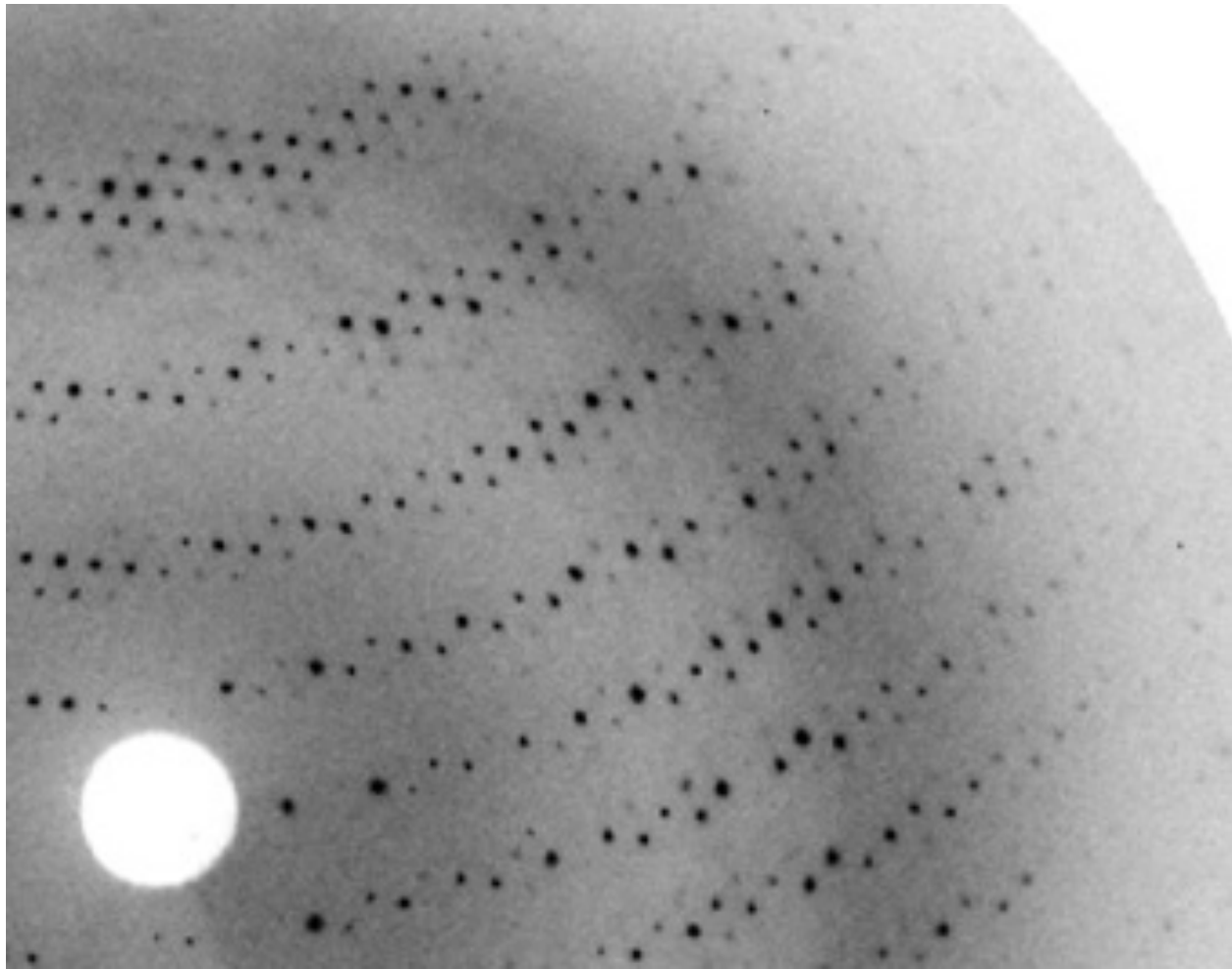
I-11 High resolution powder diffraction beamline

I-13 X-ray imaging and coherence

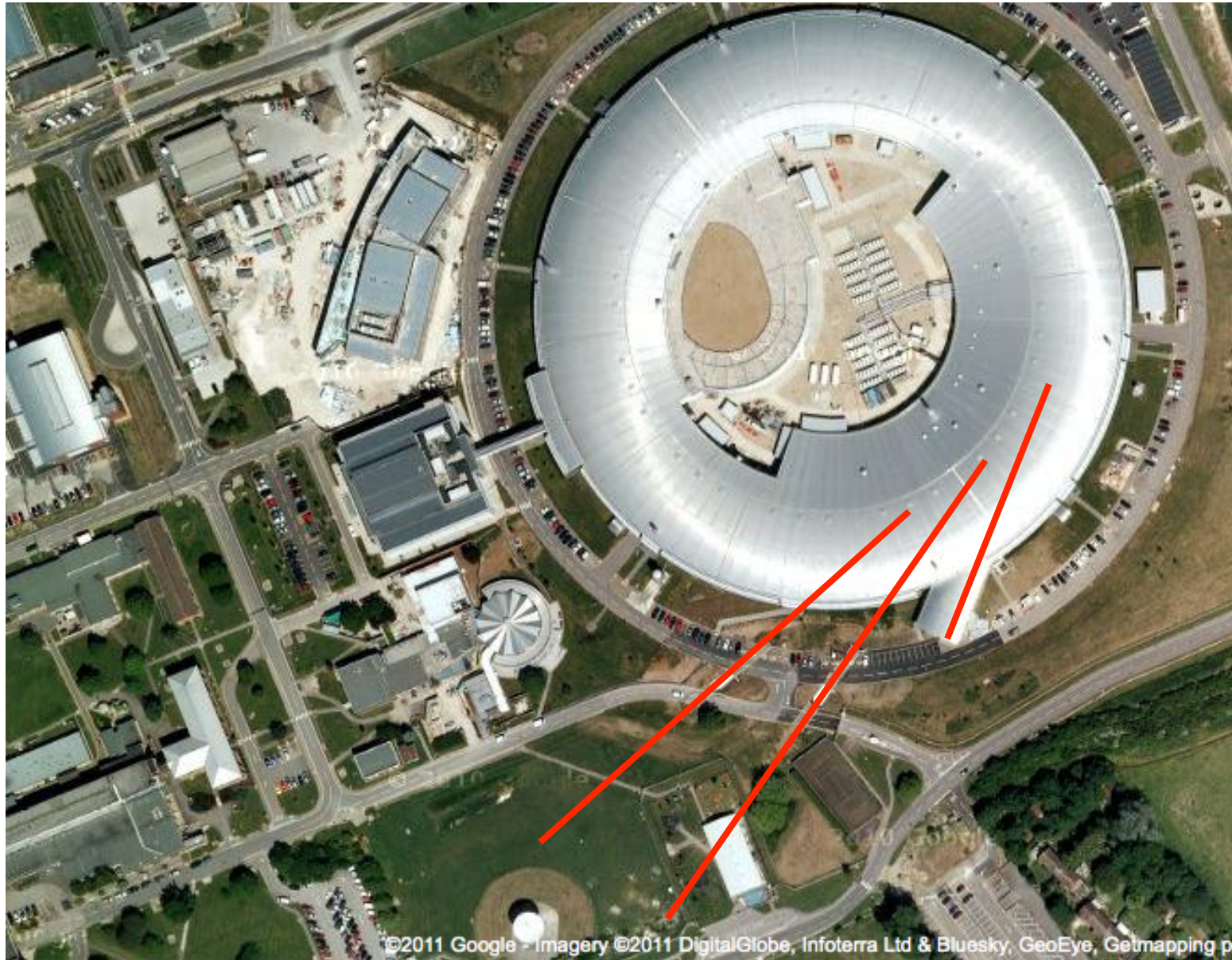
Phase III (2015)

I-15-1 X-ray Pair Distribution Function

Macromolecular X-ray Crystallography (MX)

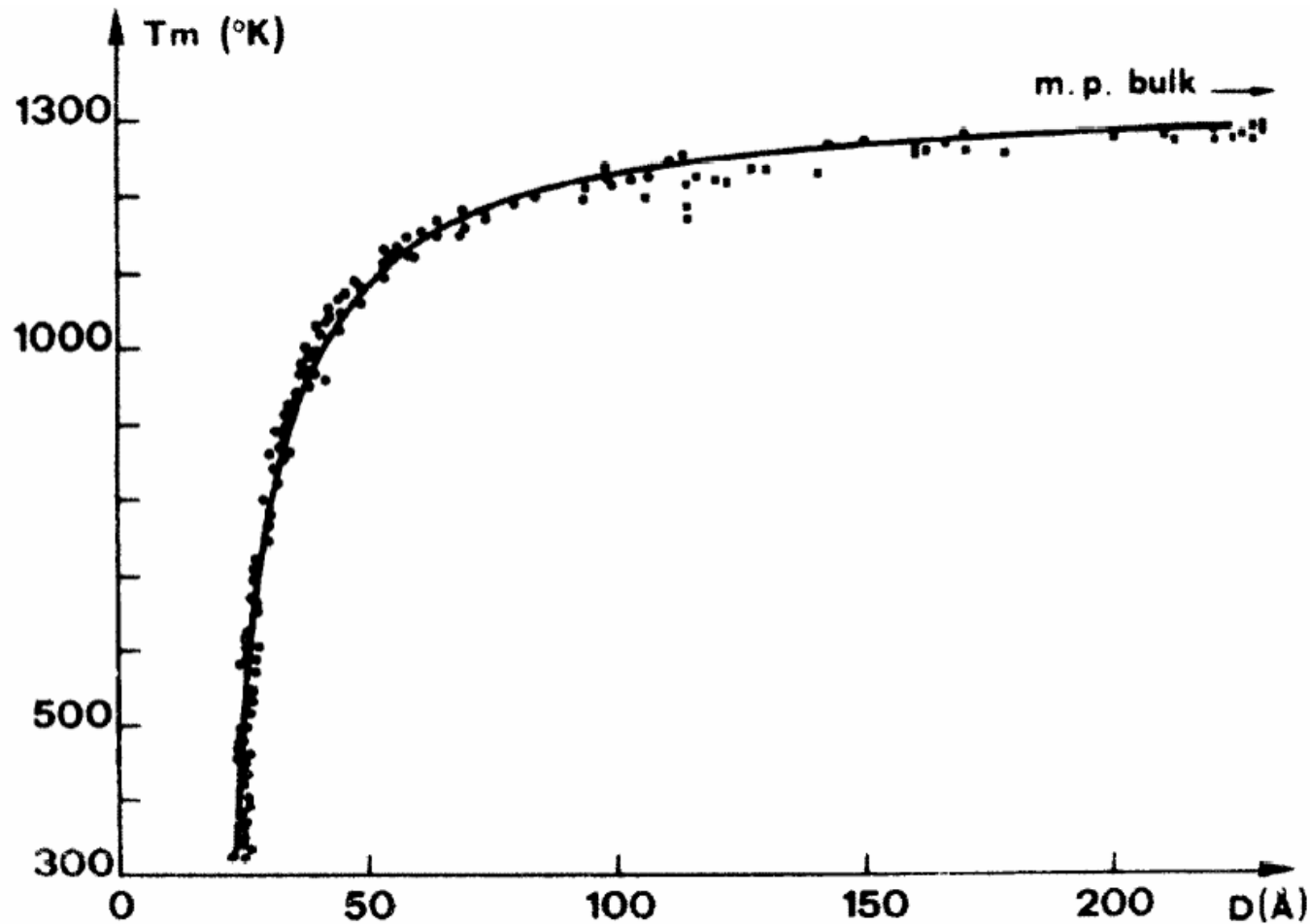


Diamond Light Source, 2009

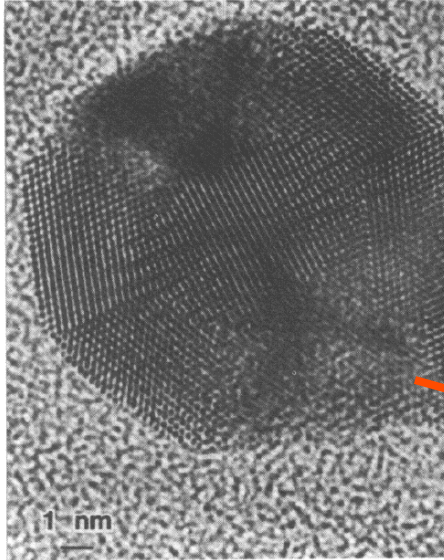


Size-dependent Melting of Au Particles

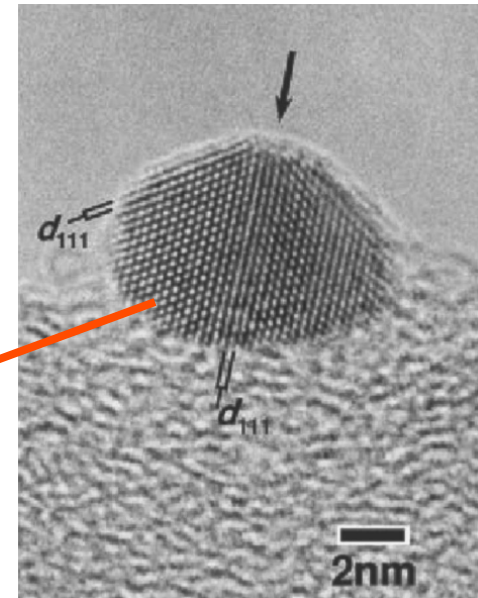
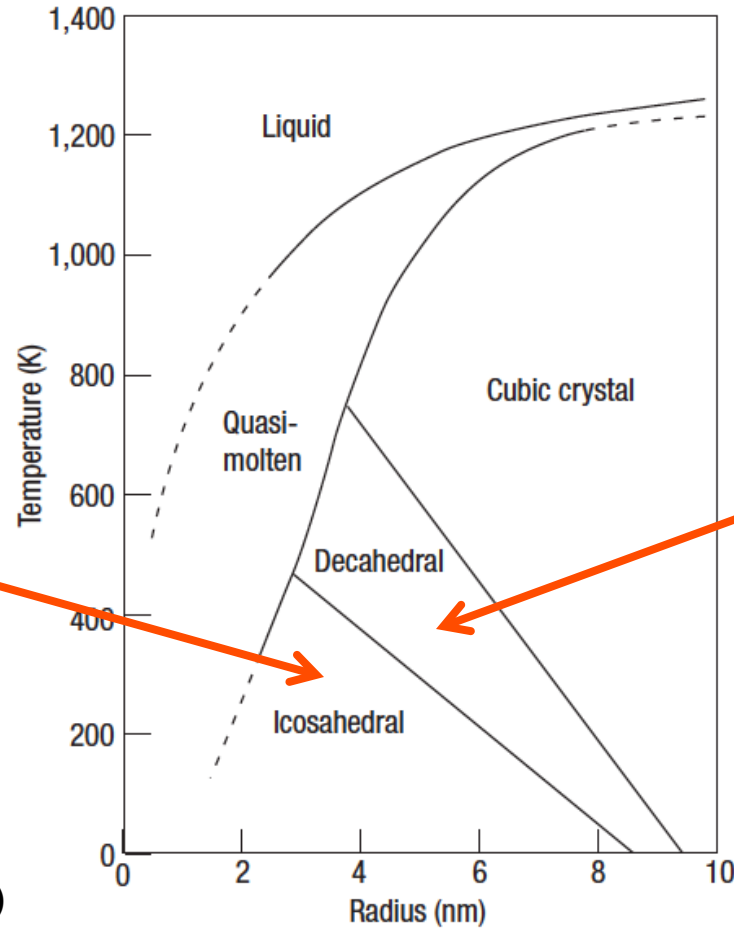
P. Buffat and J-P. Borel, Phys. Rev. A 2287-97 (1975)



