Synchrotron X-rays in the Physics of Nanomaterials

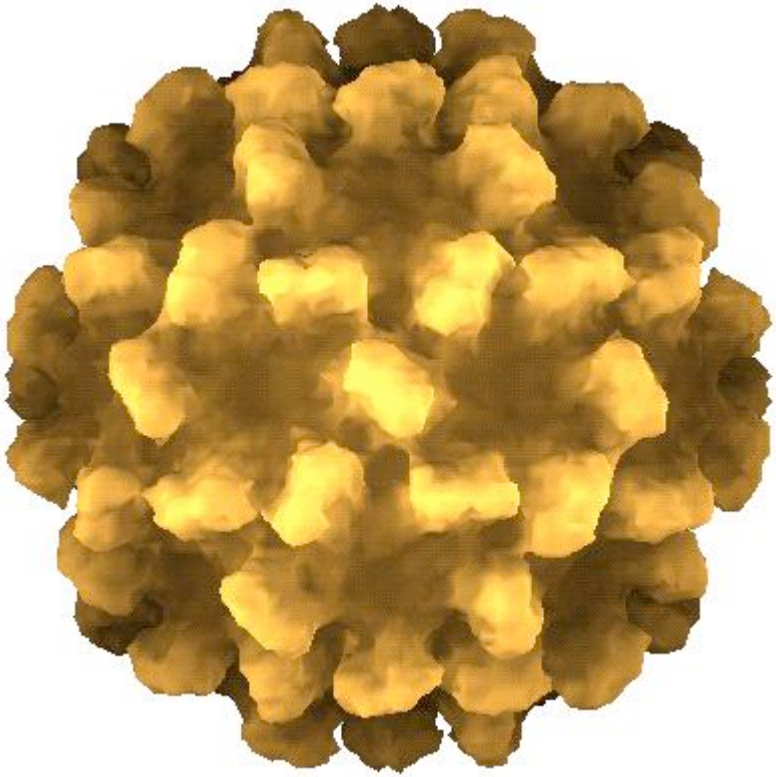
Ian Robinson

University College London

Diamond Light Source

Inaugural Lecture, Feb 27, 2008

Tomato Bushy Stunt Virus
1980



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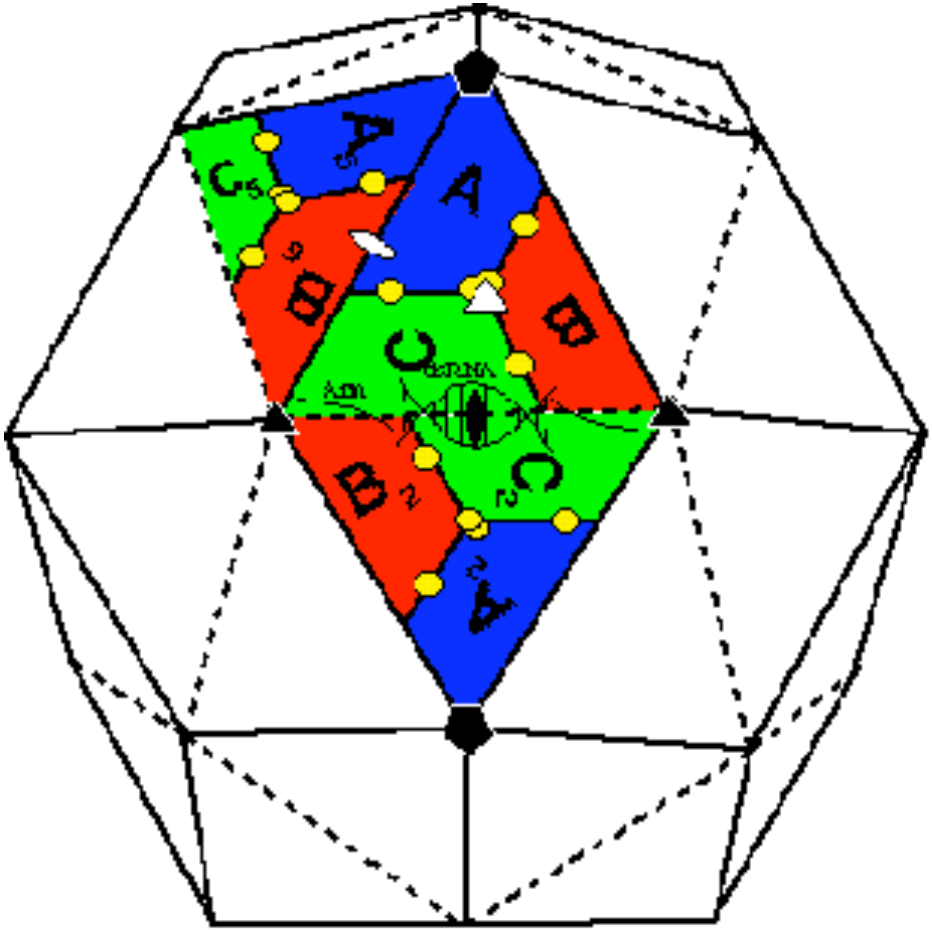
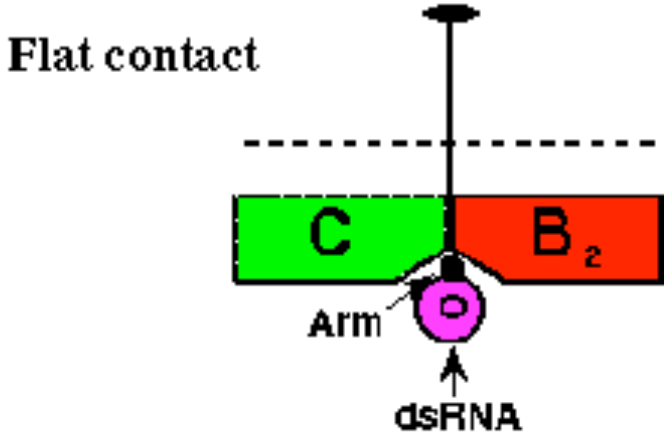
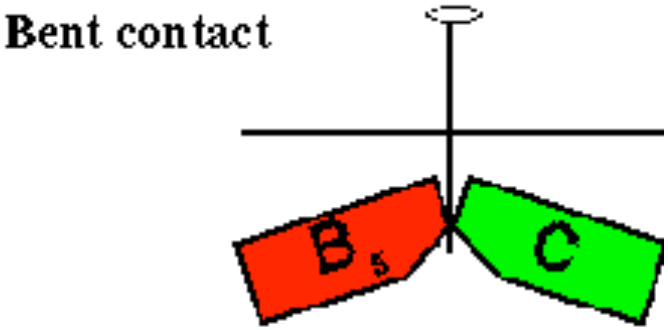
Quasiequivalent Contacts

Caspar and Klug, 1971

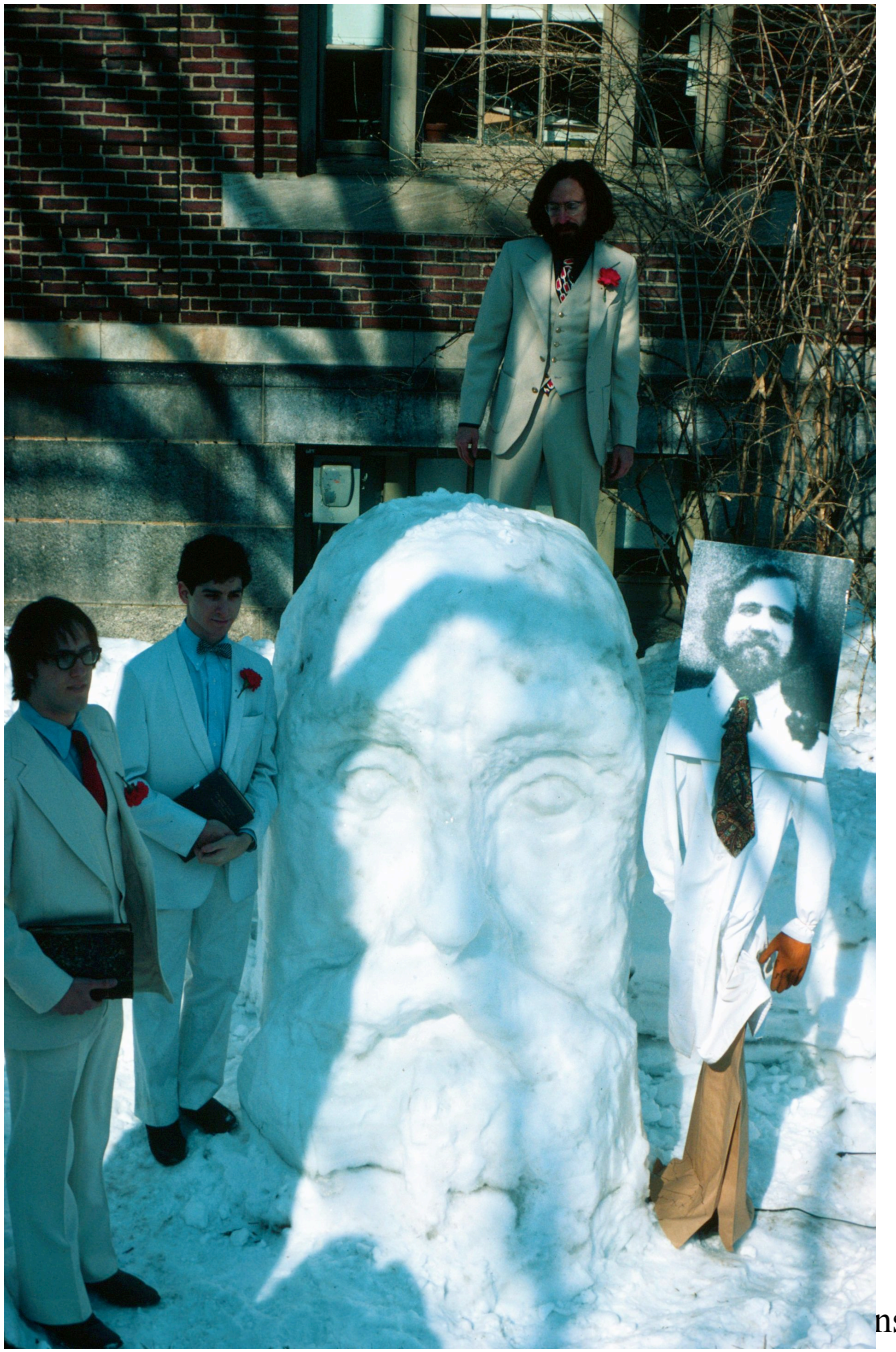


T=3





Harvard 1978



First UHV Experiments (1981)

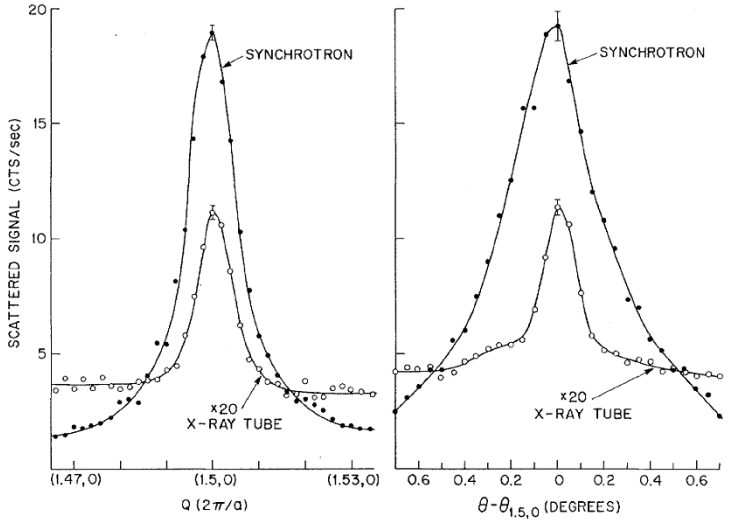
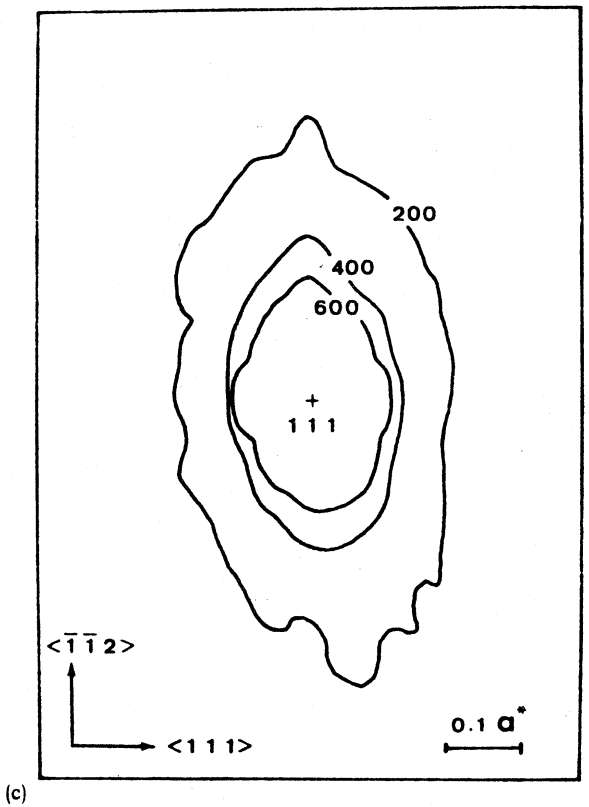


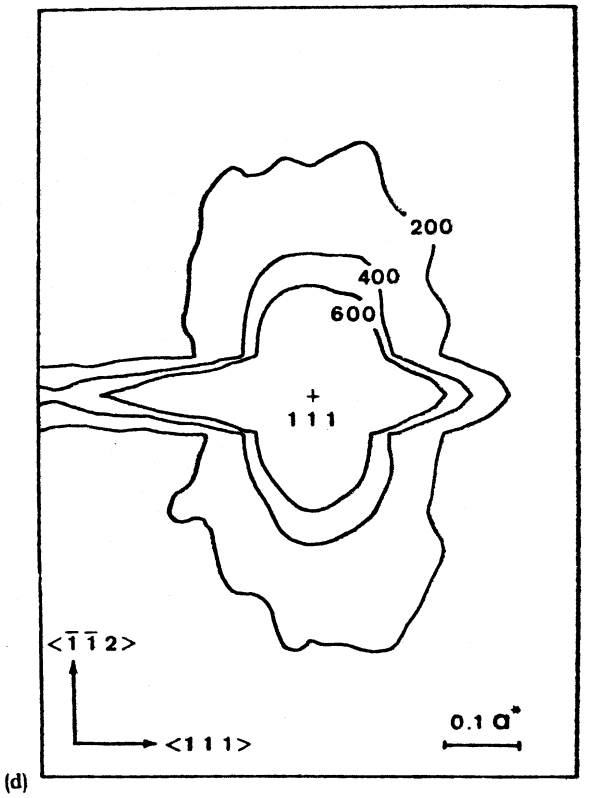
FIG. 1. A plot of the $(\frac{3}{2}, 0)$ Bragg reflection as a function of the momentum transfer $Q(2\pi/a)$ and the crystal's mosaic spread (deg).

P. Eisenberger and W. C. Marra,
PRL **46** 1081 (1981)
experiments done at SSRL

Diffuse Scattering from Si Wafer



Unpolished wafer



40 microns removed

N. Kashiwagara, J. Harada and M. Ogino, J. Appl. Phys 54 2706 (1983)

Surfaces in Dynamical Diffraction

A. M. Afanas'ev *et. al.*, Acta Cryst **A40** 352 (1984)

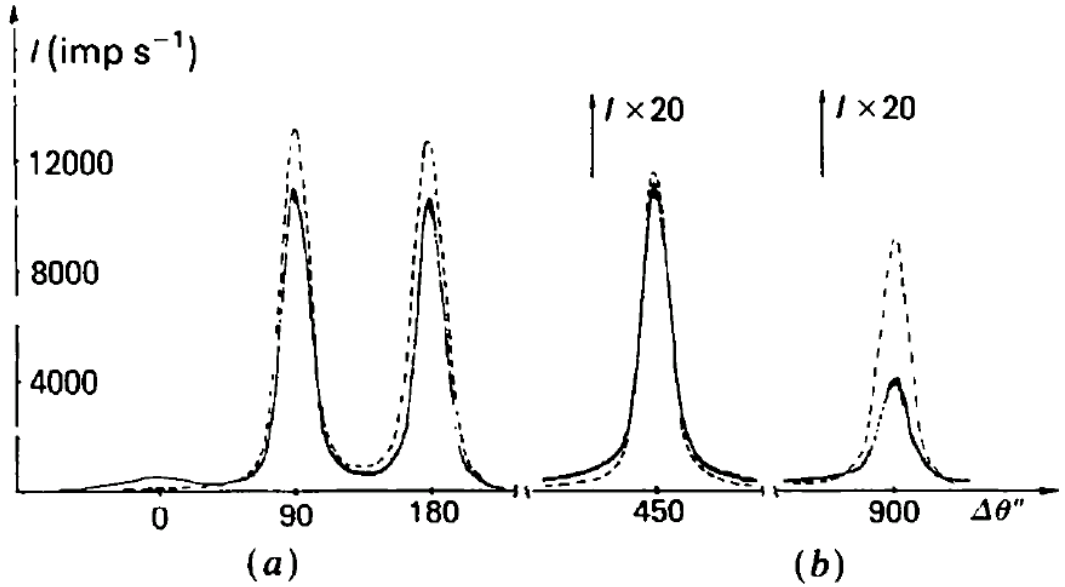


Fig. 2. TDC spectra from Ge(111) crystals, $(n, -n, n)$ arrangement, symmetric Bragg diffraction for (a) $\alpha = 90''$, (b) $\alpha = 450''$. Dashed line for ideal crystal, solid line for crystal first polished with diamond paste and then partially etched.

Effect of etching seen in triple-axis measurement of rocking curve

Scattering of X-rays From Crystal Surfaces

S.R.Andrews & R.A.Cowley JPCM **18** 6427 (1985)

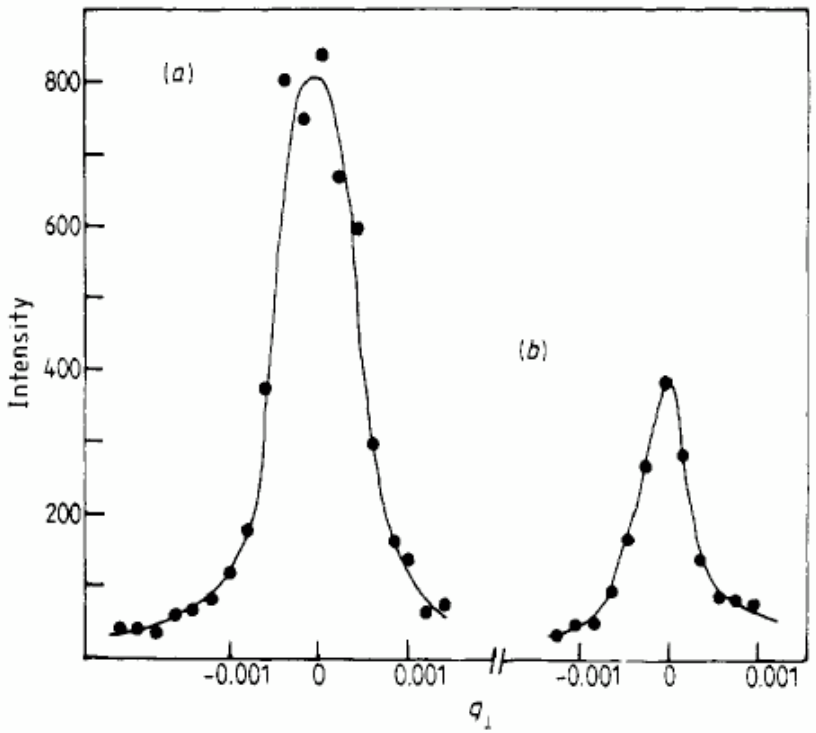
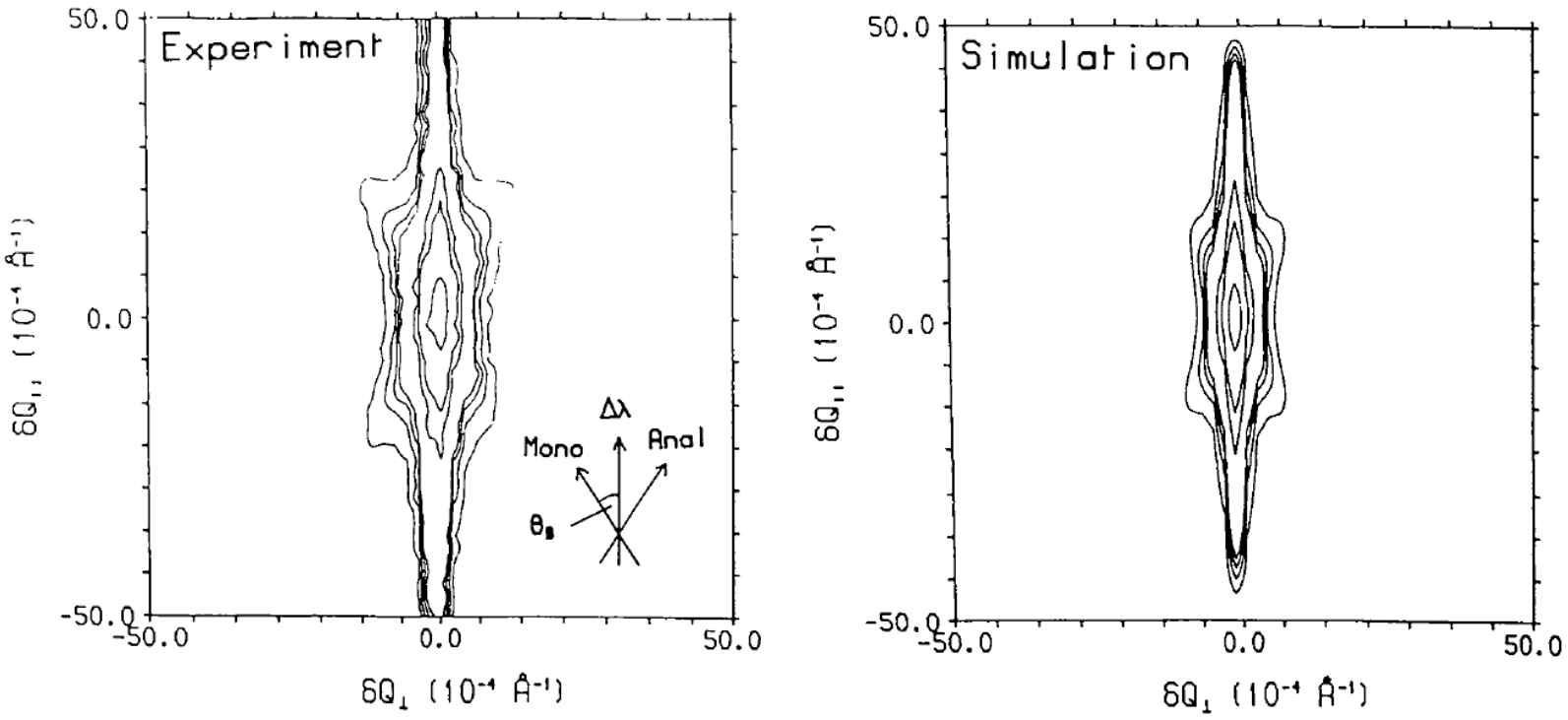