Structure of Gold vs Size

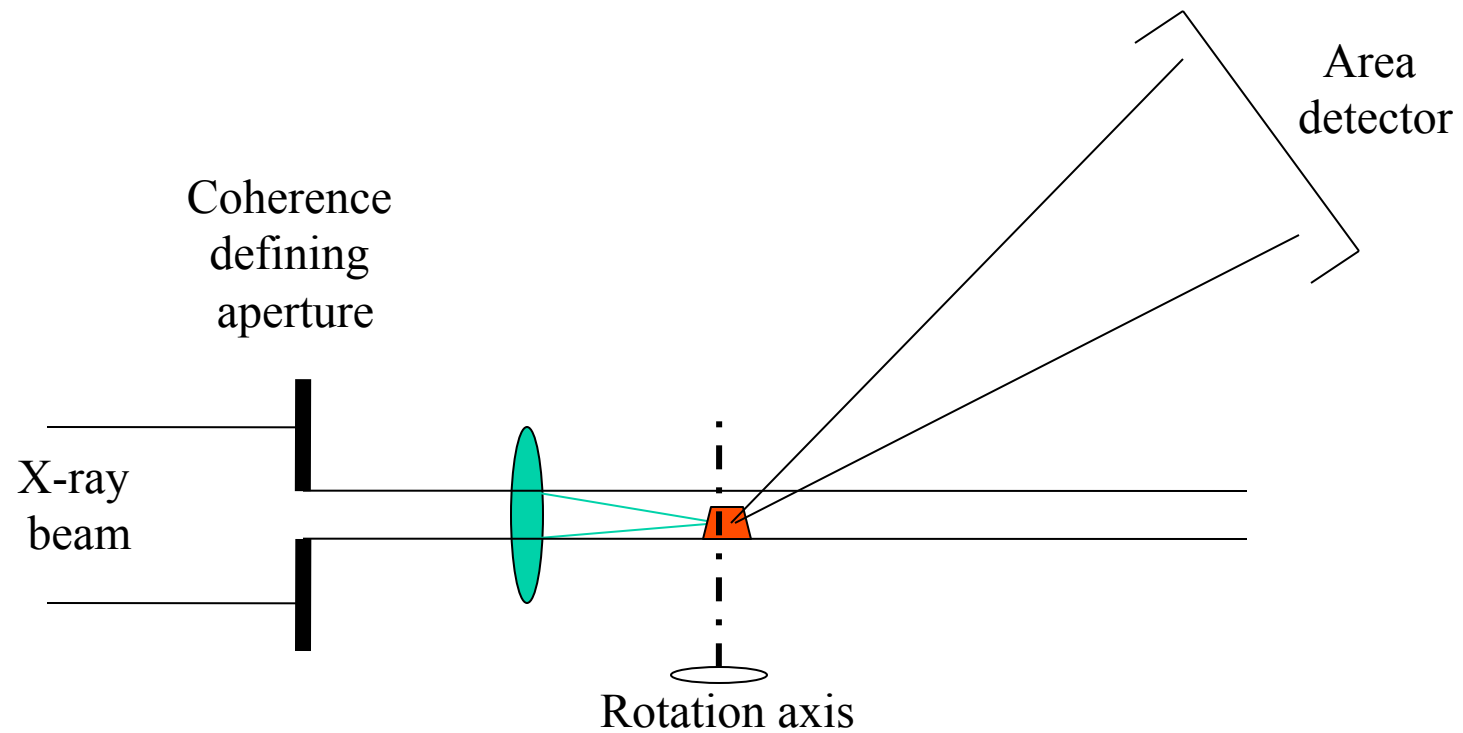


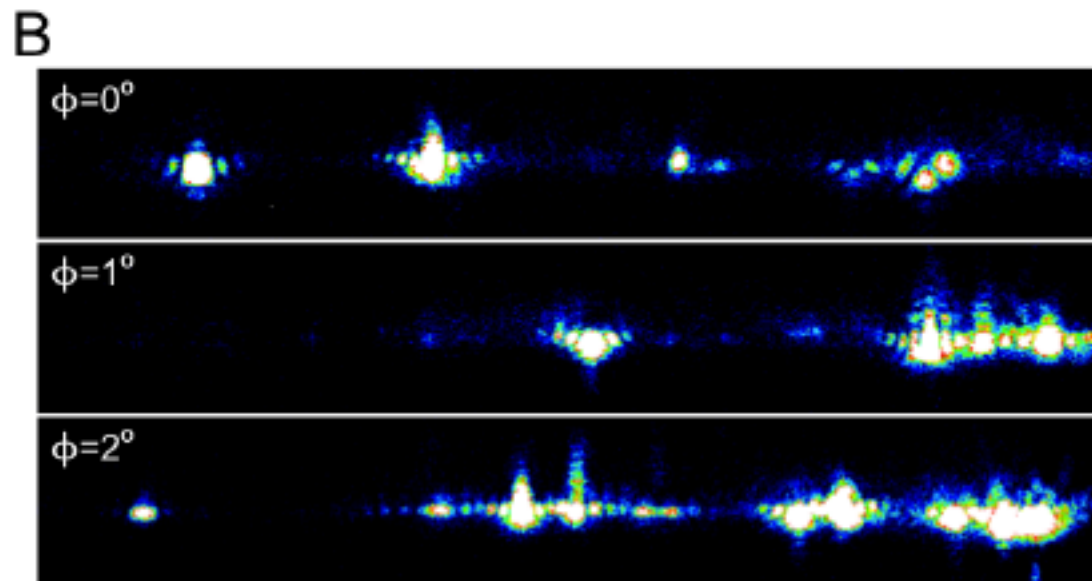
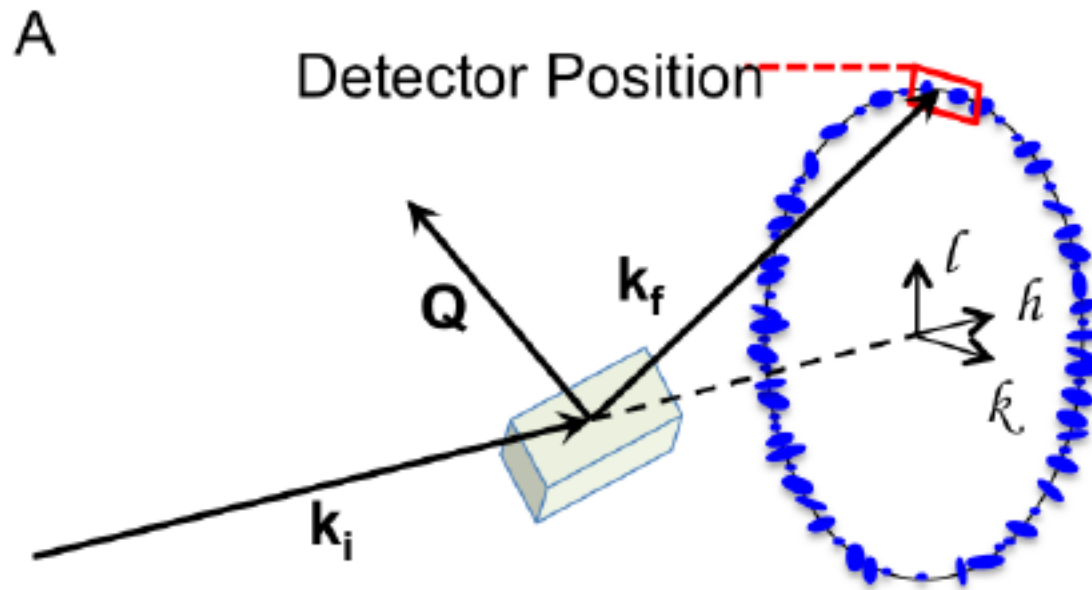
L. D. Marks, RPP (1994)



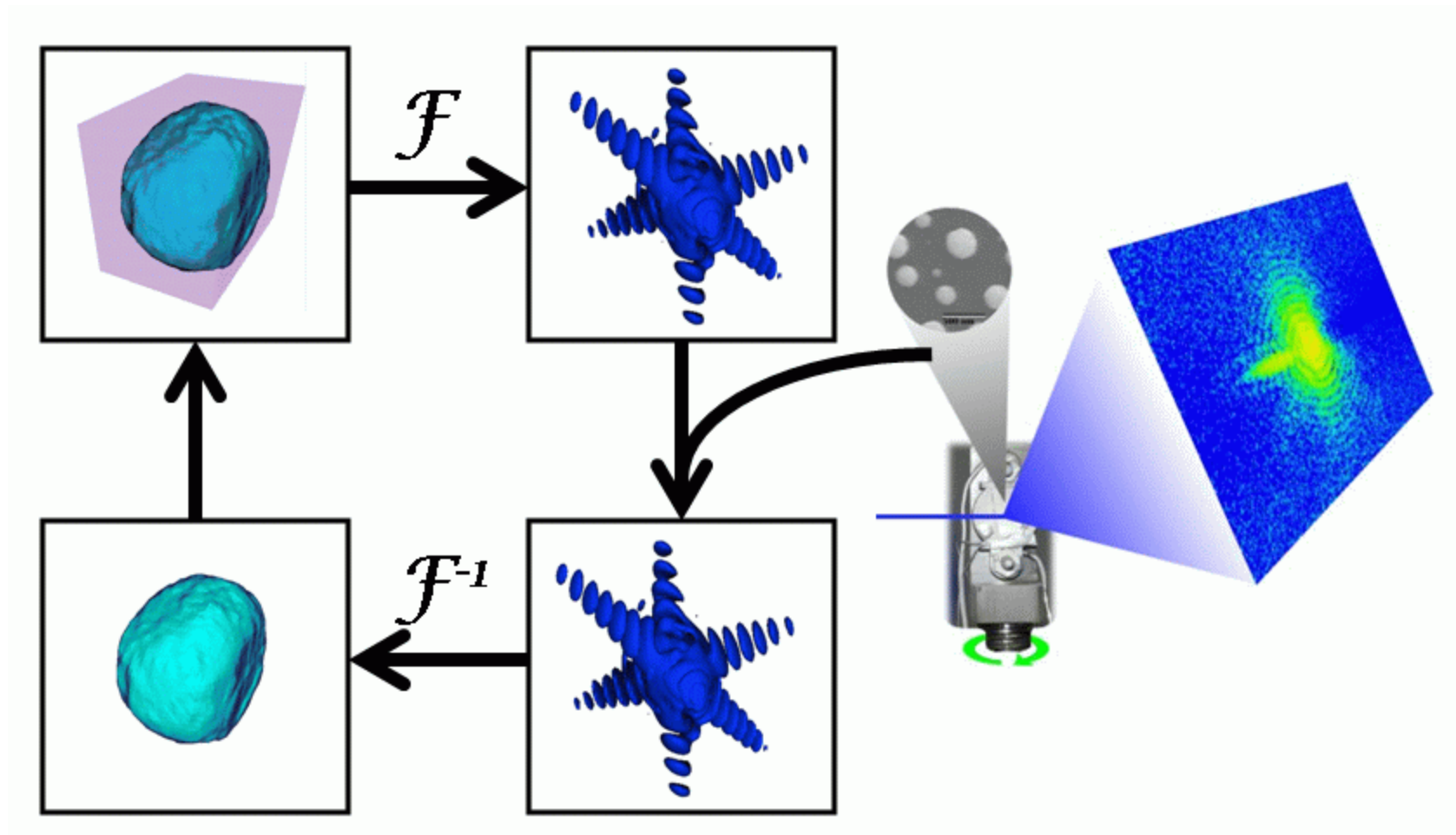
Koga and Sugawara (2003)

Lensless X-ray Microscope, 2003





Generic “Error Reduction” method



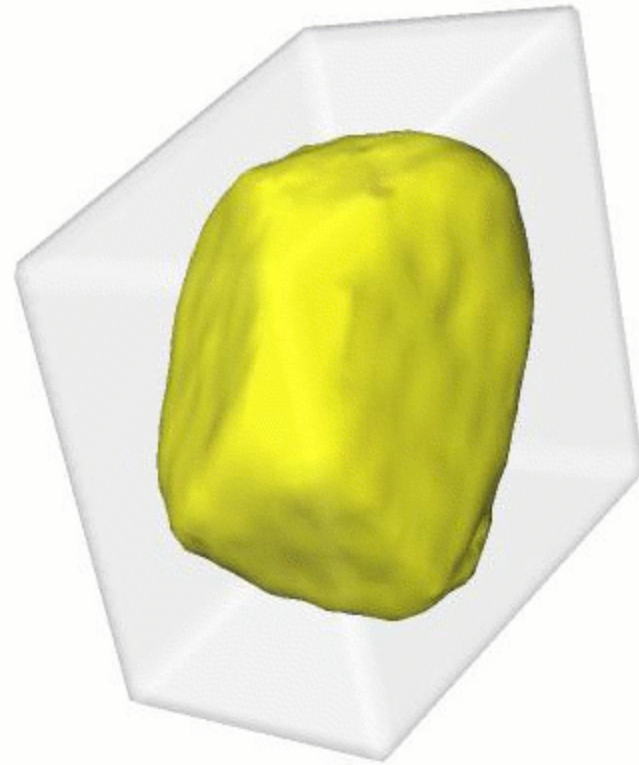
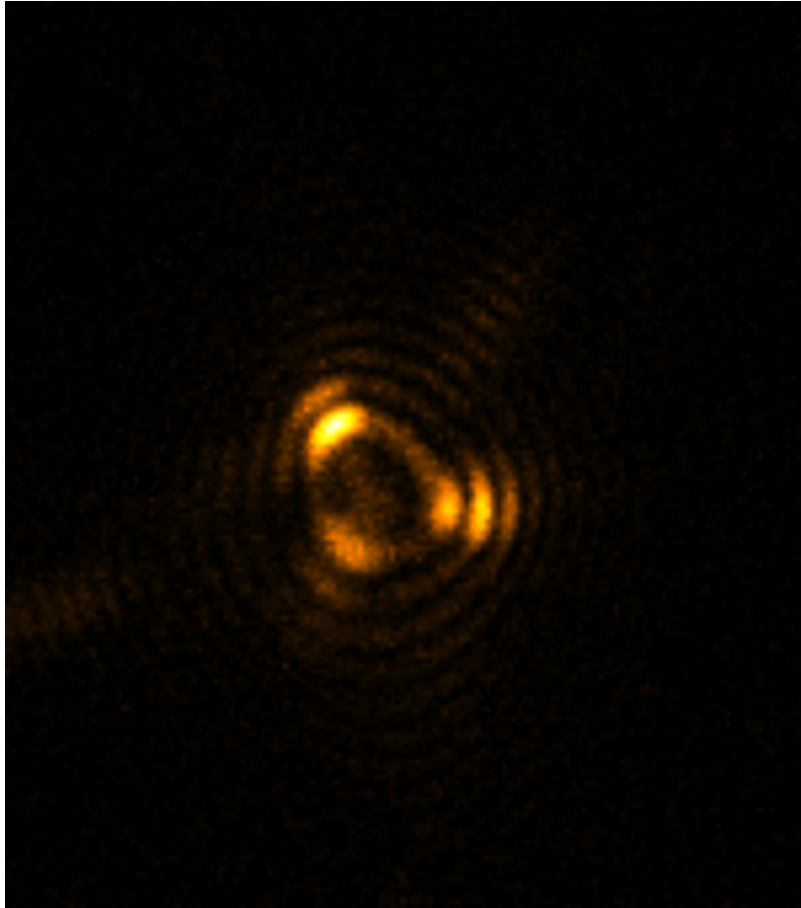
J. R. Fienup Appl. Opt. 21 2758 (1982)

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

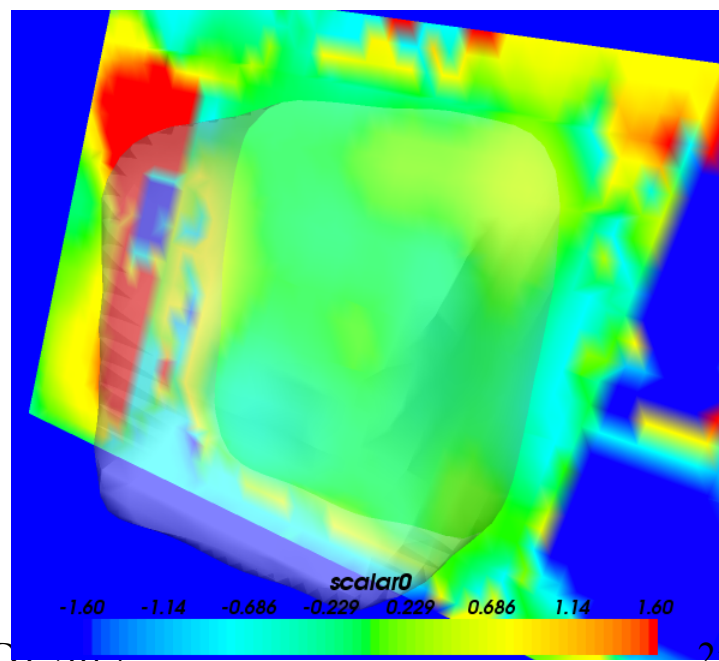
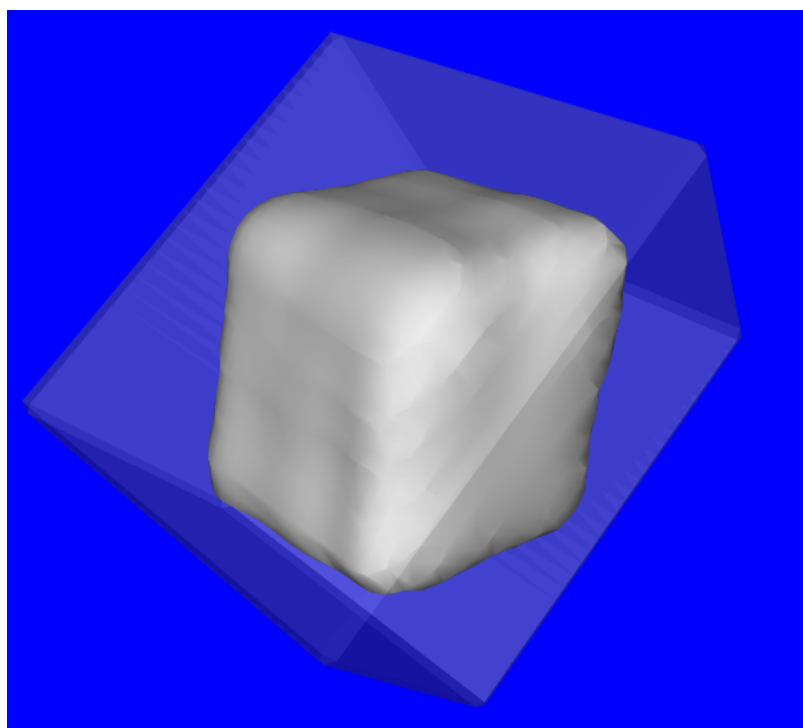
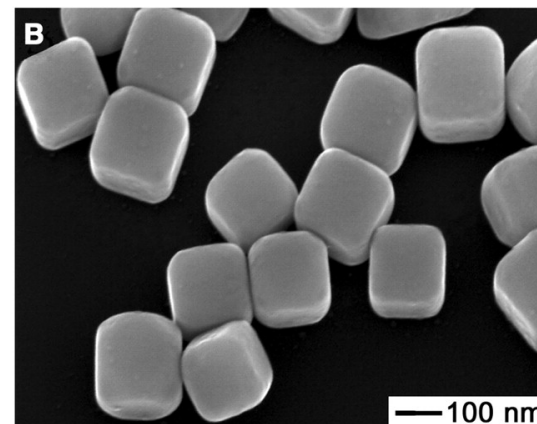
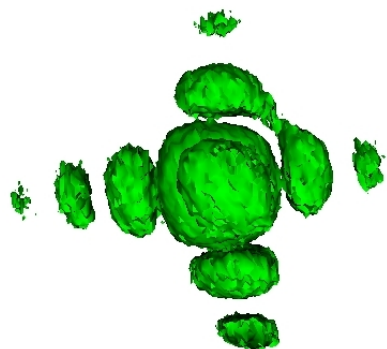
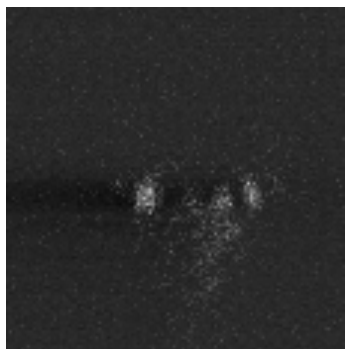
I. K. Robinson, CDT 2015

Gold nanocrystal reconstruction

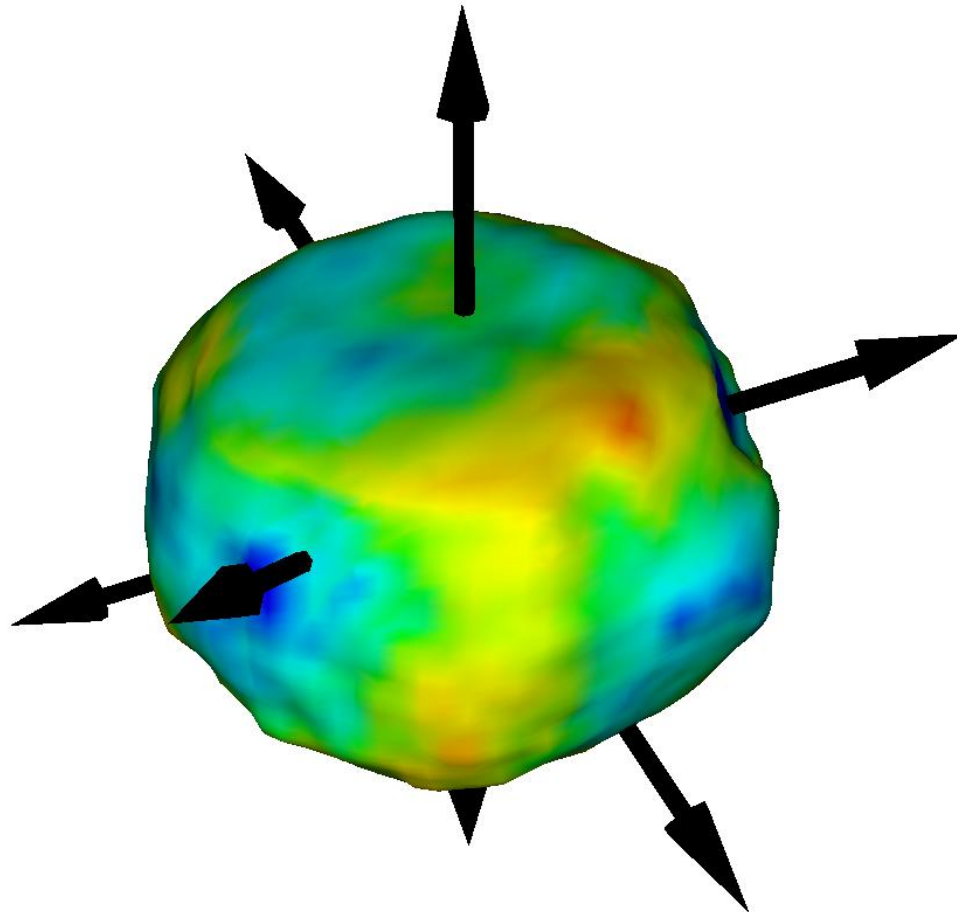
showing support used for 20 HIO followed by 10 ER