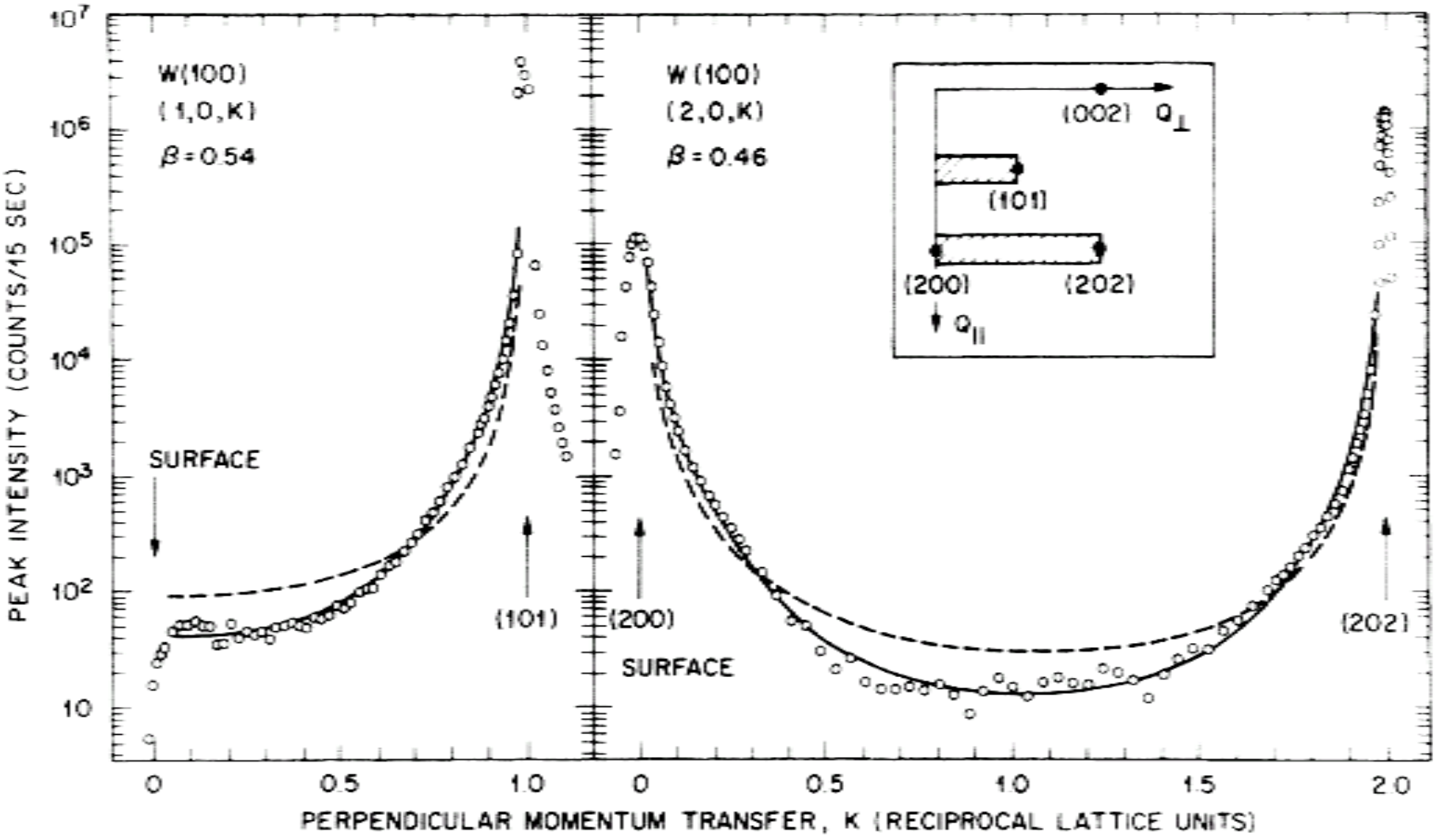
Figure 1. The intensity of scattering, as a function of q , (*a*) for $\boldsymbol{Q}$ = (0, 0, 4.005) (vertical scale in counts per 2 s) and (*b*) $\boldsymbol{Q}$ = (0, 0, 4.025) (vertical scale in counts per 300 s) in the GaAs sample with an (001) surface corresponding to figure 2, curve B.

Resolution Function of the Triple-Axis Spectrometer

R. A. Cowley *et. al.*, Acta Cryst **A45** 415 (1989)



CRYSTAL TRUNCATION RODS AND SURFACE ROUGHNESS



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Diffraction as a Surface Integral

Die äußere Form der Kristalle
in ihrem Einfluß auf die Interferenzerscheinungen
an Raumgittern

Von M. v. Laue

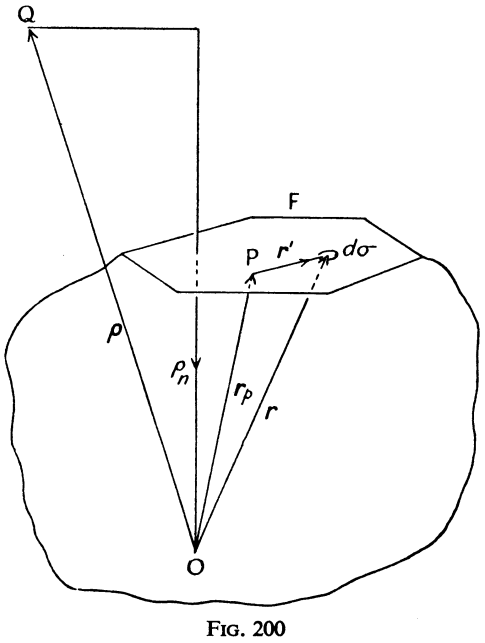
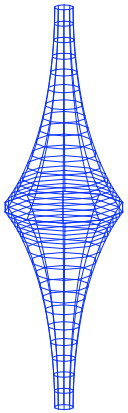


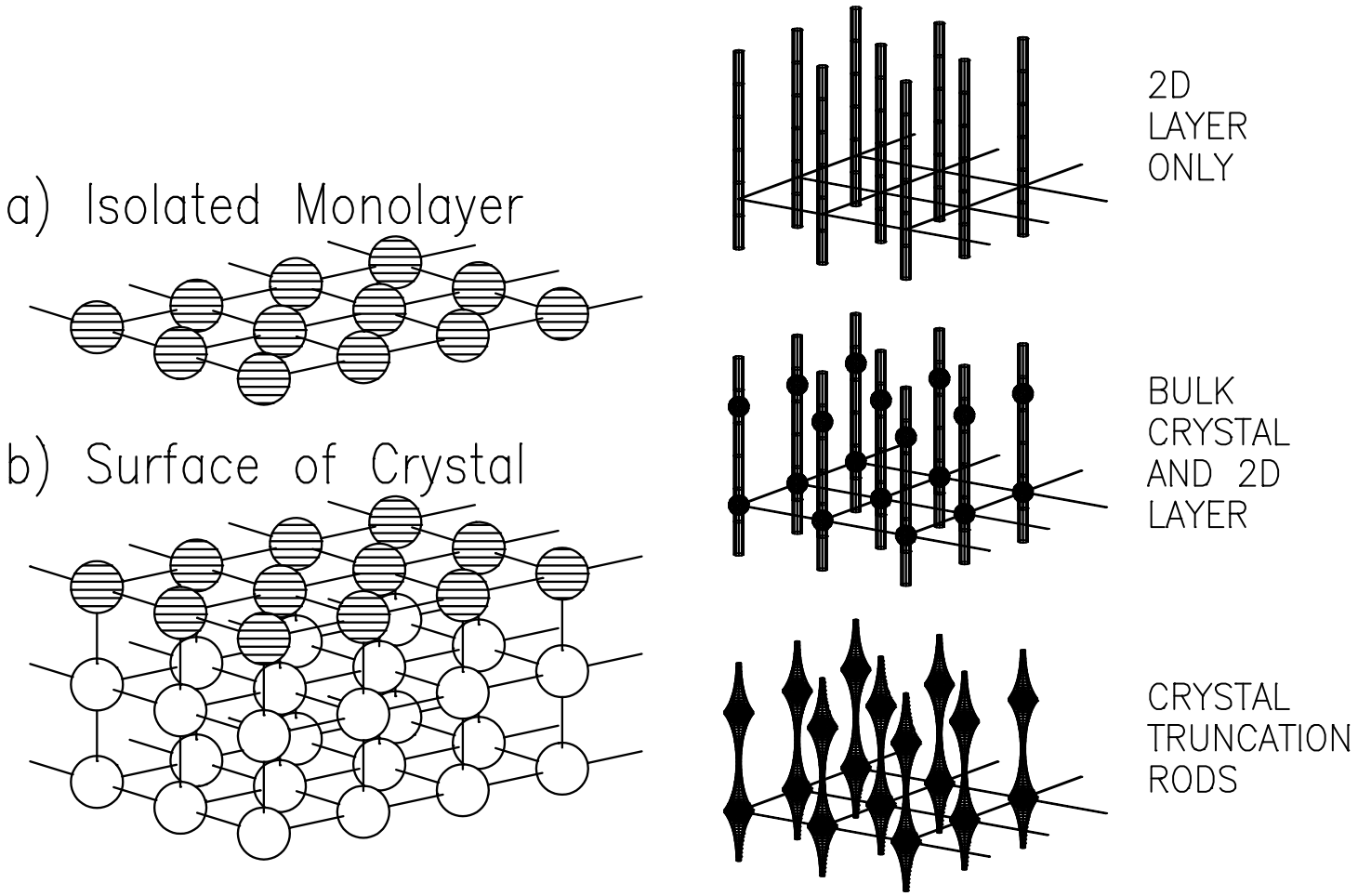
FIG. 200

Annalen der Physik [5] 26 55 (1936)

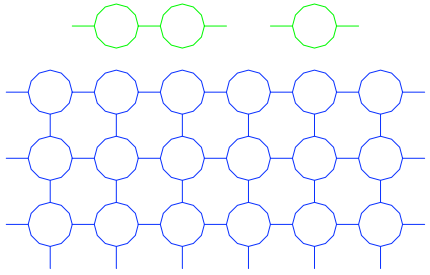


"Stacheln"

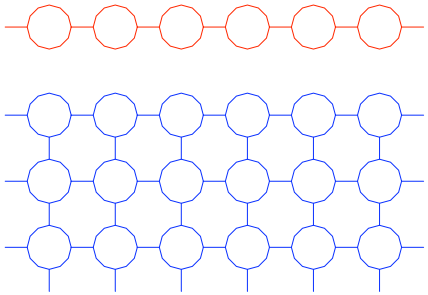
Crystal Truncation Rods (1986)



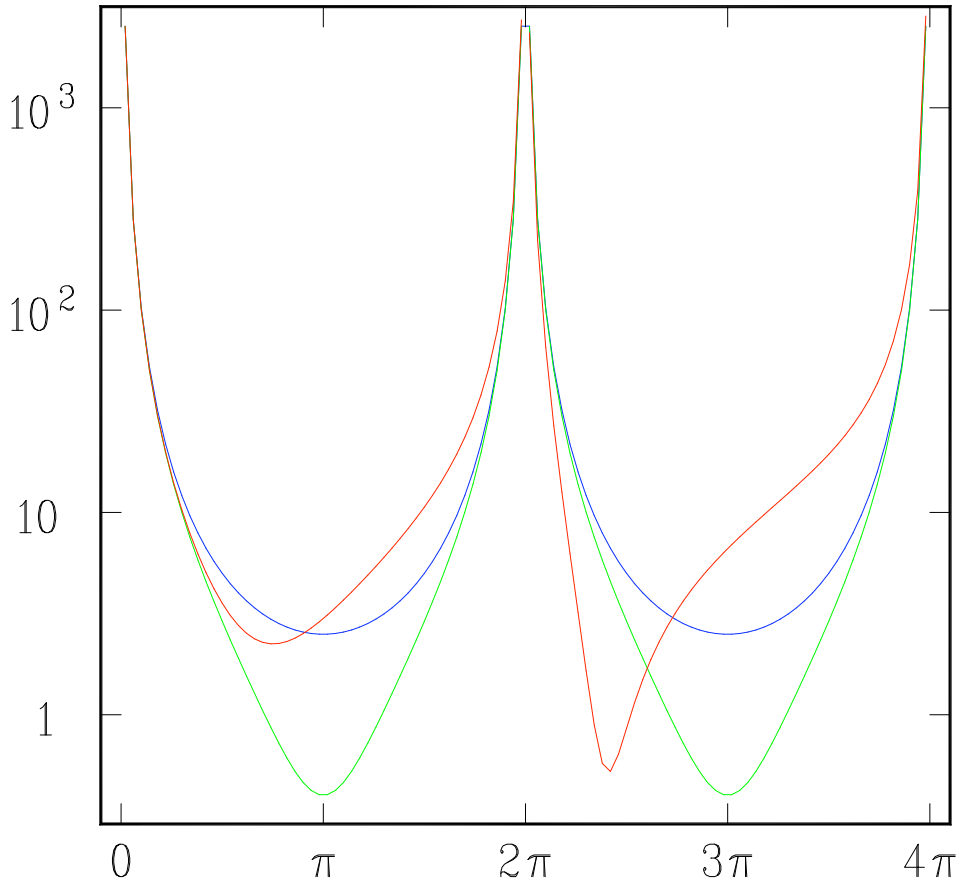
CTR is Sensitive to Surface Structure



ROUGH SURFACE

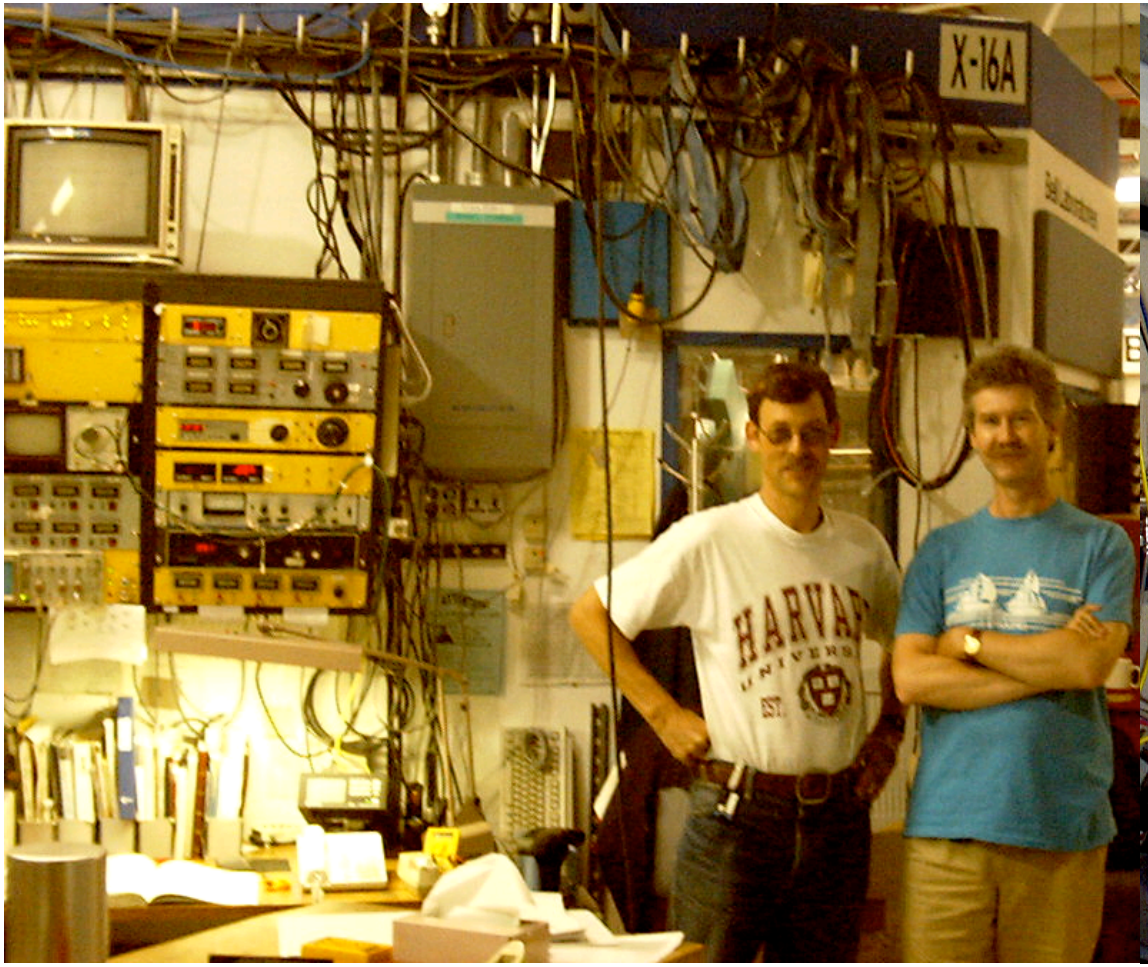


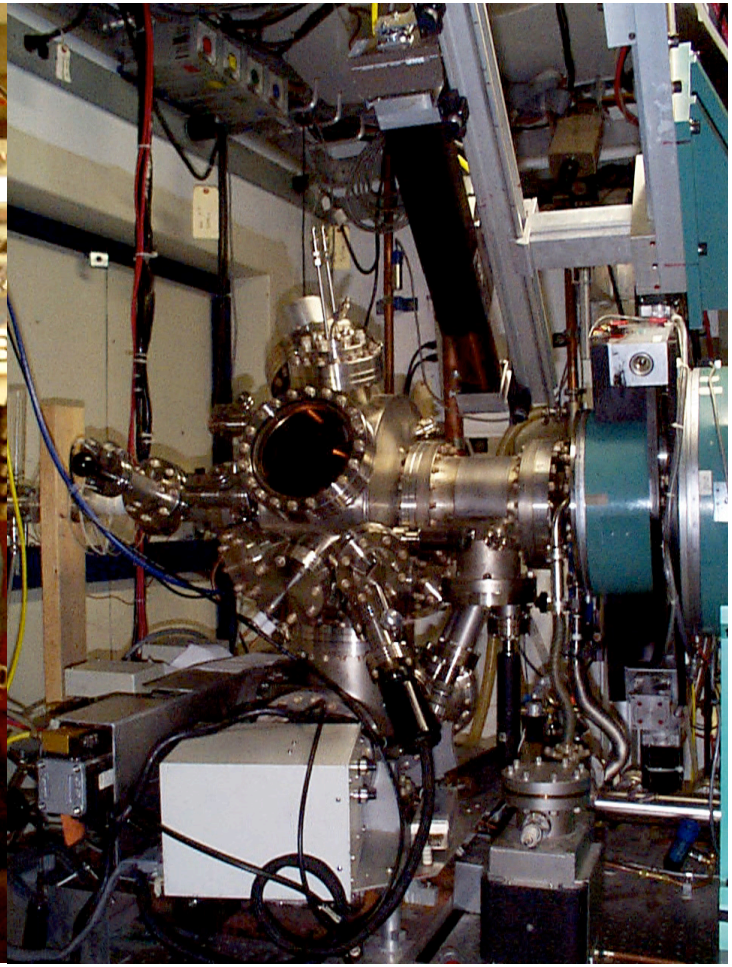
EXPANDED LAYER

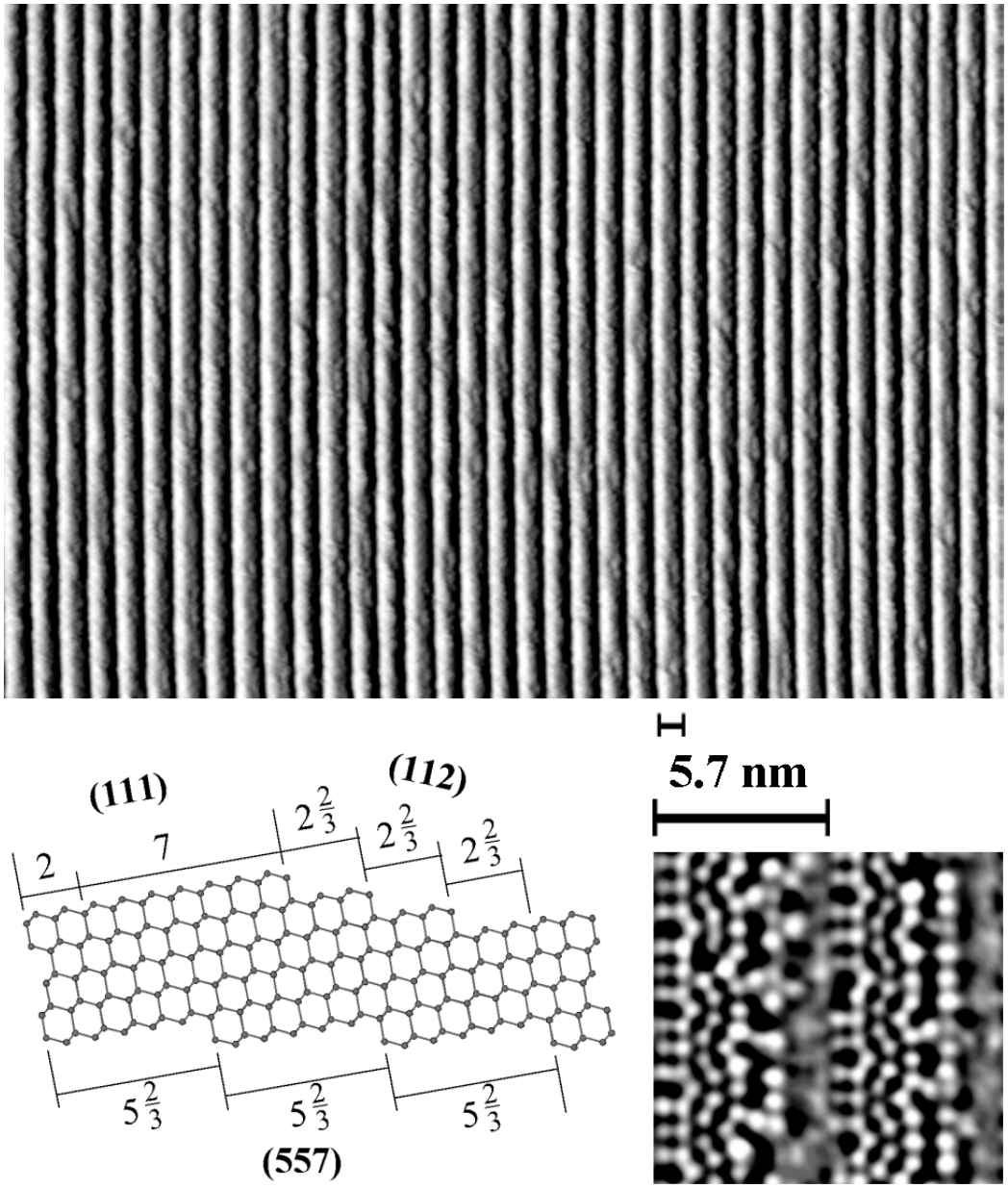


X16A Surface X-ray Diffraction

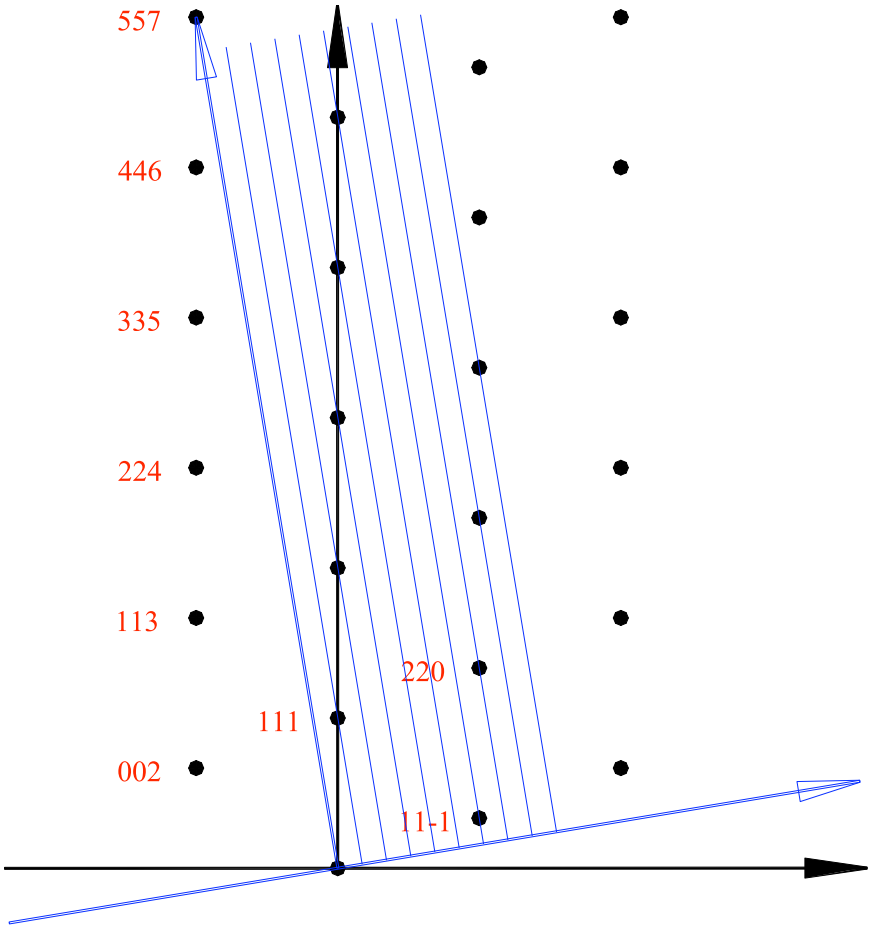
Brookhaven National Lab (1987 – 2004)



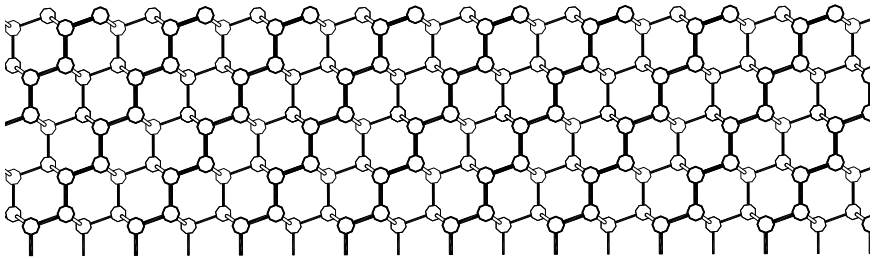


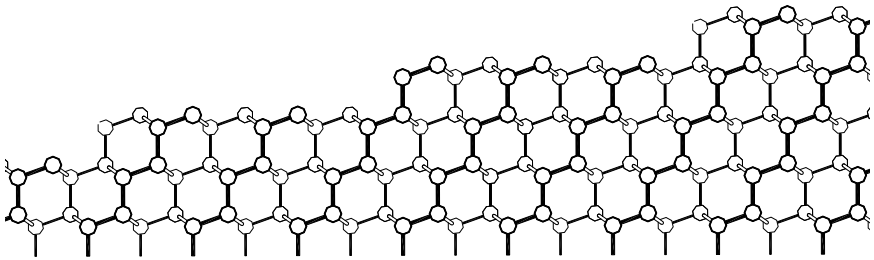

5.7 nm
(111)
(112)
2
7
$2\frac{2}{3}$
$2\frac{2}{3}$
$2\frac{2}{3}$
$5\frac{2}{3}$
$5\frac{2}{3}$
$5\frac{2}{3}$
(557)

Crystallography of Stepped Surfaces



Silicon (111)