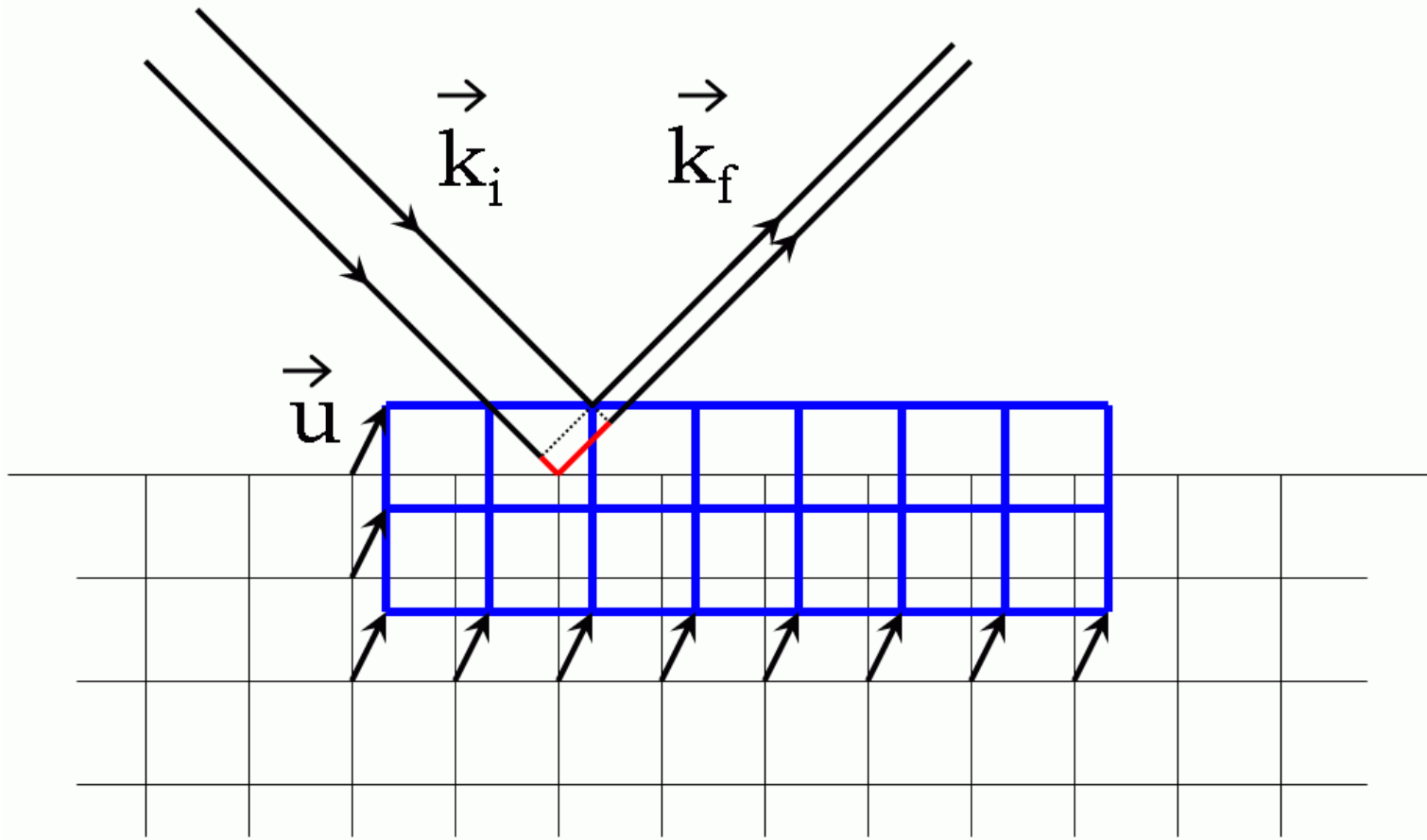
3D Ag Nano Cube



Phase isosurface of residual strain

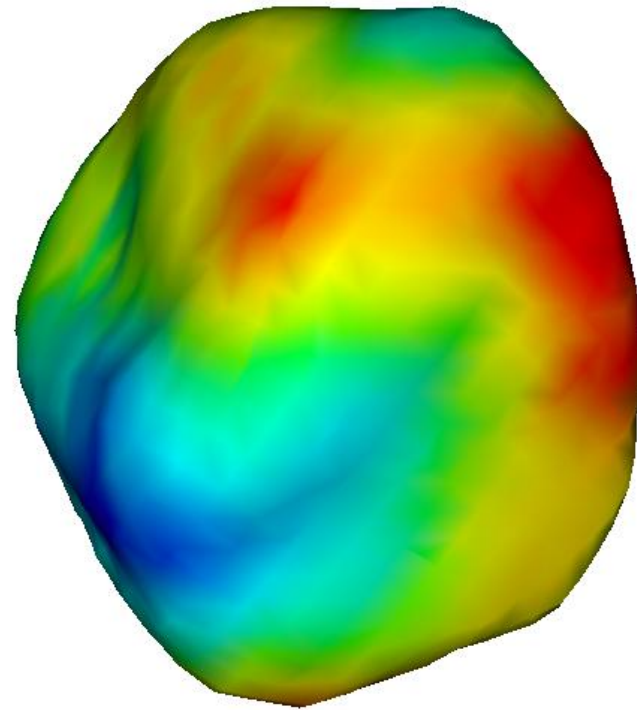
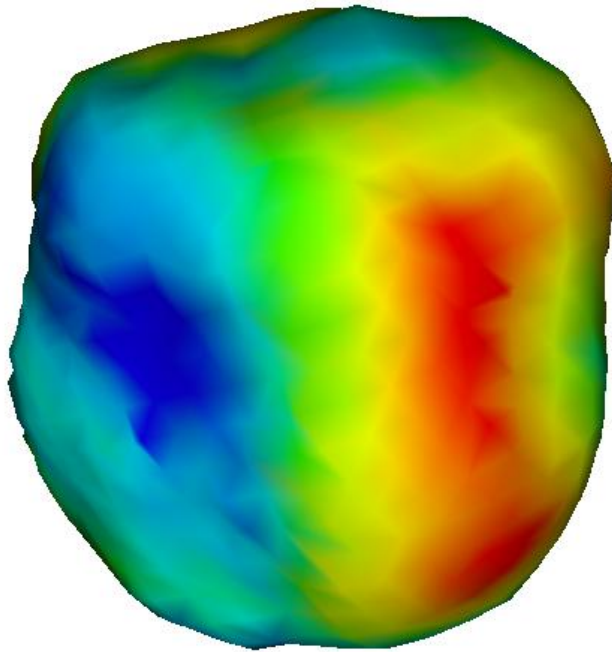


Sensitivity to strain

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


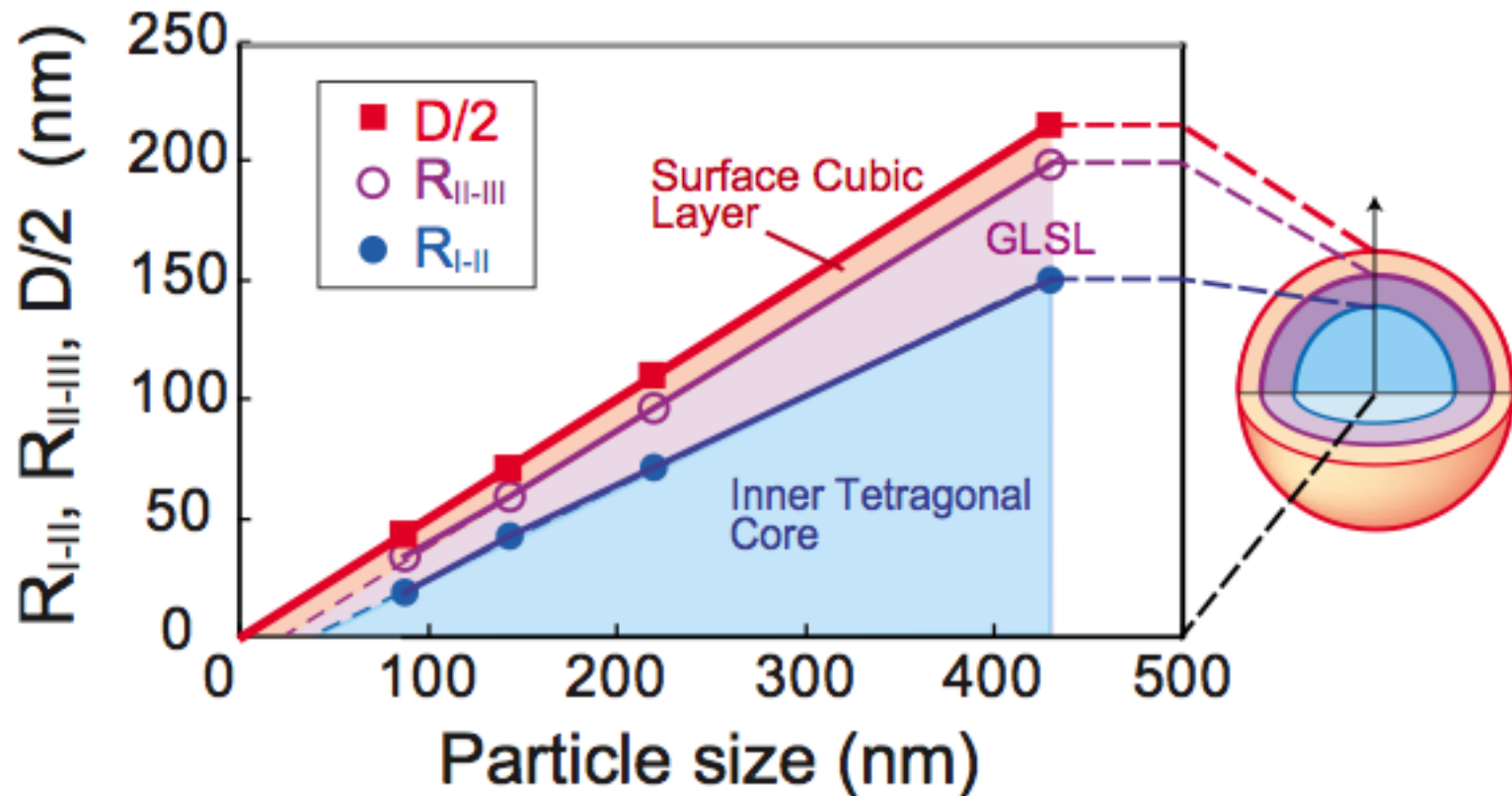
Phase isosurface of residual strain

200nm Barium Titanate (BTO) crystals



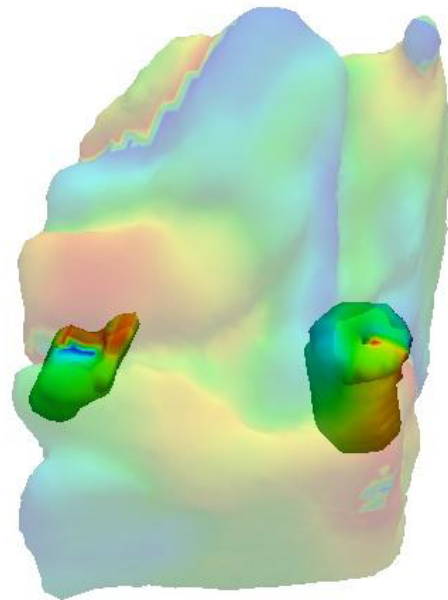
Core-shell structure of BTO

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



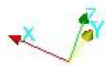
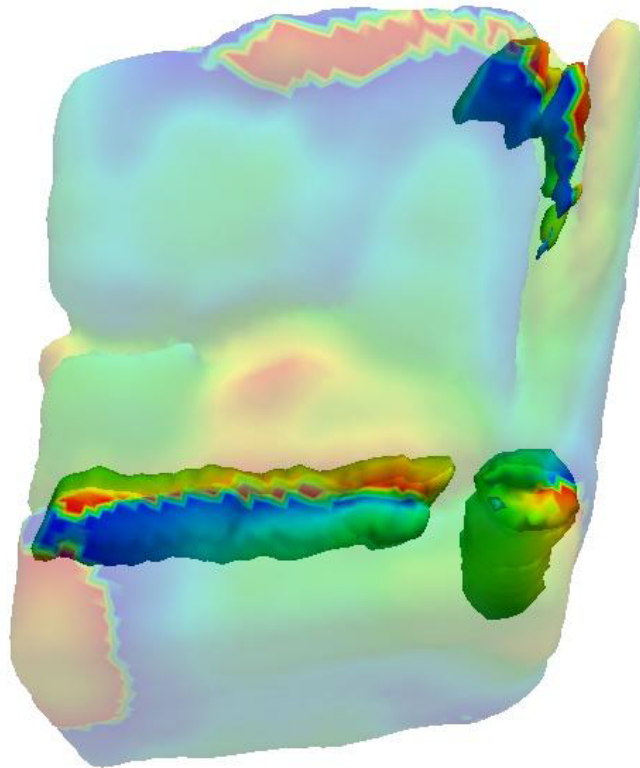
Calcite crystal growth

Jesse Clark and Johannes Ihli unpublished



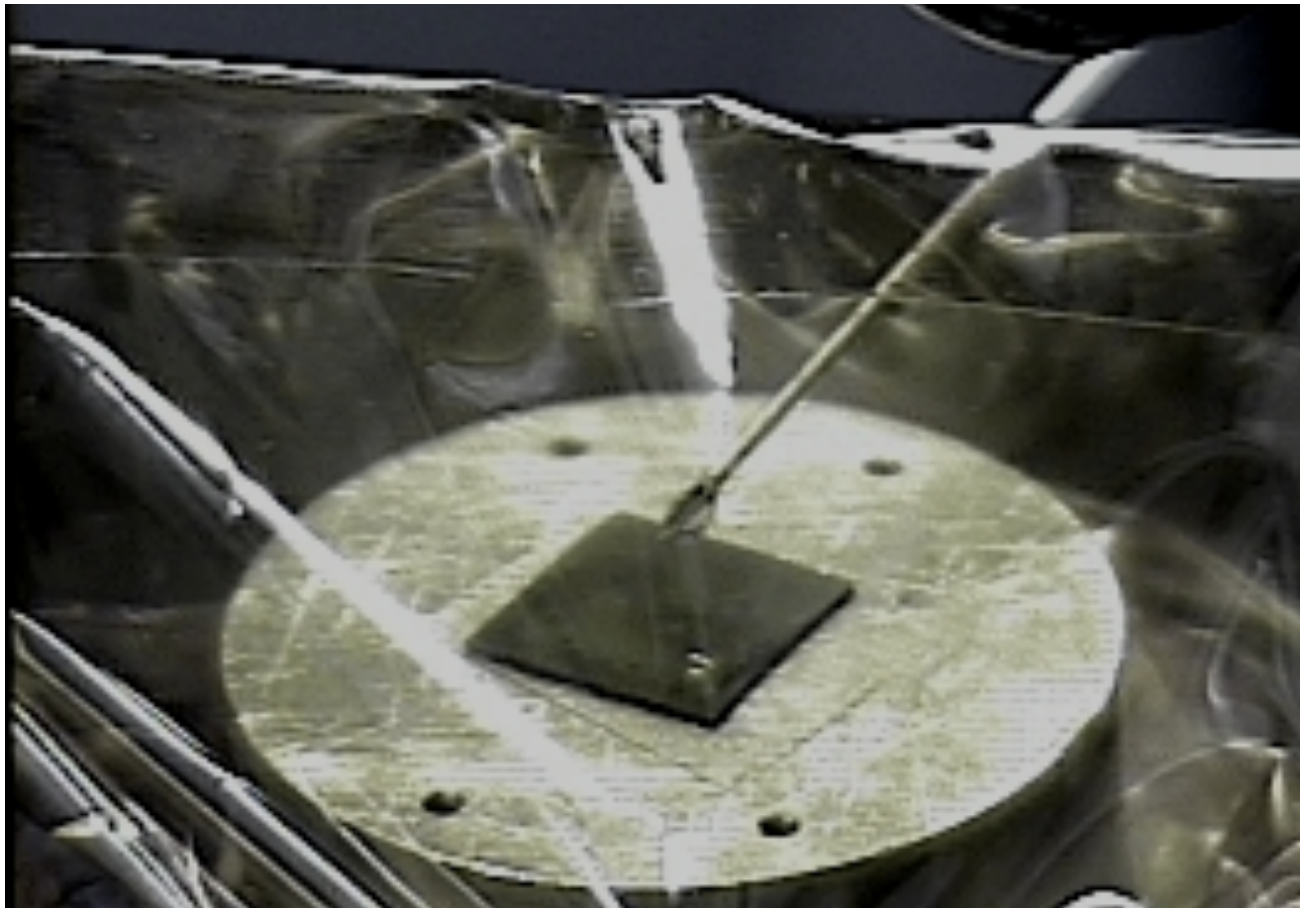
Calcite crystal growth

Jesse Clark and Johannes Ihli unpublished



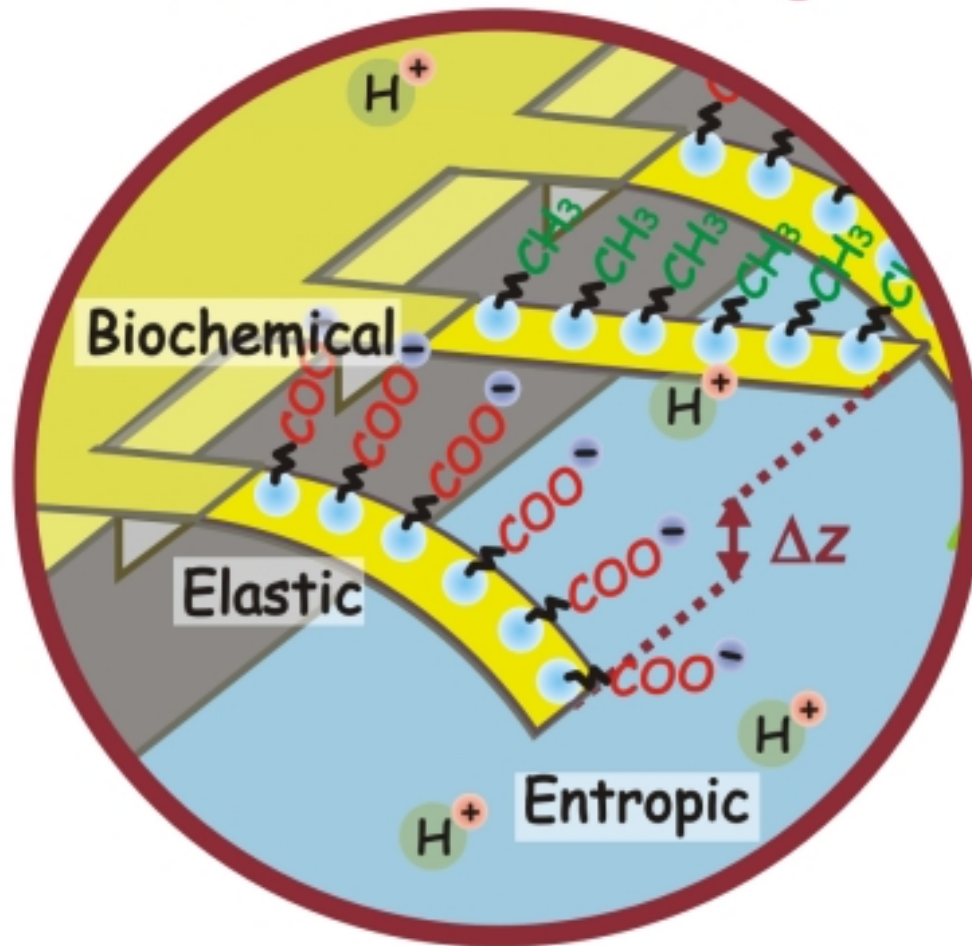
Dosing with C_3H_7SH in ethanol

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)