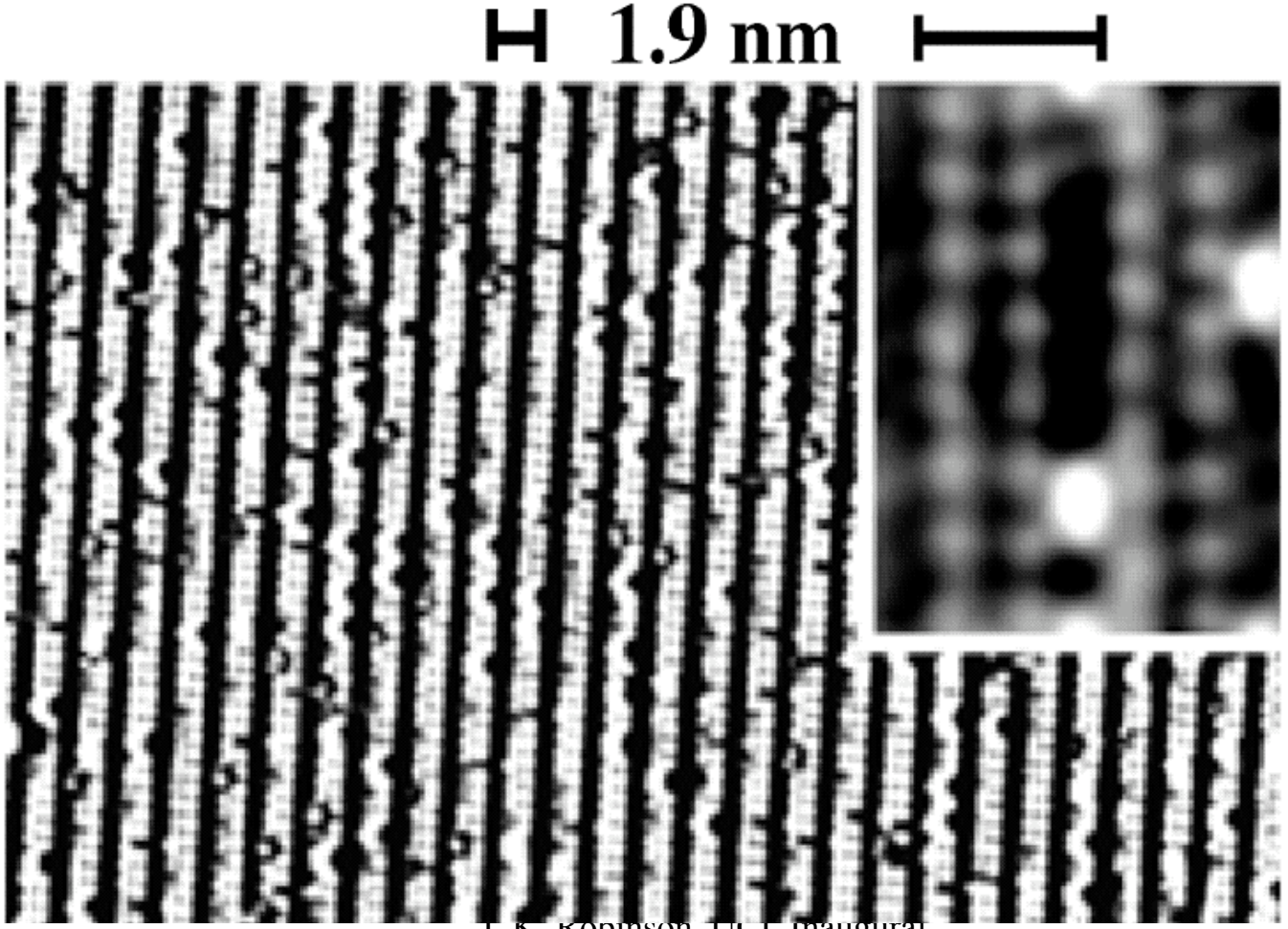


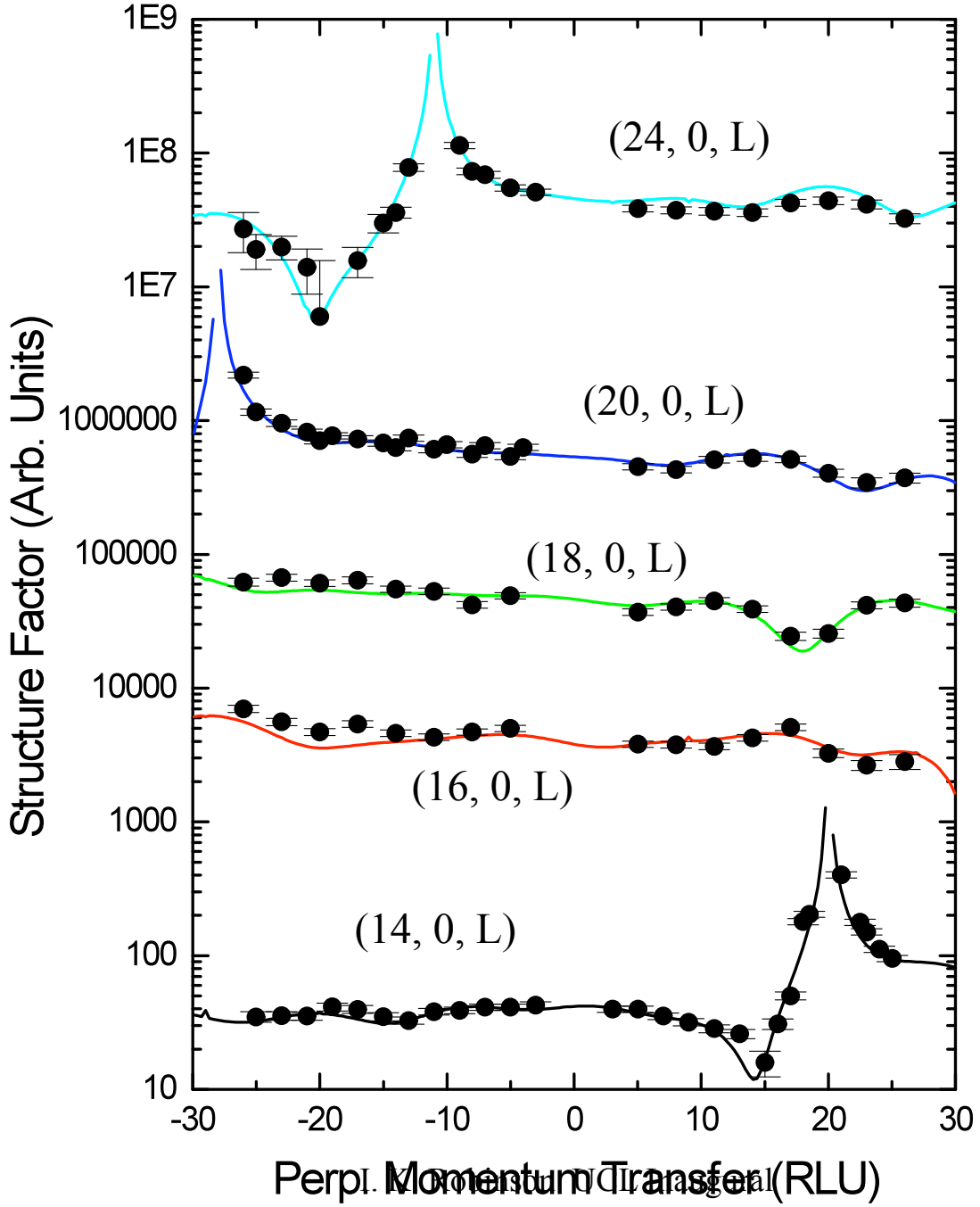


Si(557) surface

Si(557) coated with Au

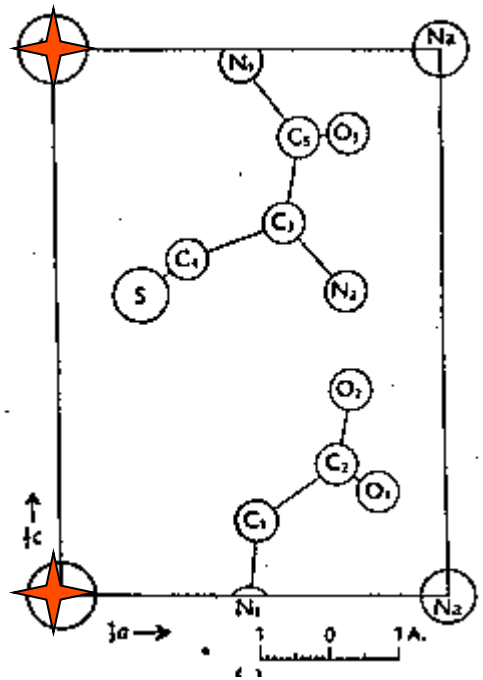
R. Losio, et. al., Phys. Rev. Lett. 86 4632 (2001)



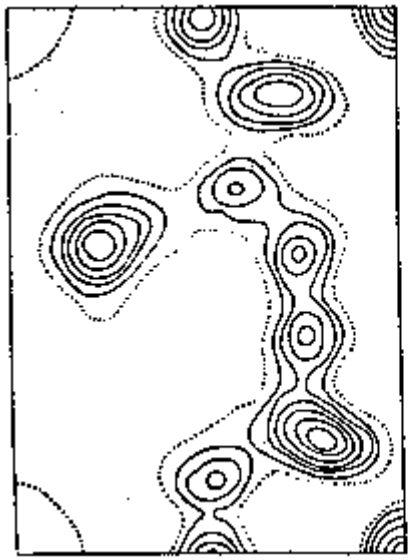

1E9
1E8
1E7
1000000
100000
10000
1000
100
10
Structure Factor (Arb. Units)
(24, 0, L)
(20, 0, L)
(18, 0, L)
(16, 0, L)
(14, 0, L)
-30
-20
-10
0
10
20
30
Perp. Momentum Transfer (RLU)

Phasing by a Single Heavy Atom

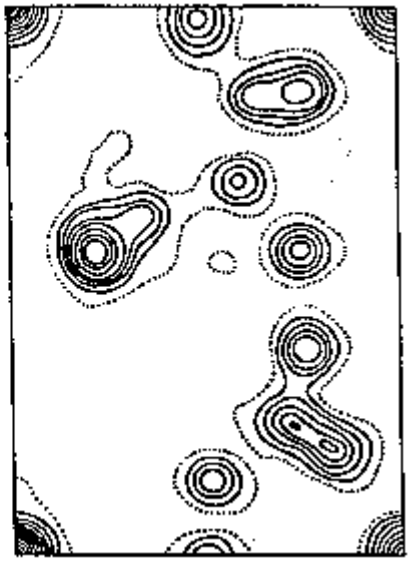
H. B. Dyer, Acta Cryst. 4 42 (1951)