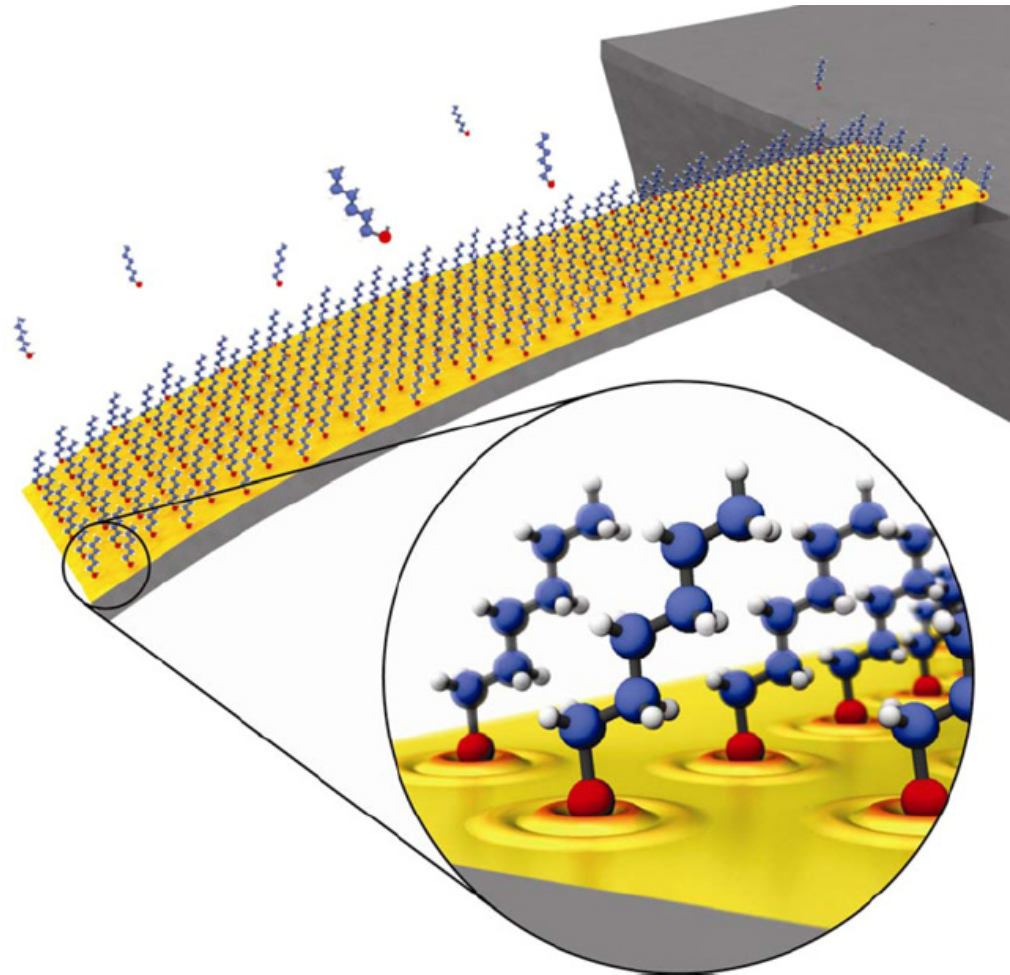
Nanocantilevers

Dr Rachel McKendrie



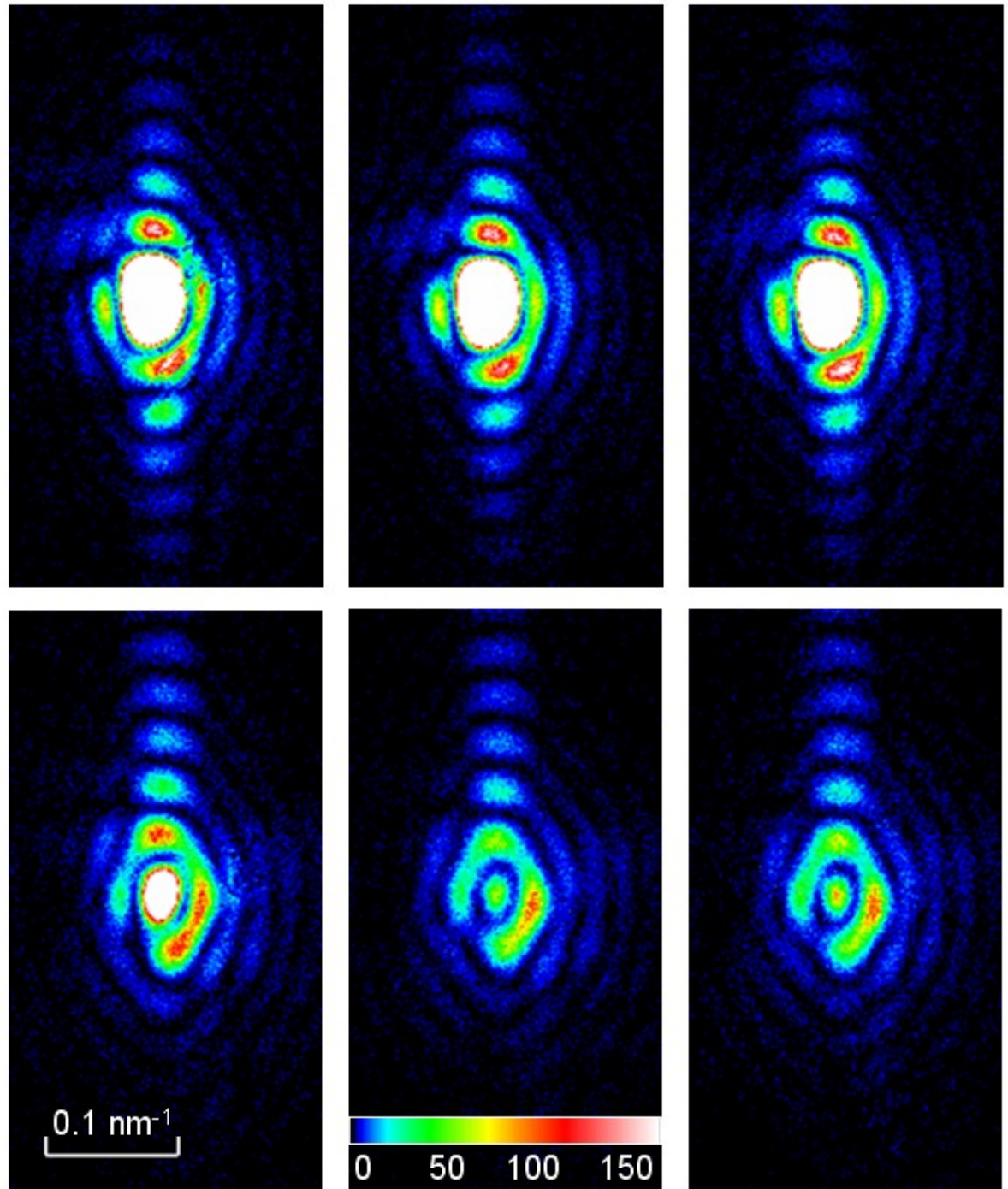
Formation of SAM on cantilever

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



Rocking curve of Au crystal

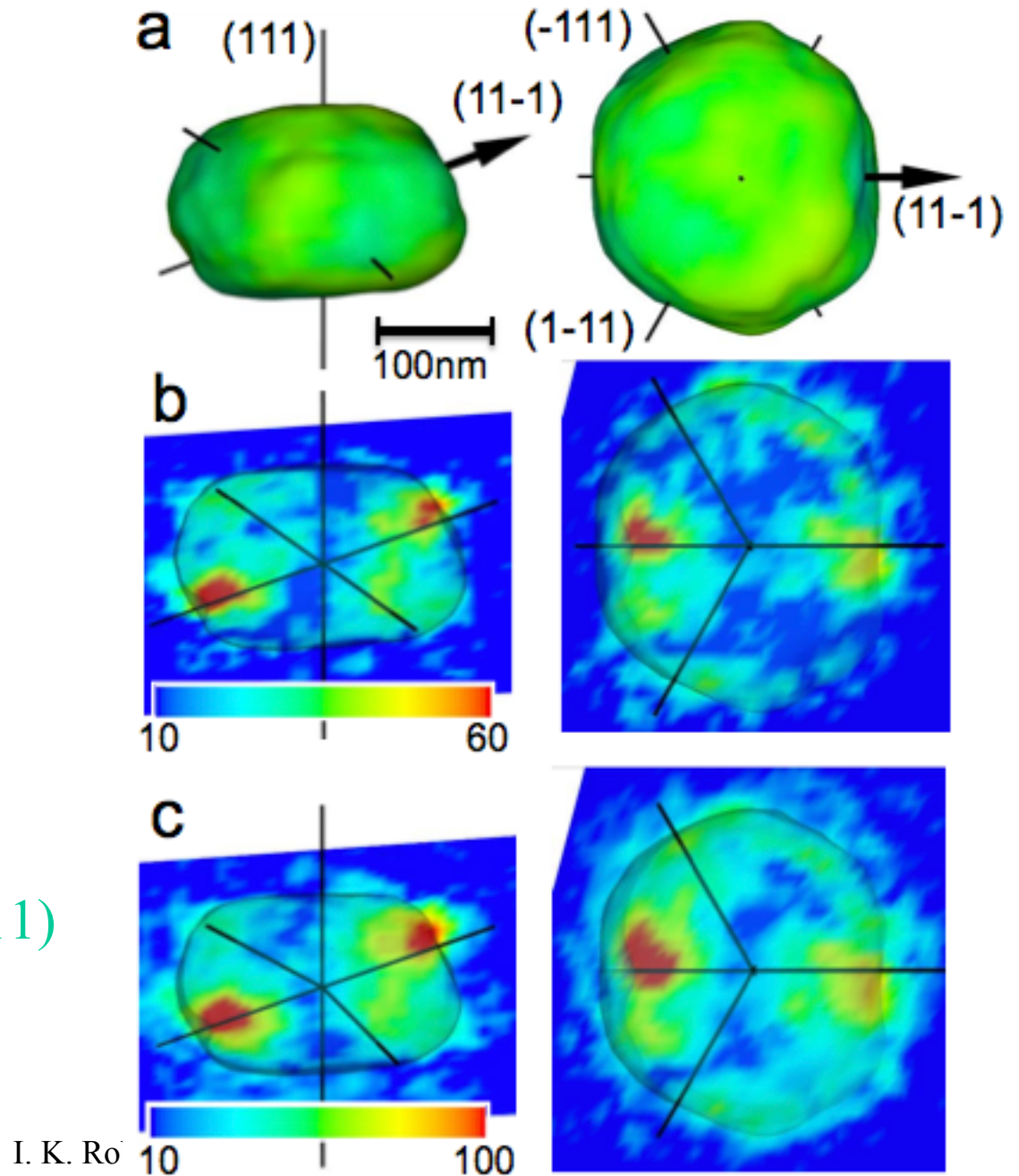
Before dosing /
dose 1 / dose 2



Difference Fourier:

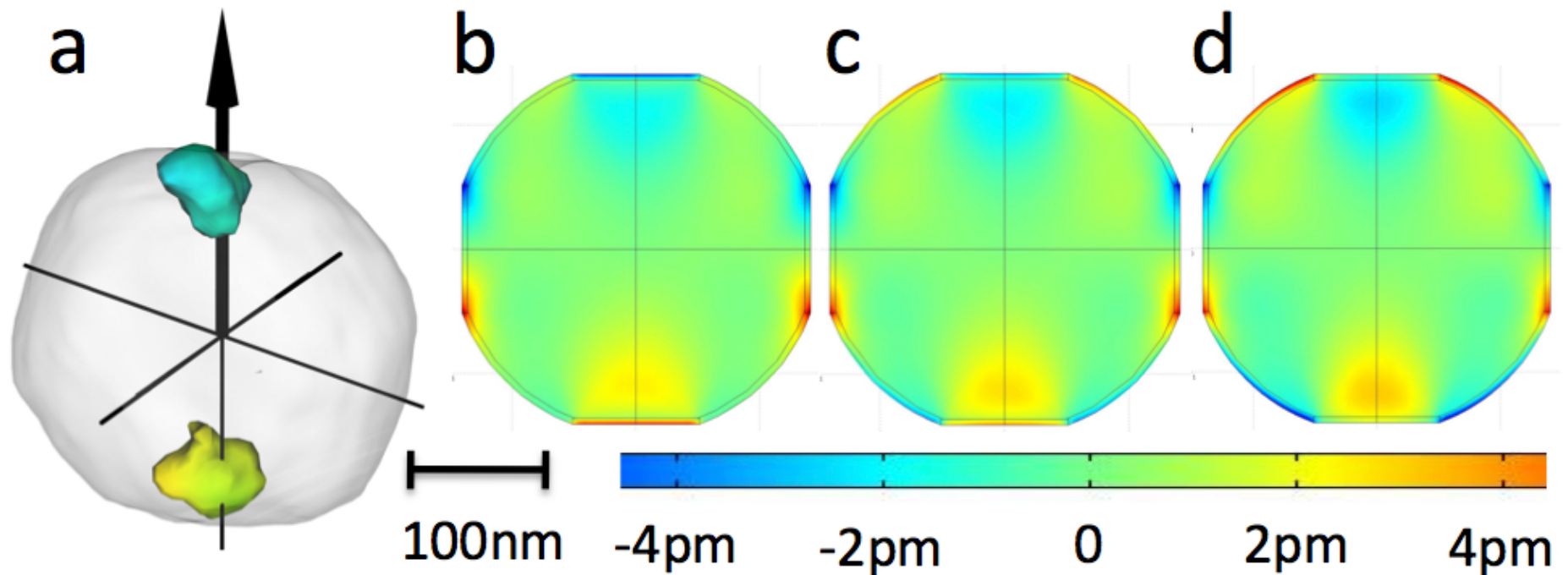
Magnitude of phase
change caused by
dosing

Moyu Watari et al,
Nature Materials (2011)



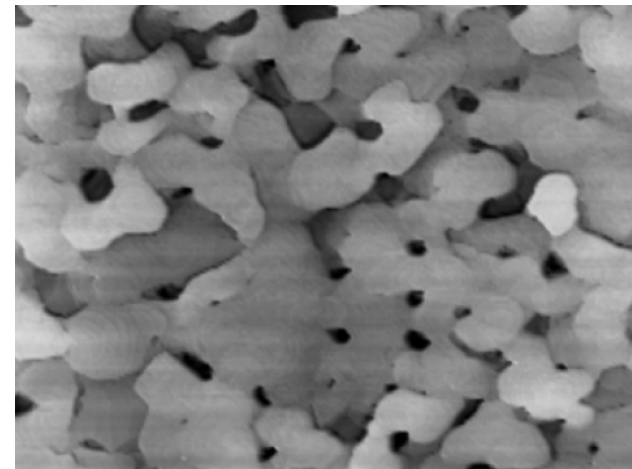
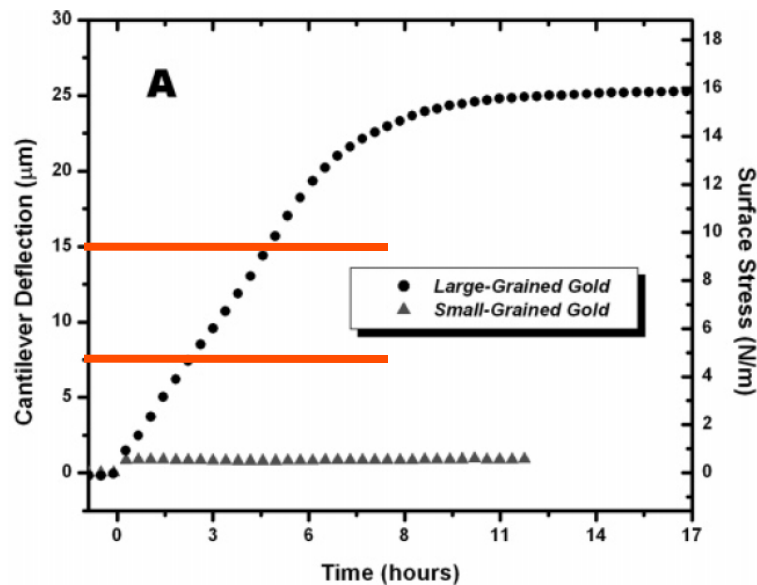
Finite-element Analysis

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



Cantilever strain experiment

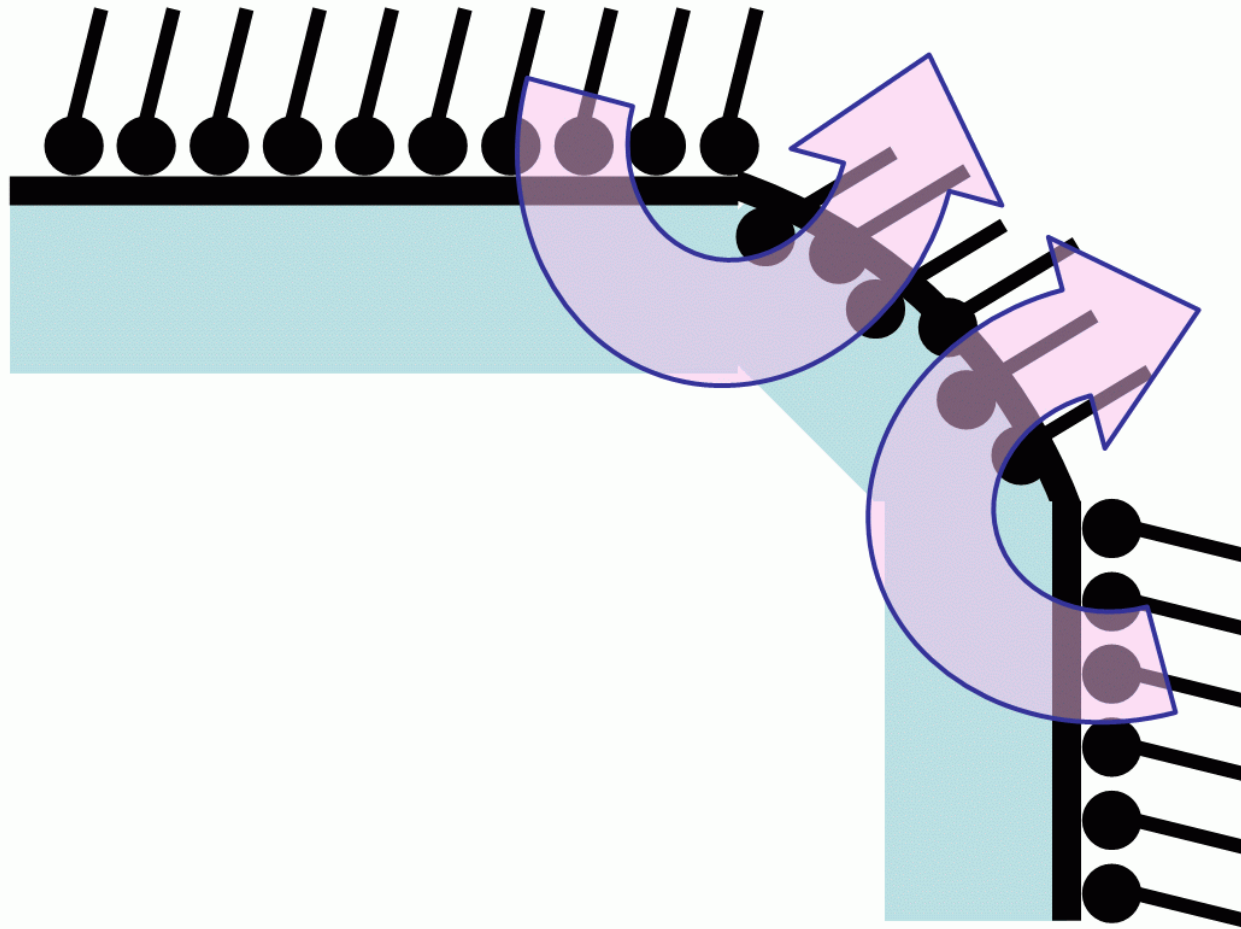
M. Godin et al Langmuir **20** 7090 (2004)



3 × 2 μm STM image

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

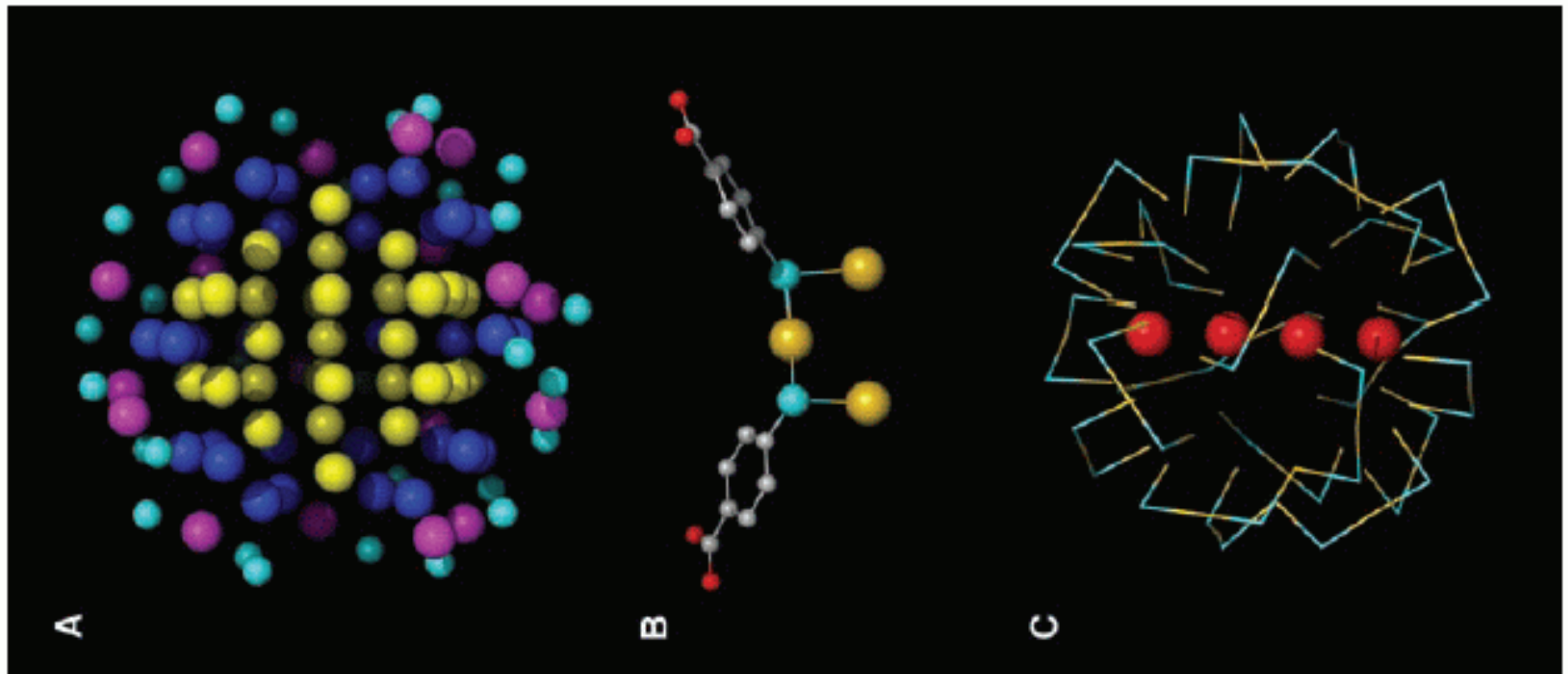
Differential adsorption, thiol/Au

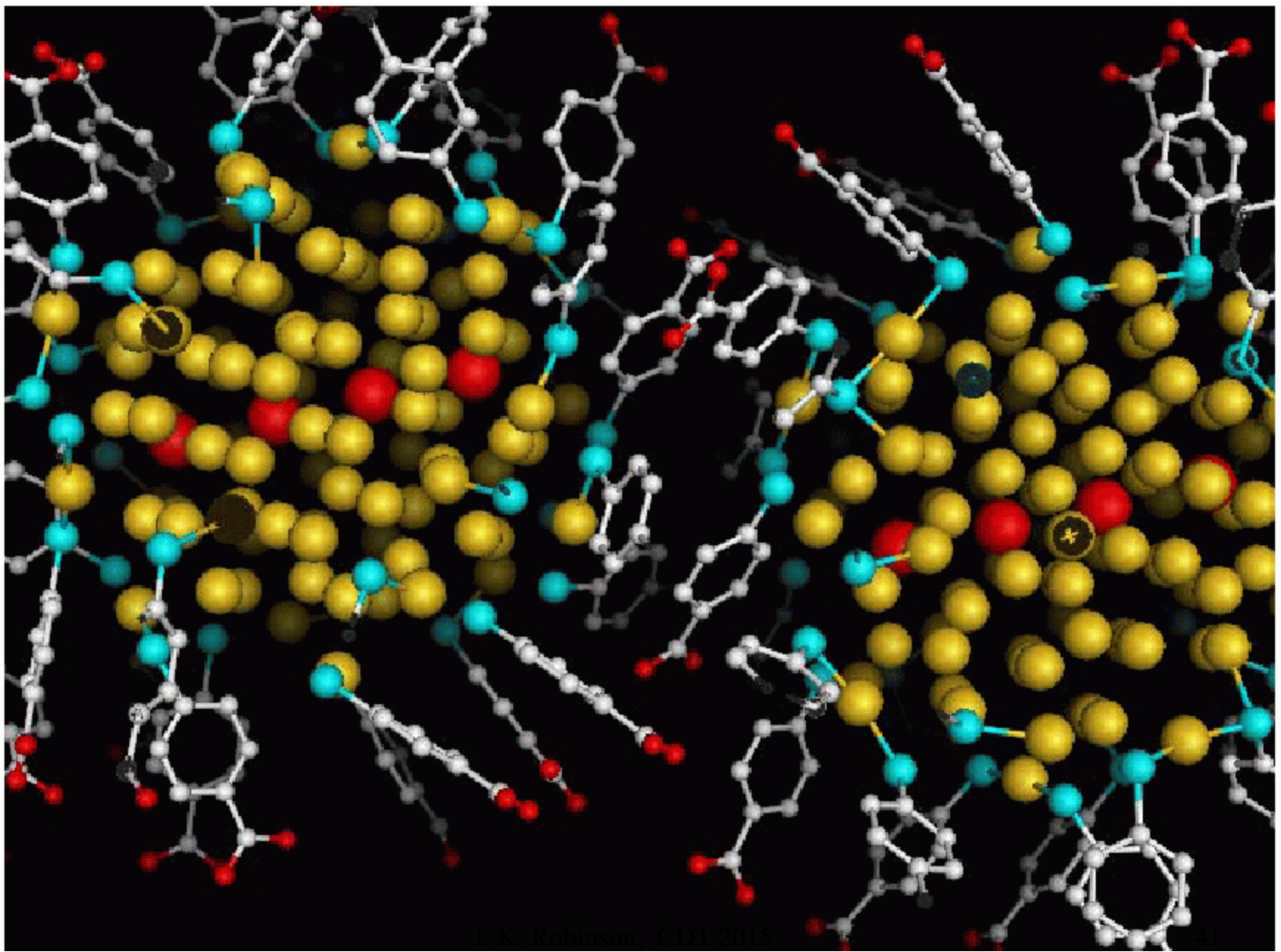


Gold-sulphur network in 1.6nm NC

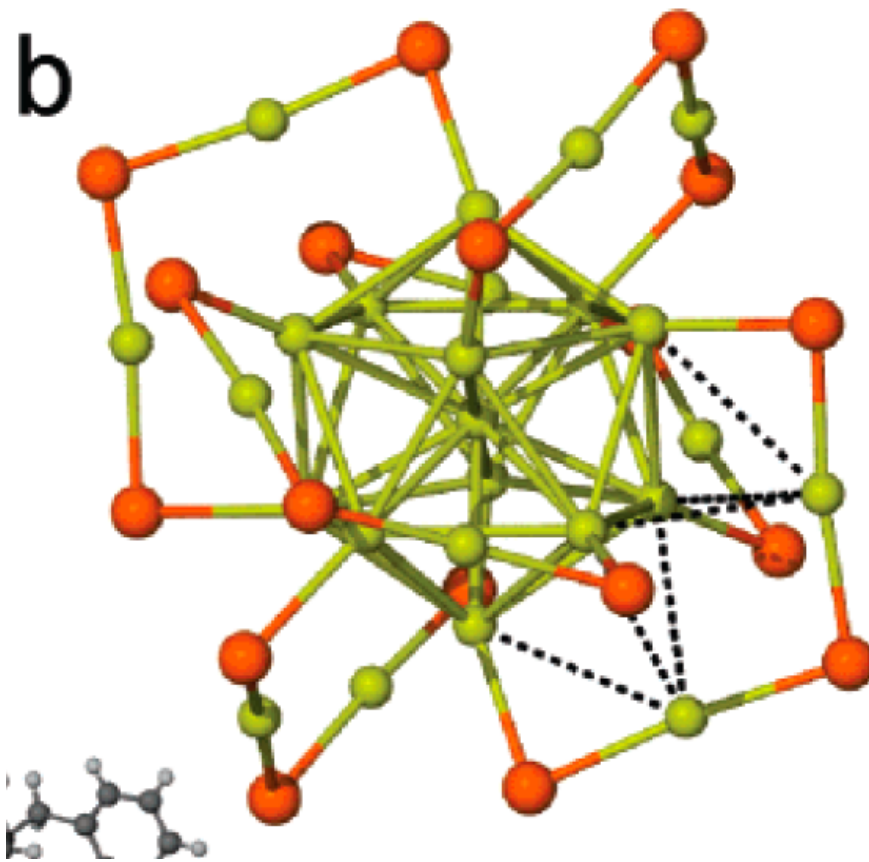
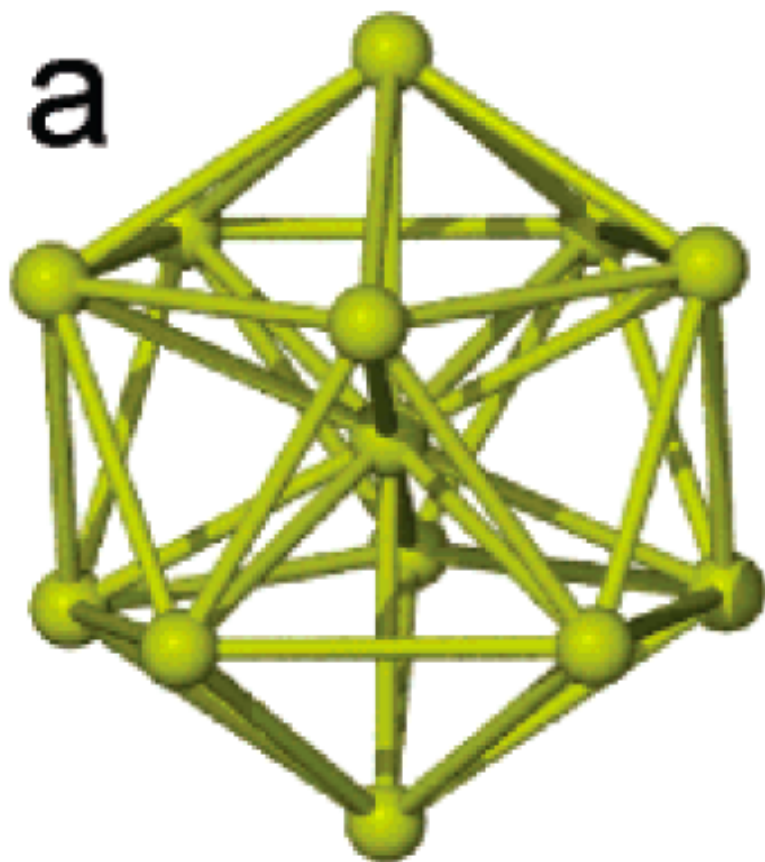
p-mercaptobenzoic acid (p-MBA)

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

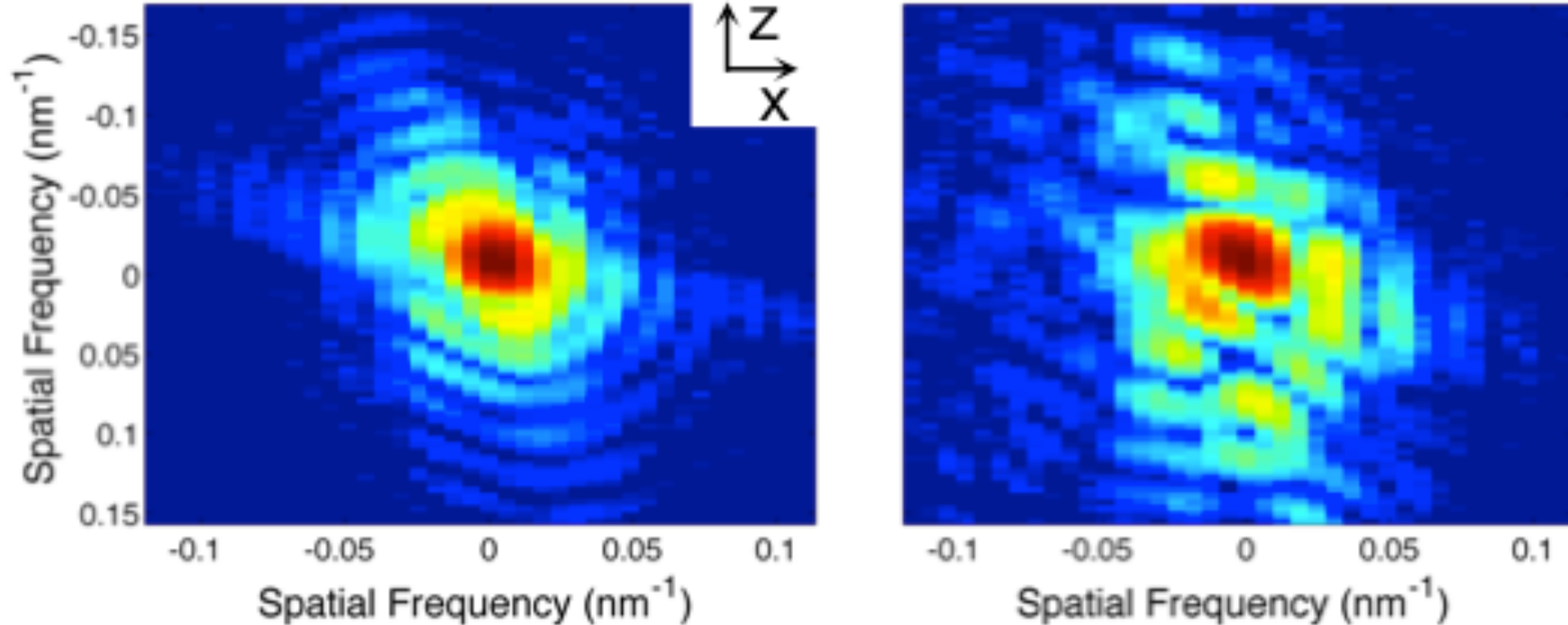




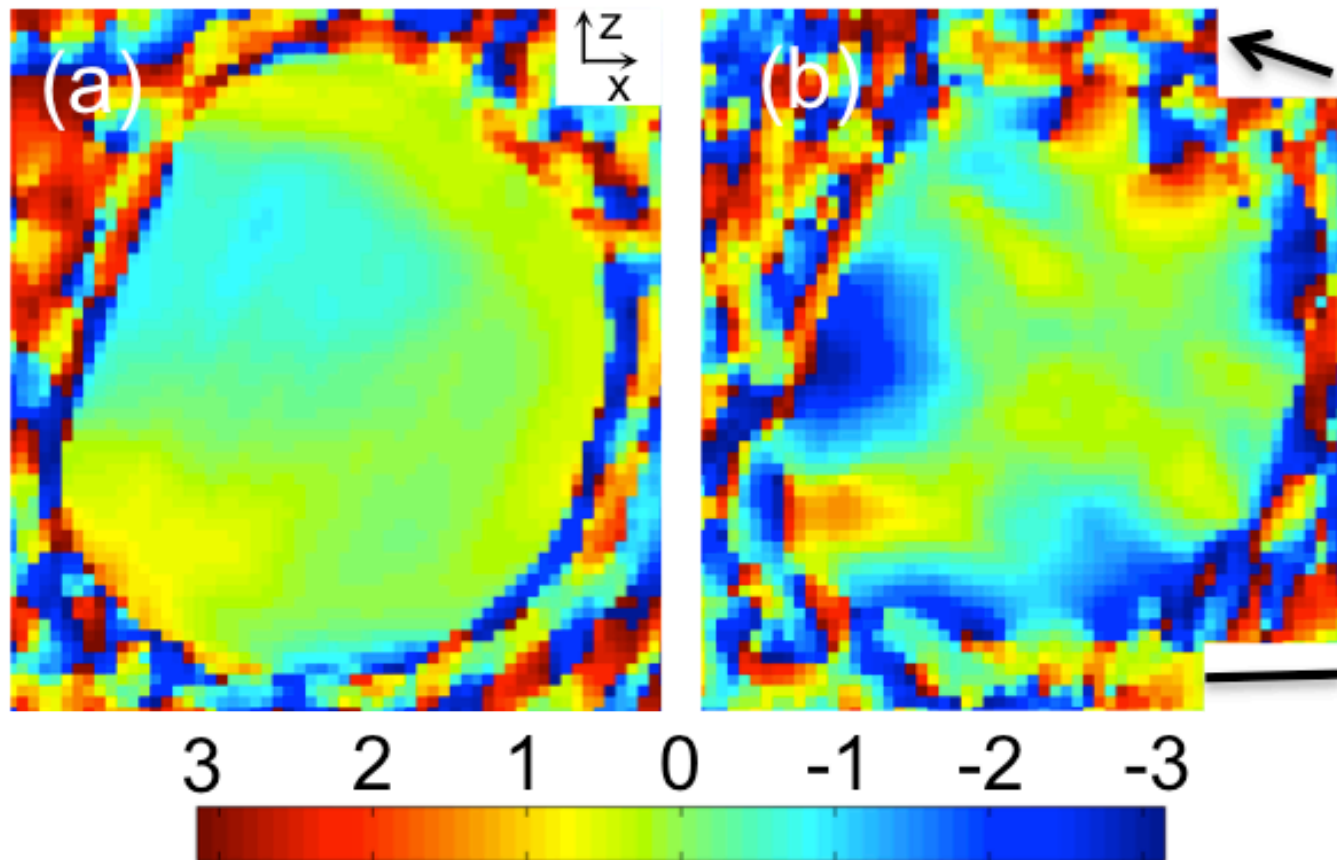
Gold-sulphur structure $\text{Au}_{13}(\text{Au}_2\text{RS}_3)_6$ $=\text{Au}_{25}\text{RS}_{18}$ Heaven et al, JACS 130 3755 (2008)