Cysteinylglycine sodium iodide

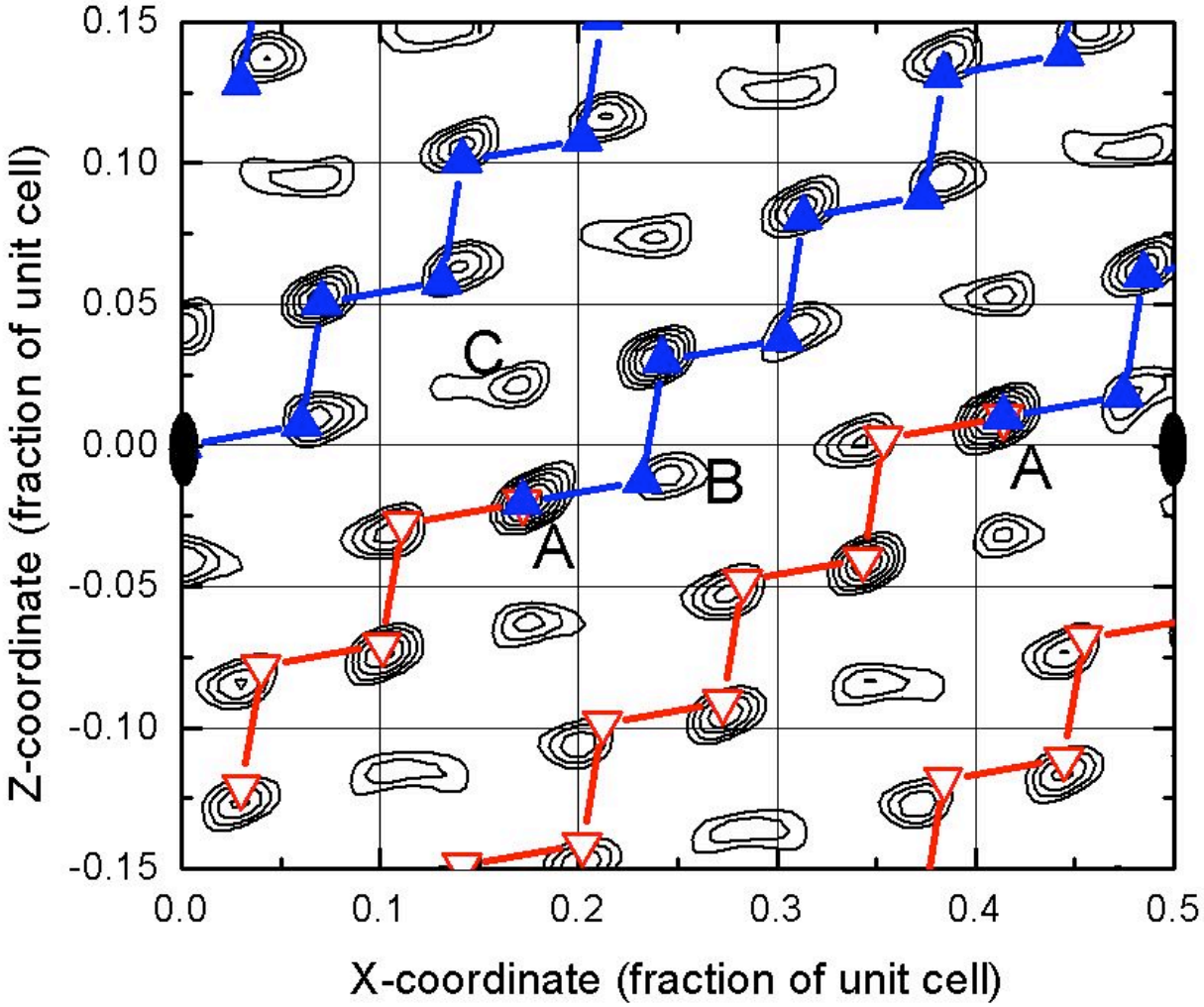


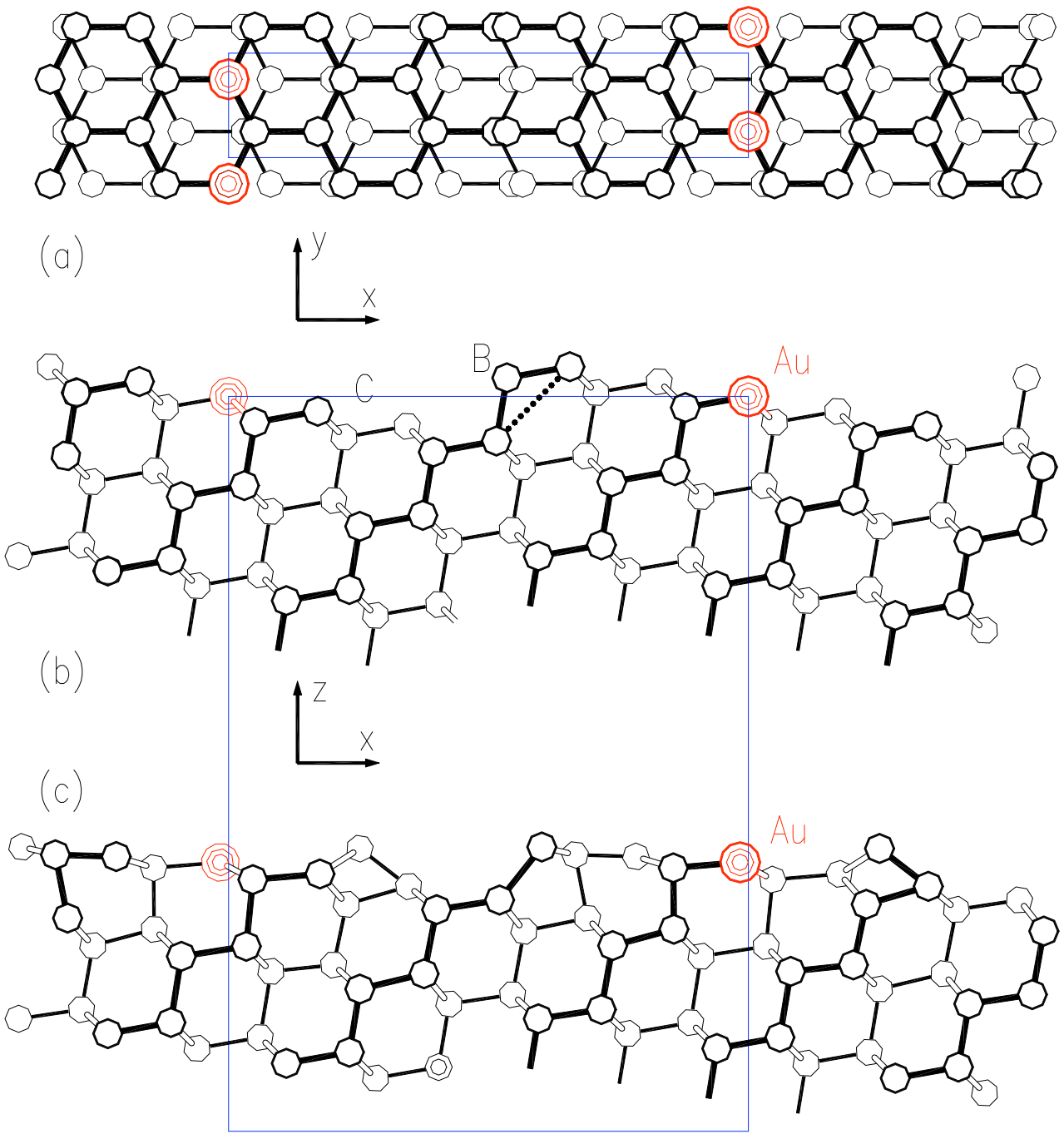
Patterson



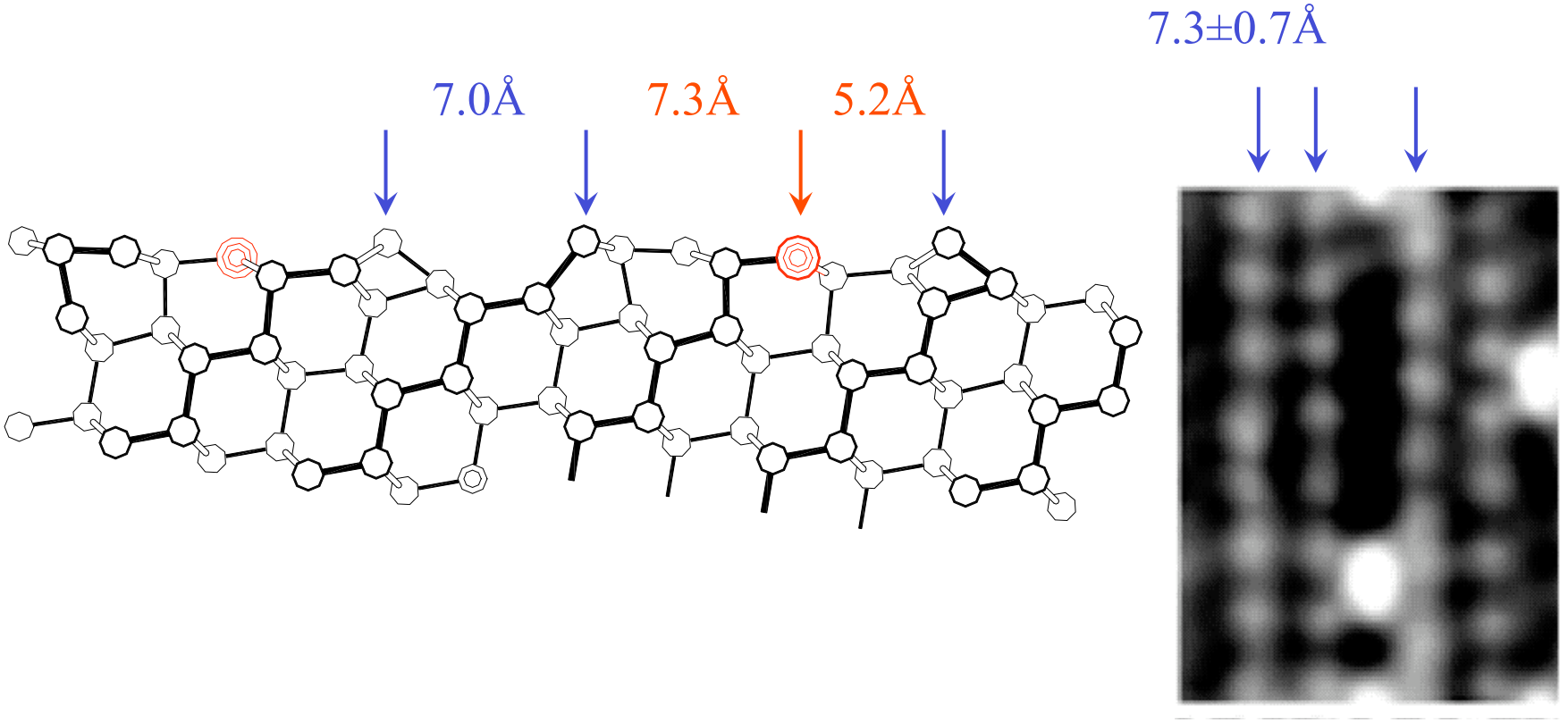
Electron density

X-Z Patterson of Au/Si(557)