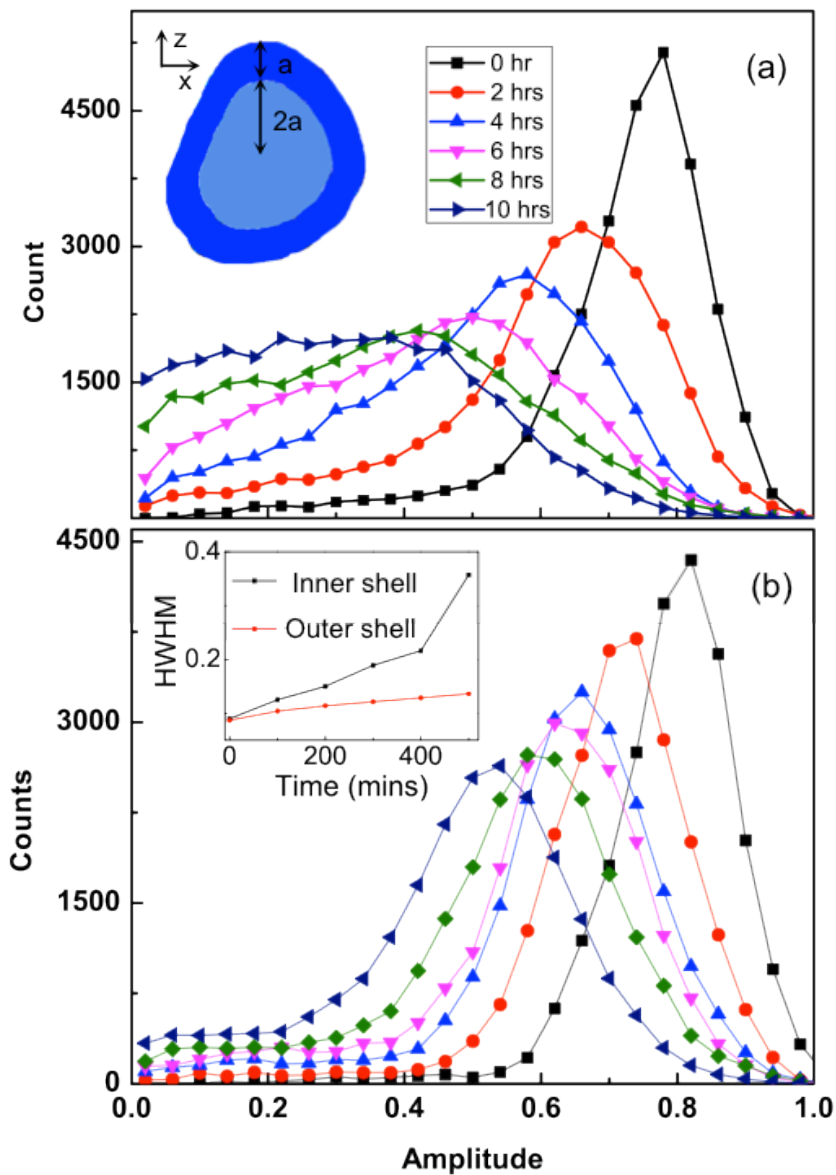
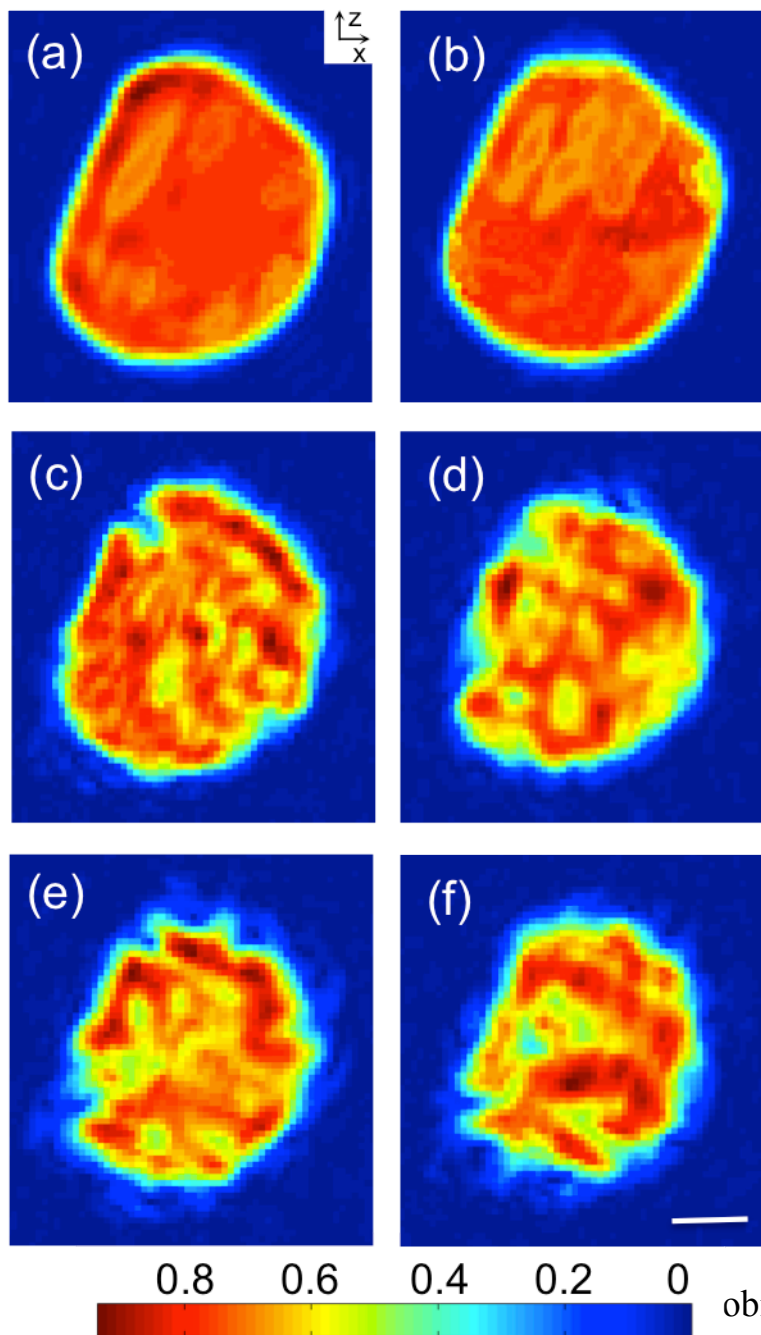


“Atomic Diffusion within Individual Au Nano-crystal”, Gang Xiong, et. al., Scientific Reports 4 6765 (2014)



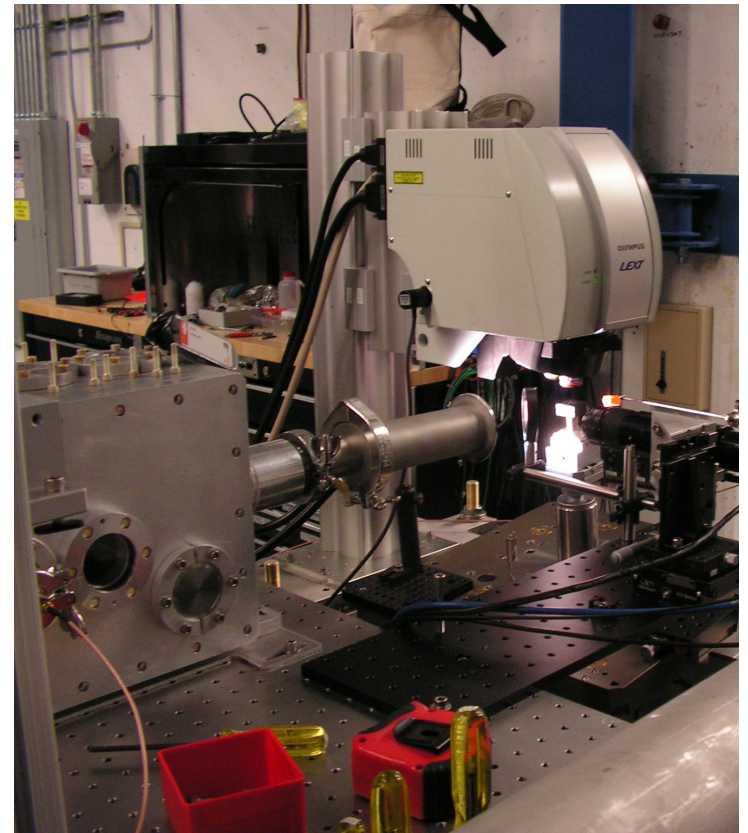
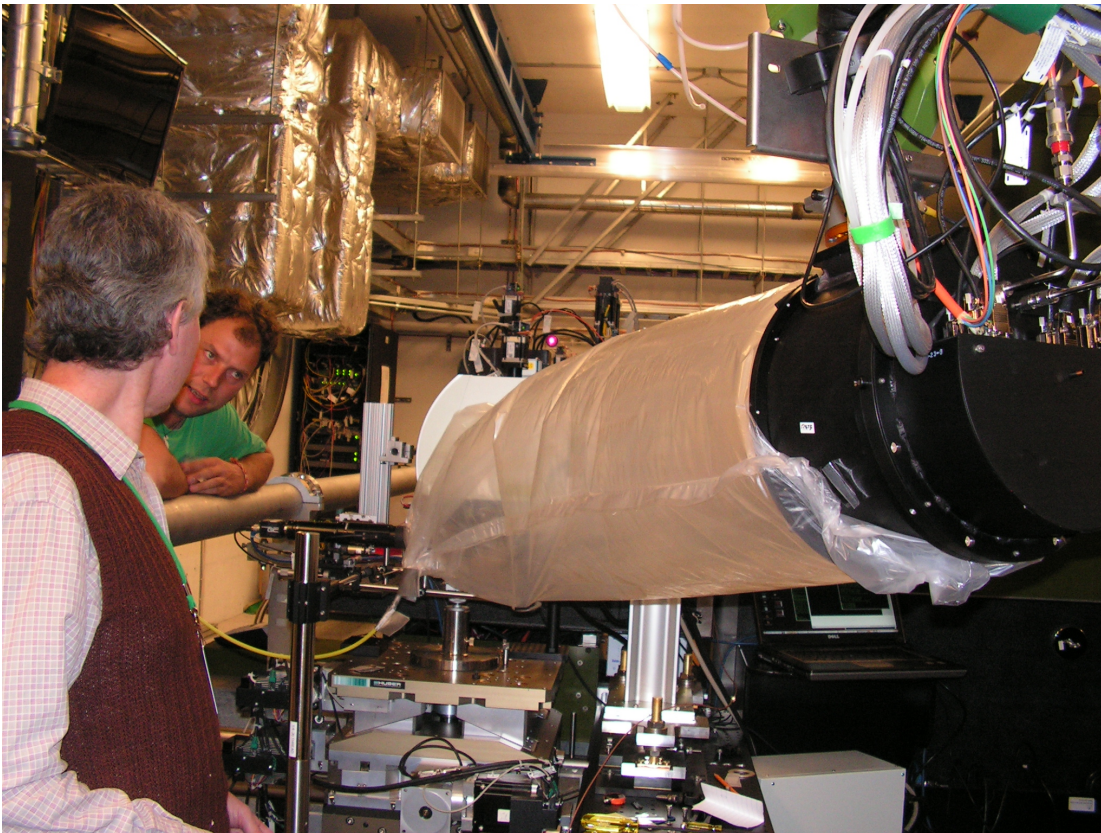
“Atomic Diffusion within Individual Au Nano-crystal”, Gang Xiong, et. al.,
Scientific Reports 4 6765 (2014)





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

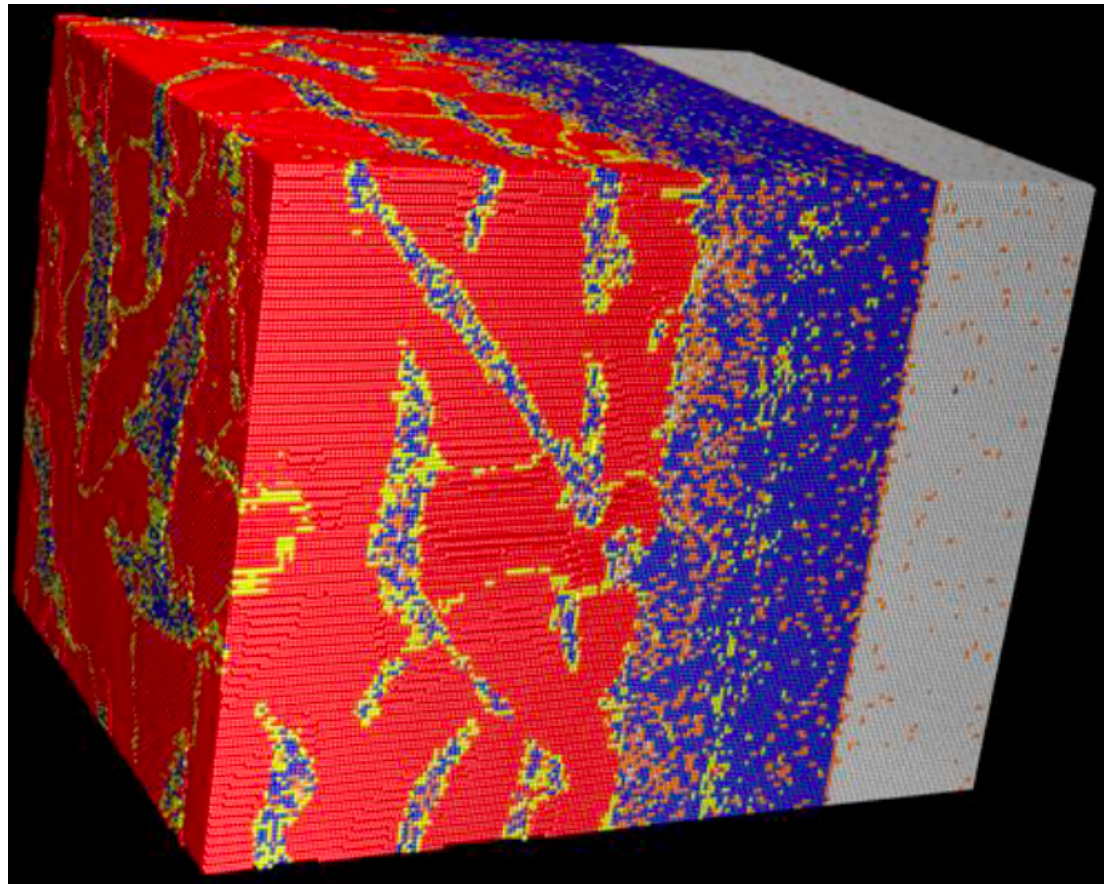


MD simulation of Shock Wave

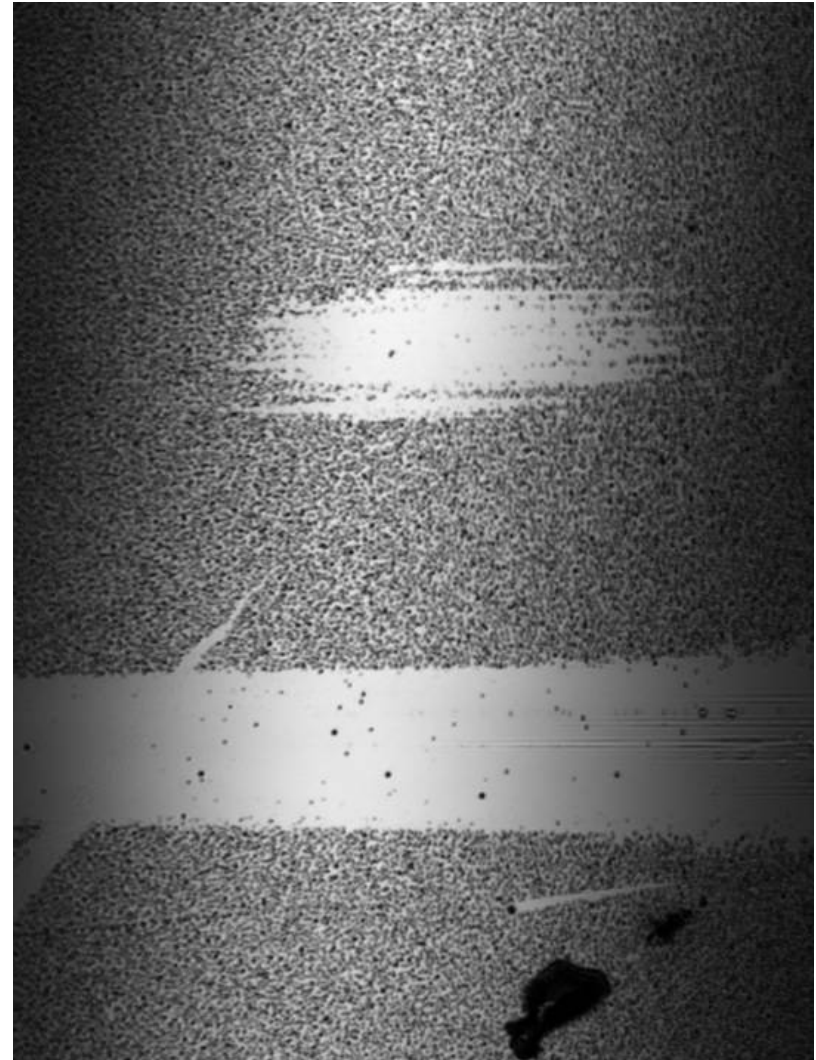
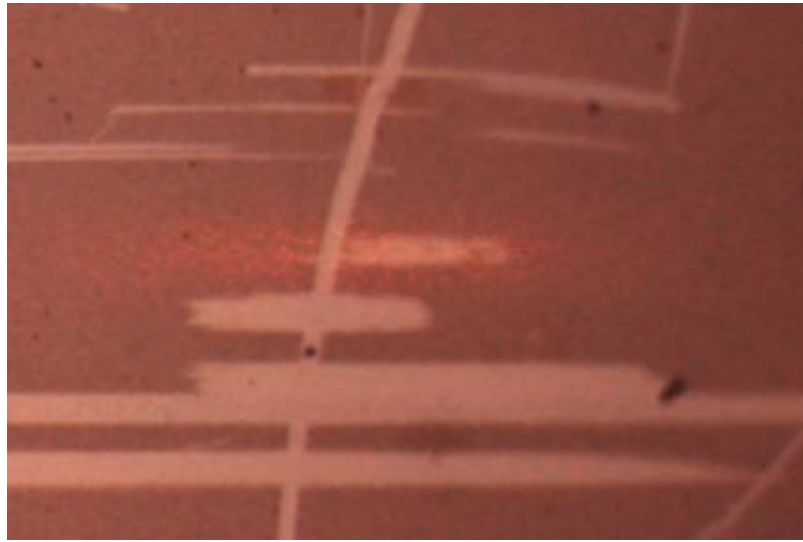
Damage in Fe along (001) direction