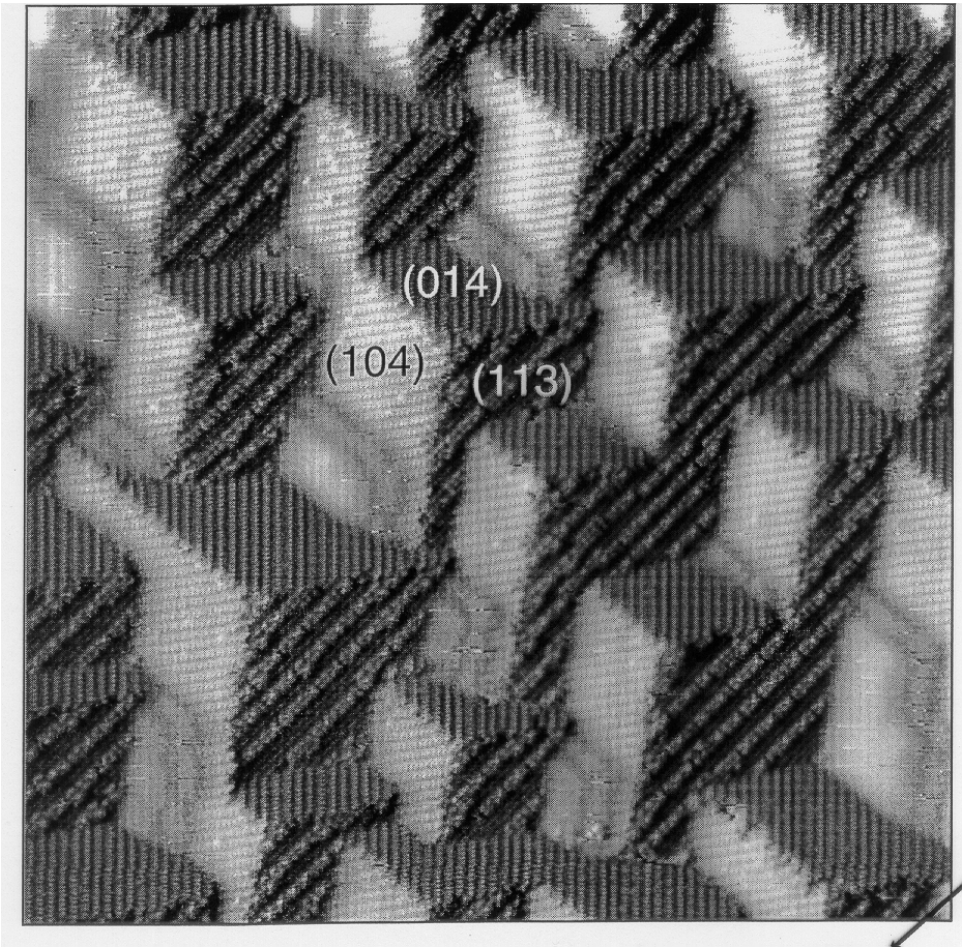

(a)
y
x
B
C
Au
(b)
z
x
(c)
Au

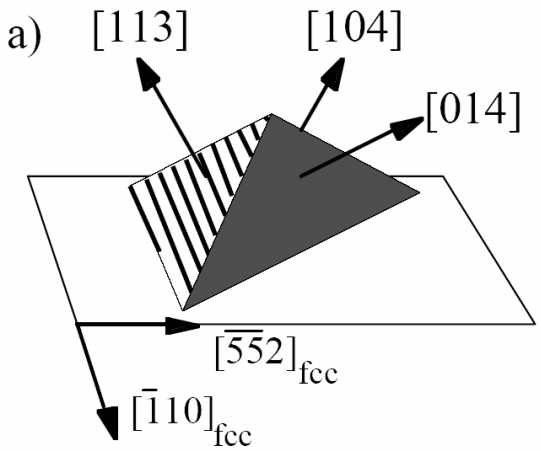
Comparison with STM

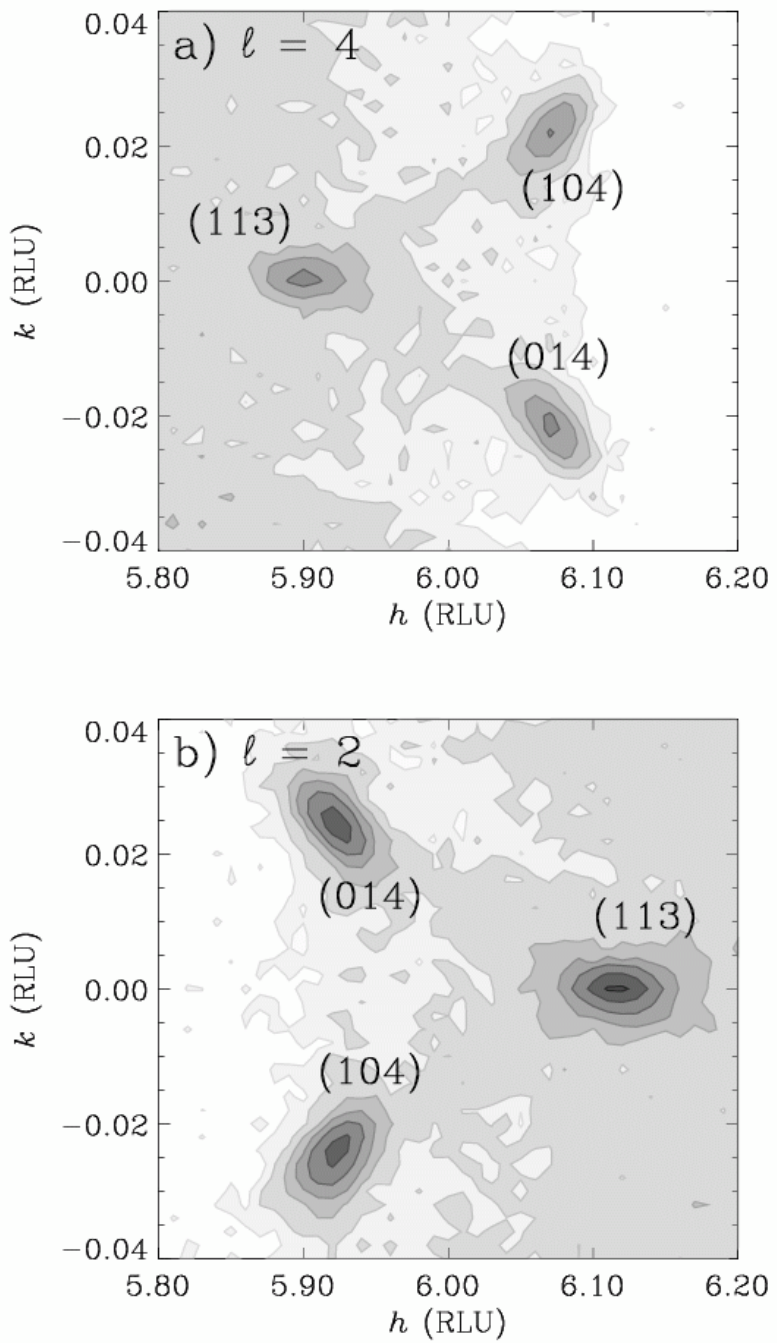


Cu(115) after Oxidation: STM

S. Reiter and E. Taglauer, Surf. Sci. **367** 33 (1996)



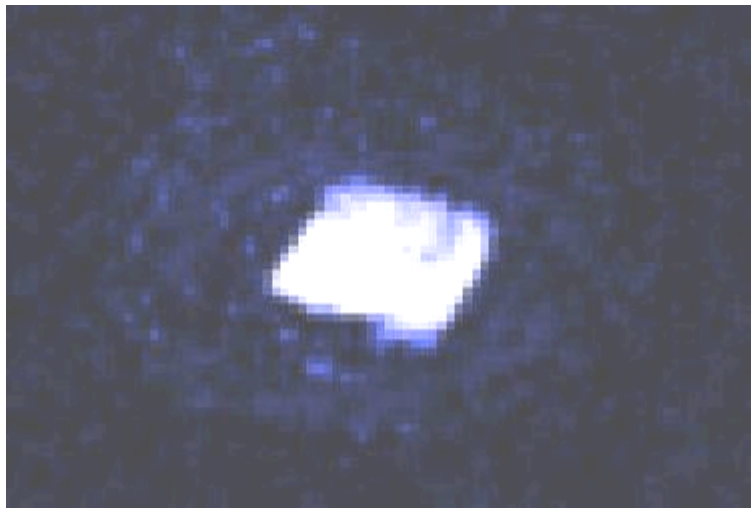




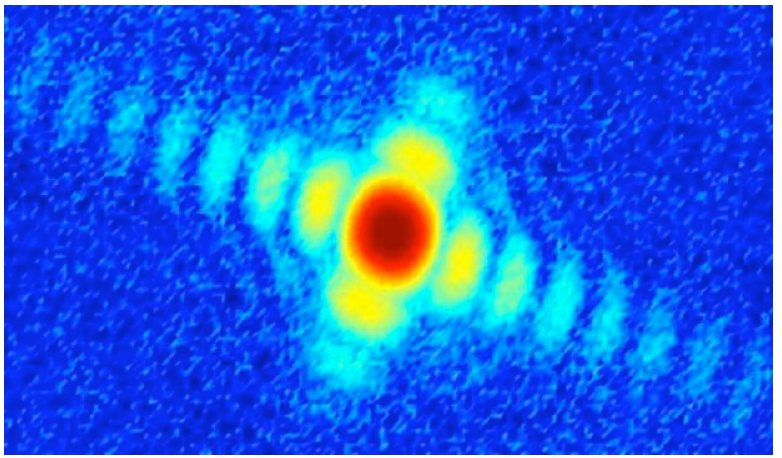
Cu(115) after Oxidation: X16A