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

Science, 296 1681 2002

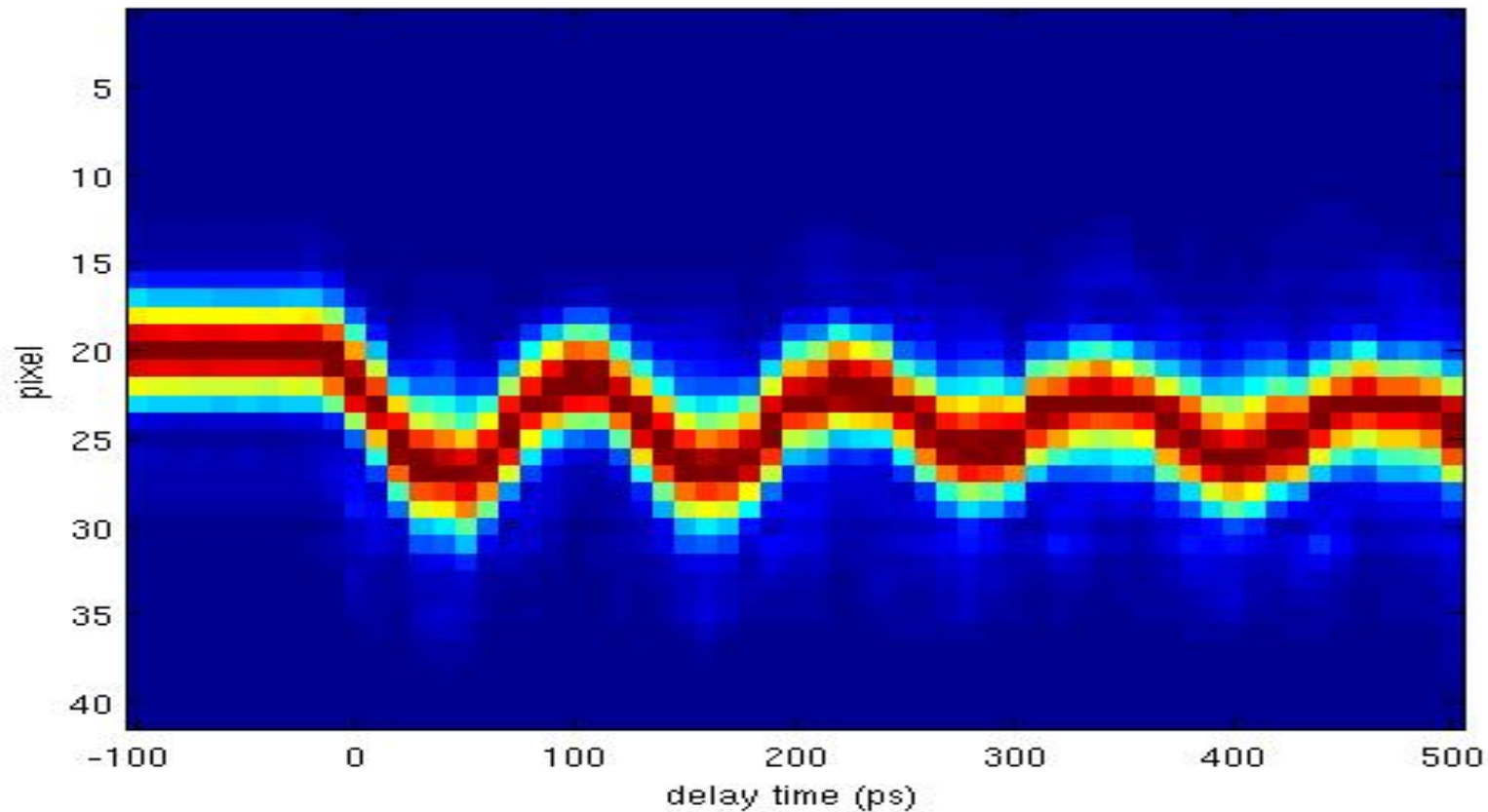


Visible and Confocal microscopy



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

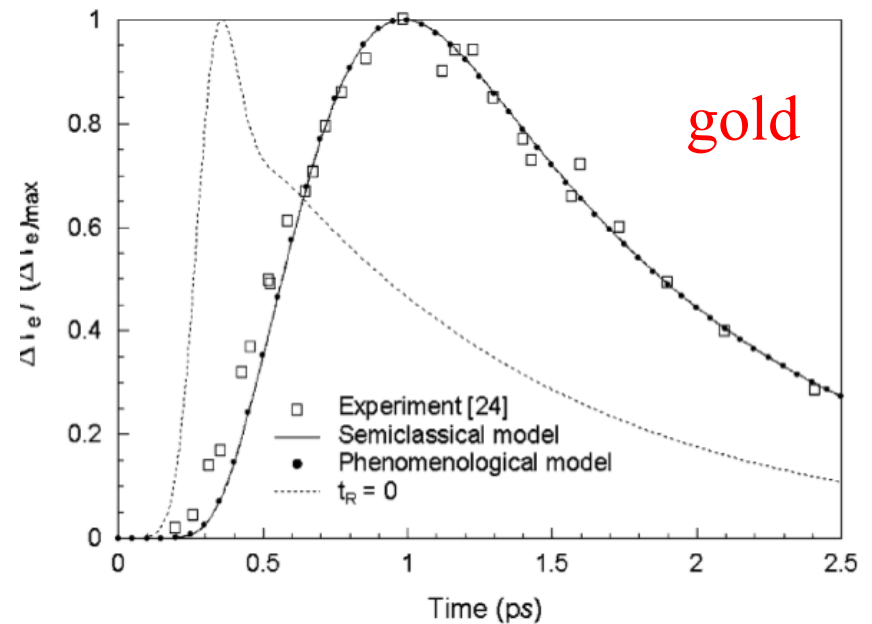
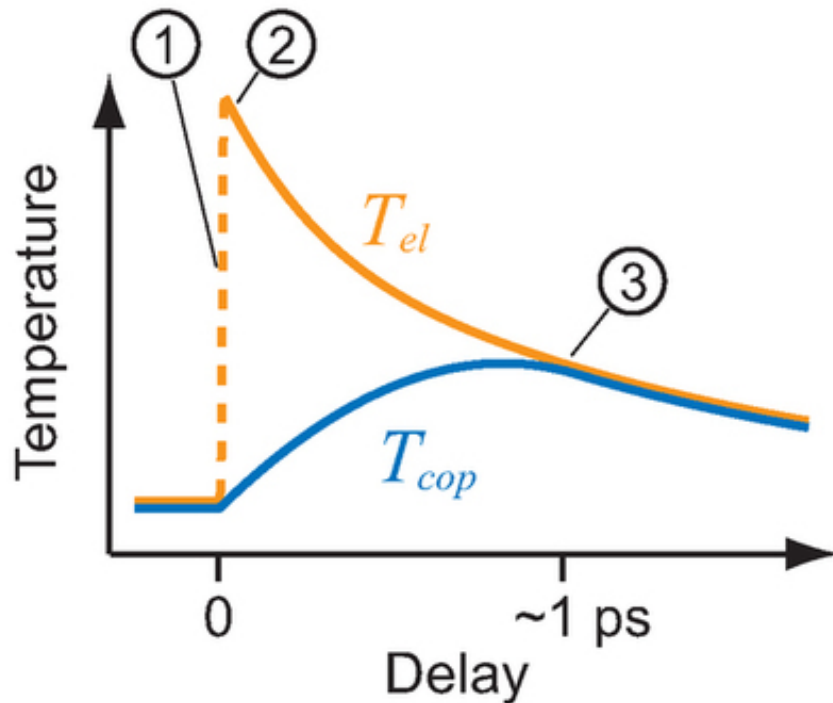
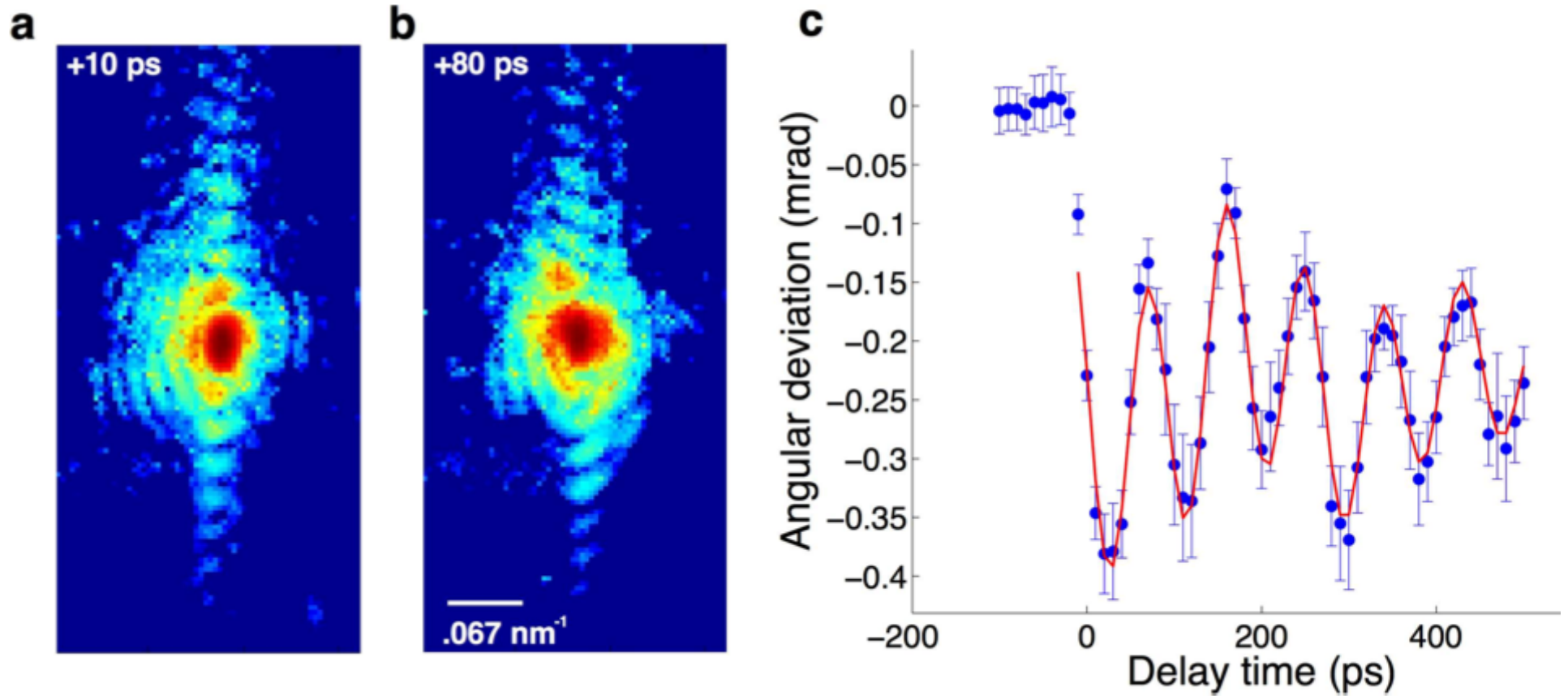


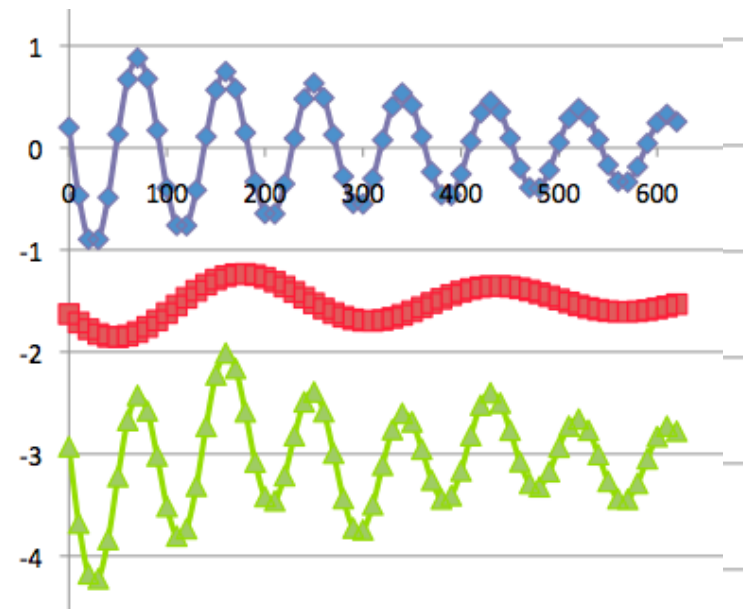
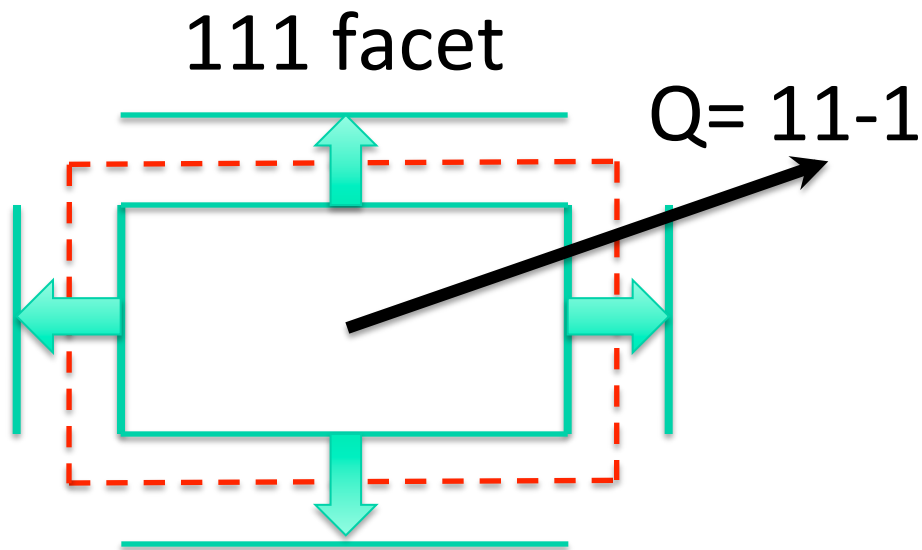
fig. 2. Comparison of the change in electron temperature at the front surface of an 80-nm gold film irradiated by a 2.8 mJ/n², 800 nm, 150-fs laser pulse.

Time resolved Bragg peak position



Two Normal Modes of Vibration

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



$$T_1 = 90\text{ps} \quad h_1 = 145\text{nm} \quad c_S = 3240\text{ m/s}$$

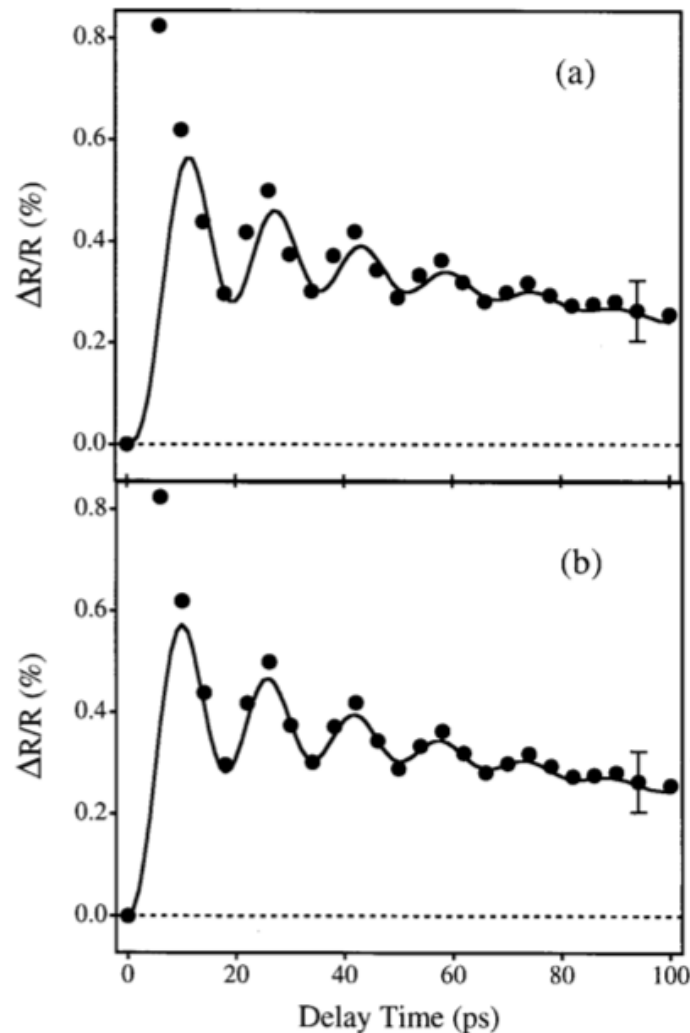
$$T_2 = 259\text{ps} \quad h_2 = 420\text{nm}$$

J. K. Robinson, CDD 2015

Ultrafast Absorption Spectroscopy

24nm Au nanoparticles in H₂O, converted to radius

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

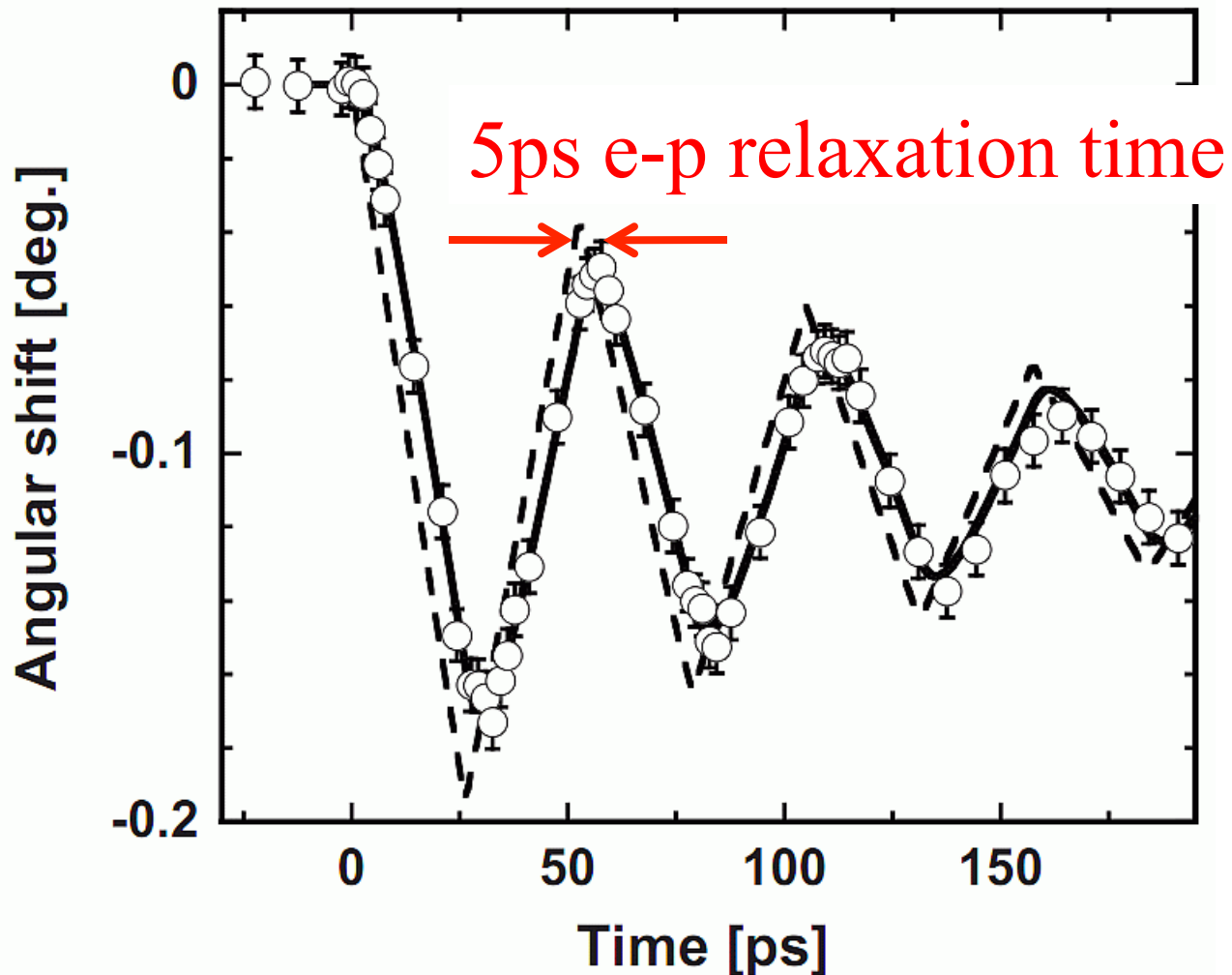


(a) Simple normal mode analysis with impulse heating.

(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)

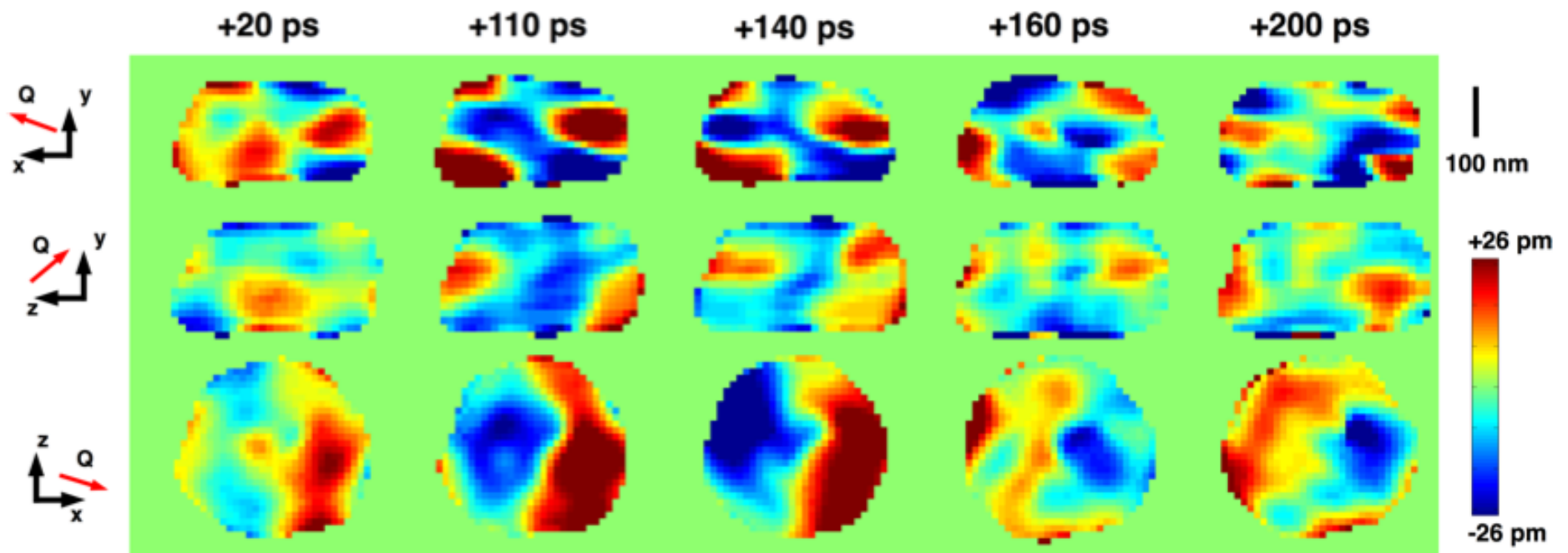


Dynamic imaging of displacements

CDI inversion of 3D diffraction patterns

1000 frames averaged at each point of rocking curve

Jesse Clark et al, Science 341 56 (2013)

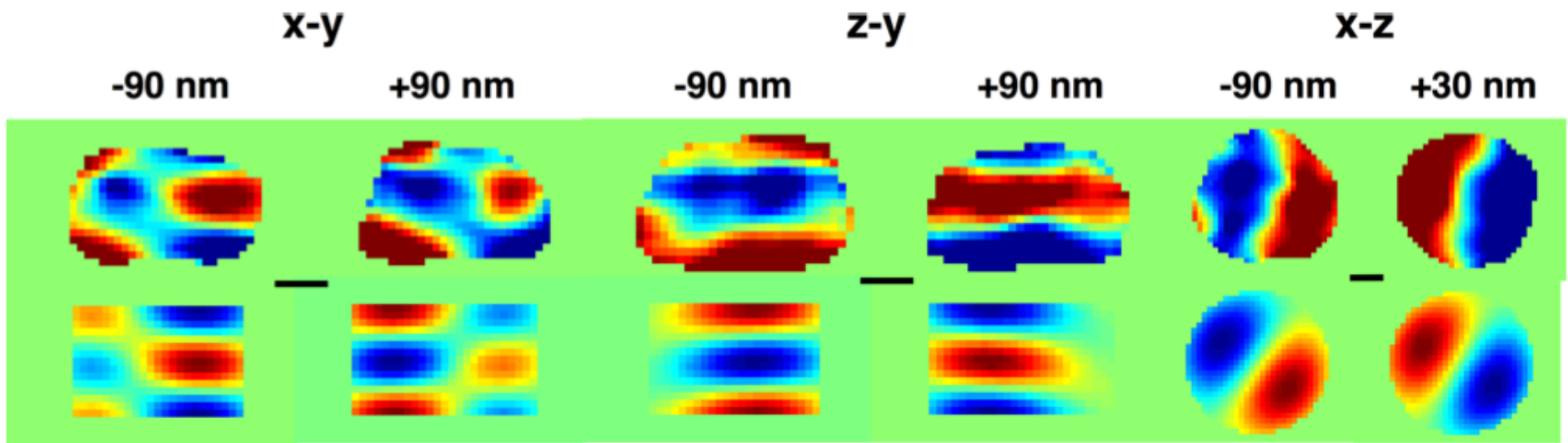


Dynamic imaging of displacements

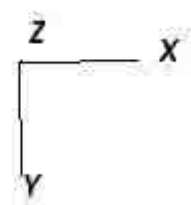
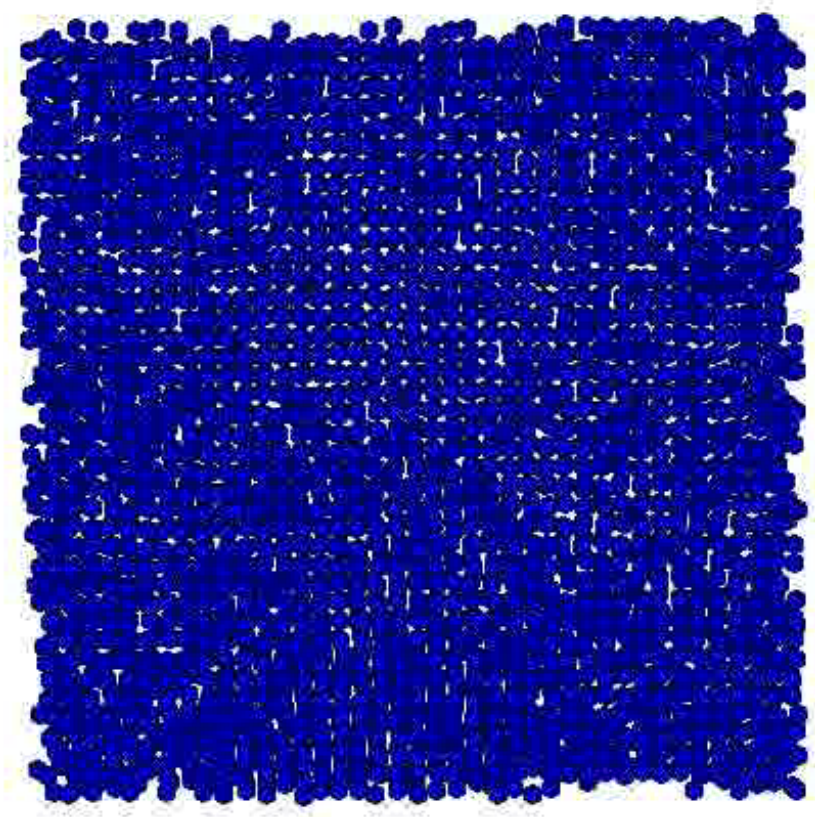
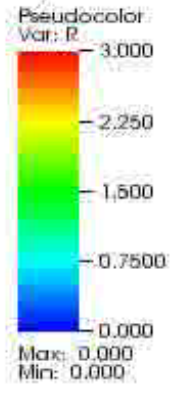
CDI inversion of 3D diffraction patterns

Comparison with (1,1) normal mode of cylinder

Jesse Clark et al, Science 341 56 (2013)

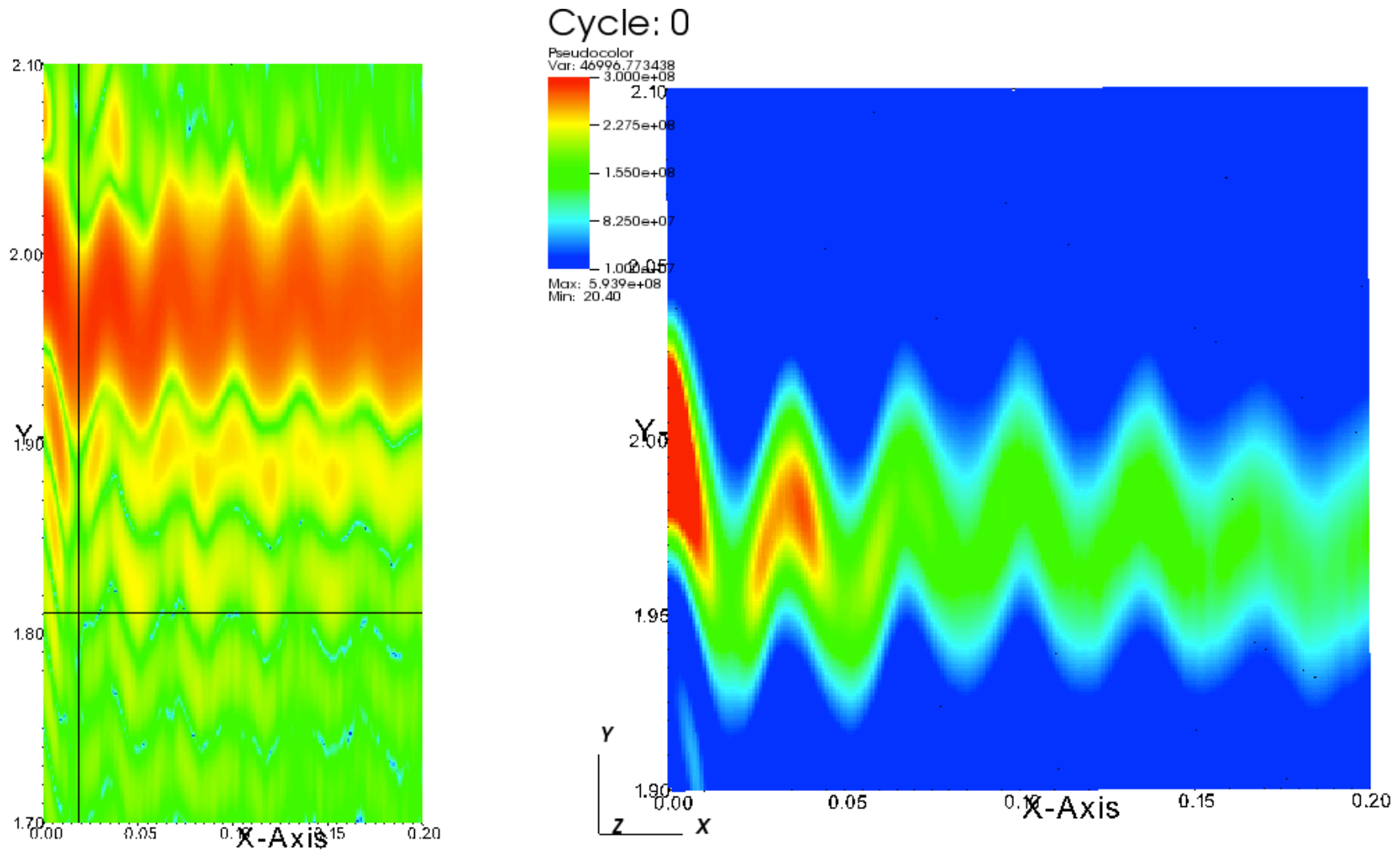


DB: dump.0
Cycle: 0 Time: 0



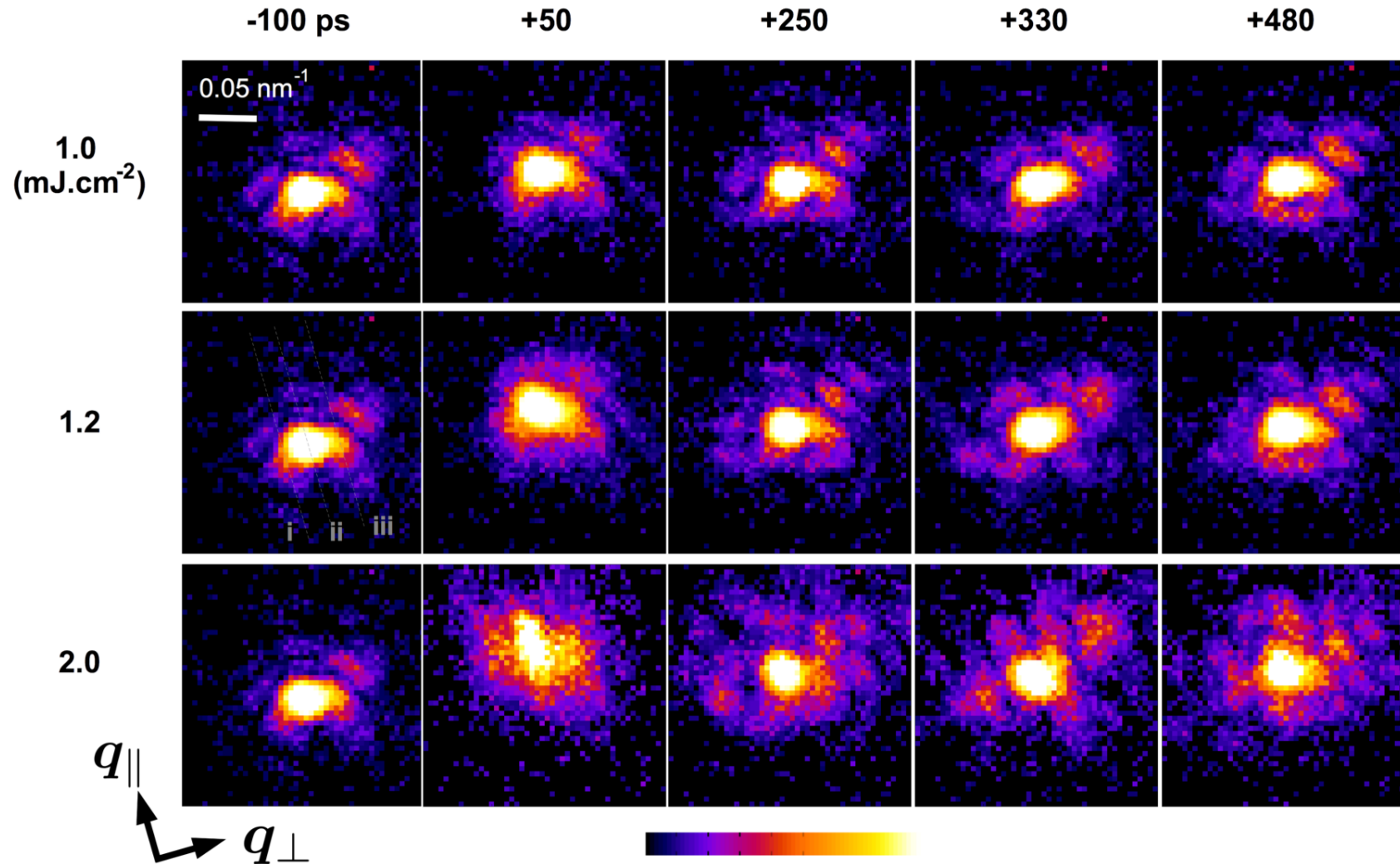
MD Simulation (LAMMPS)

Andy Higginbotham and Loren Beitra



Dependence on Laser Fluence

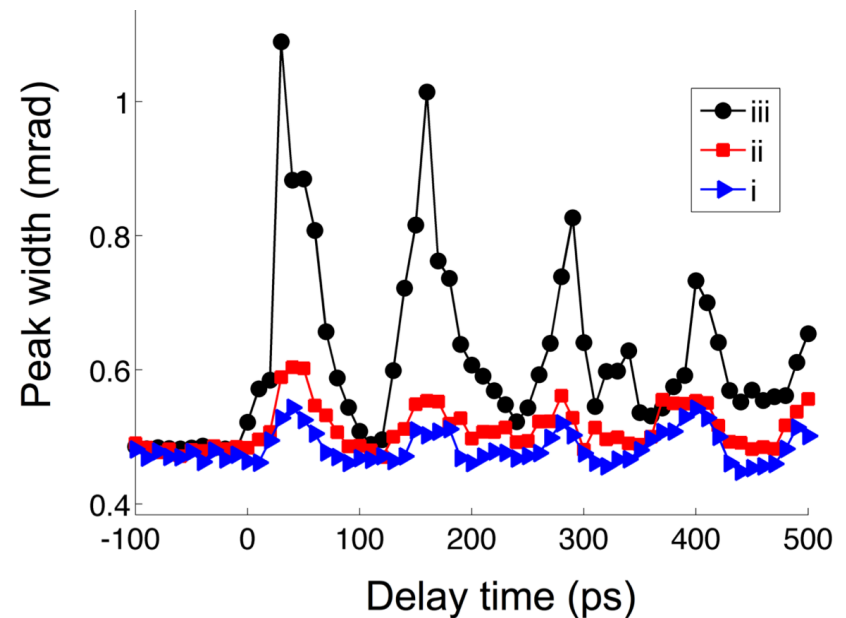
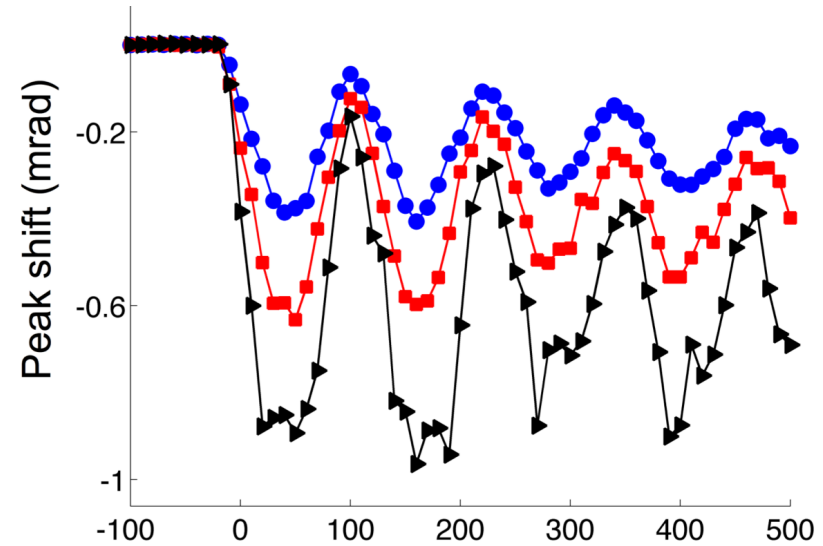
Jesse Clark et al, to be published



Dependence on Laser Fluence

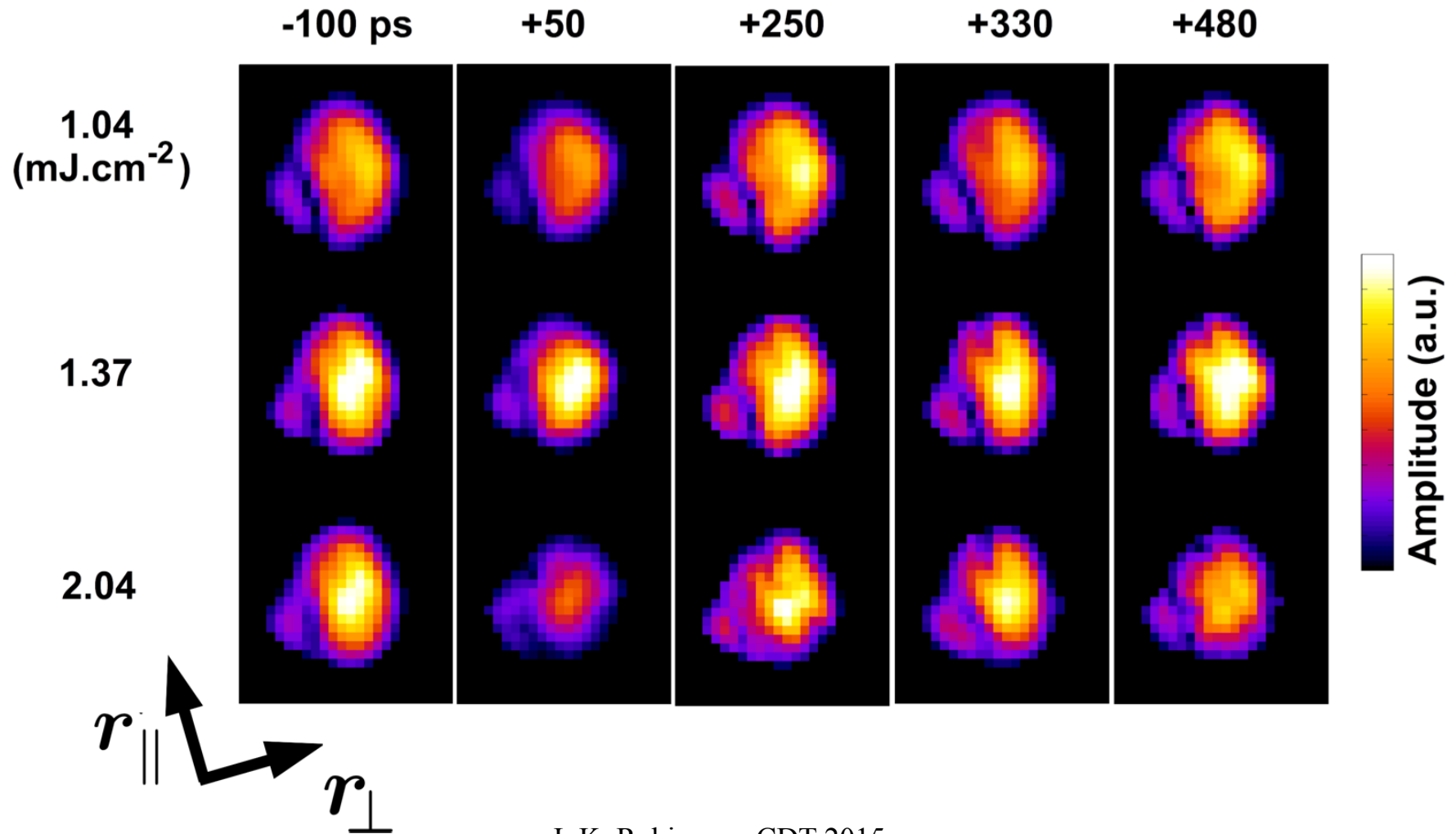
Jesse Clark et al, to be
published

1.0 mJ cm⁻²
1.2 mJ cm⁻²
2.0 mJ cm⁻²



Dependence on Laser Fluence

Jesse Clark et al, to be published



Bragg Coherent Diffraction Imaging

- Nanoparticle imaging by BCDI
- Complex density can image strain
- Strain associated with nano-shape
- Inhomogeneities during diffusion
- Ultrafast snapshots of vibrations
- Transient melting
- Materials science in the time domain