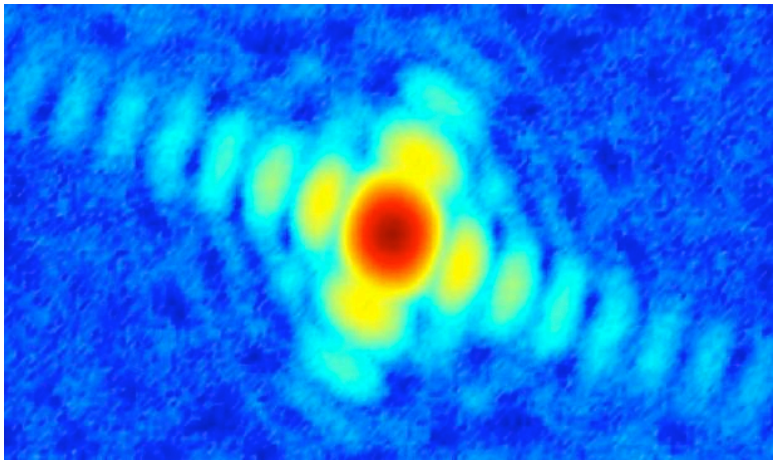
Don Walko, UIUC PhD Dissertation (2000)

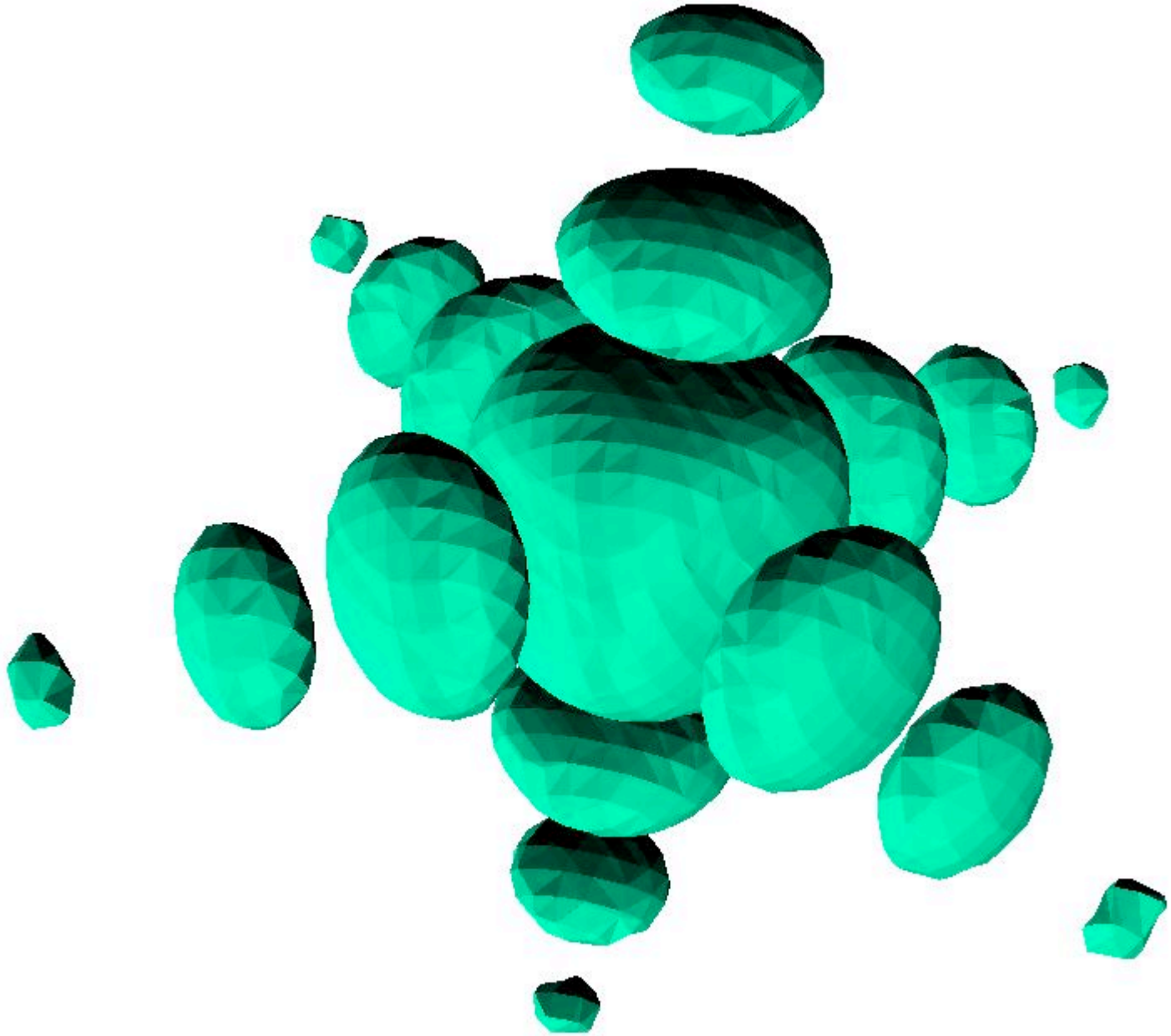
Reconstruction of Ag Nanocrystal

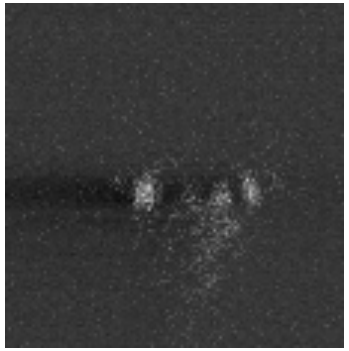


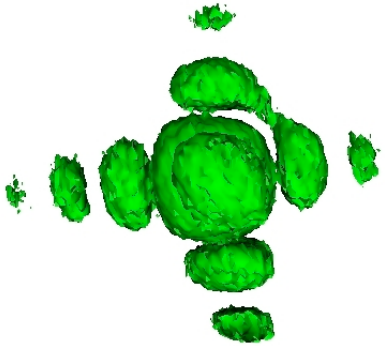
200nm

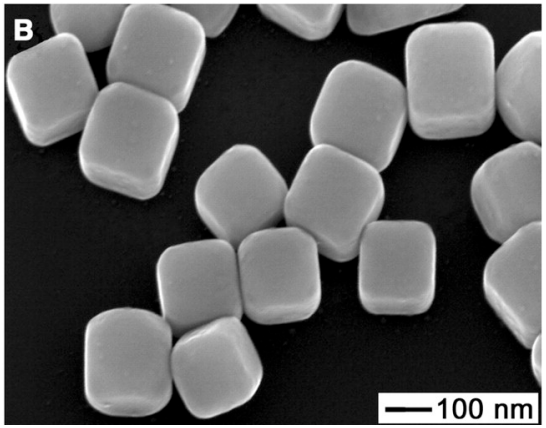


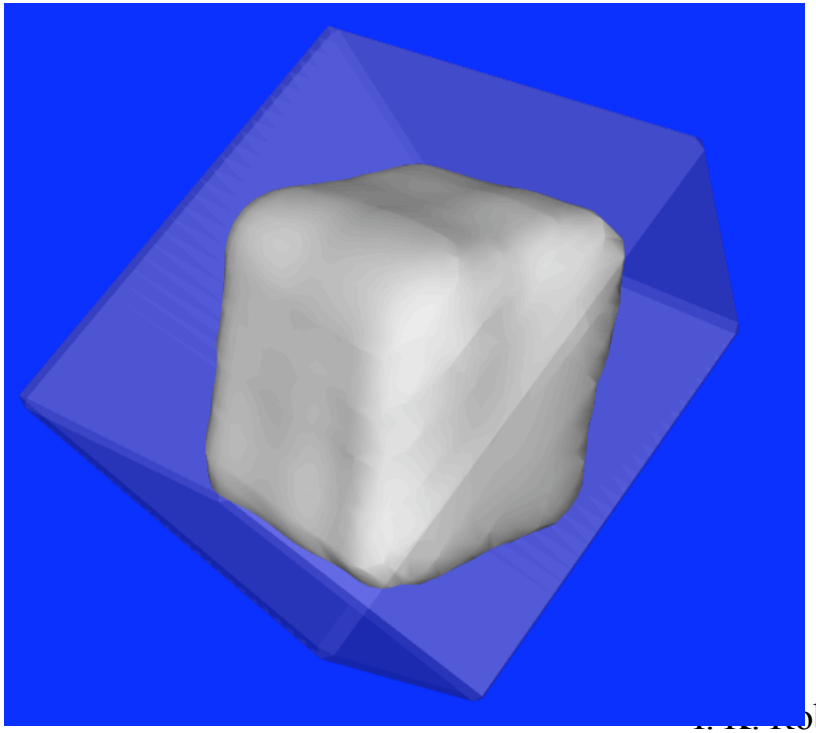


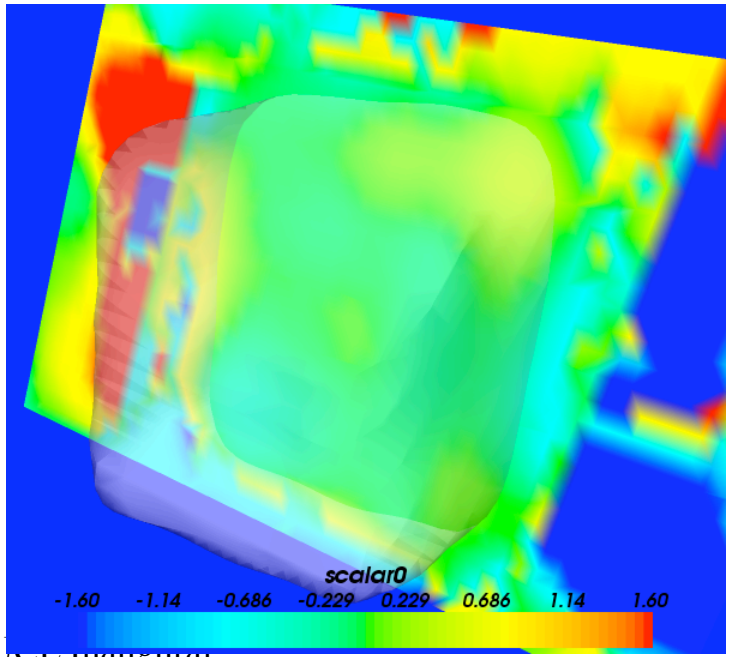


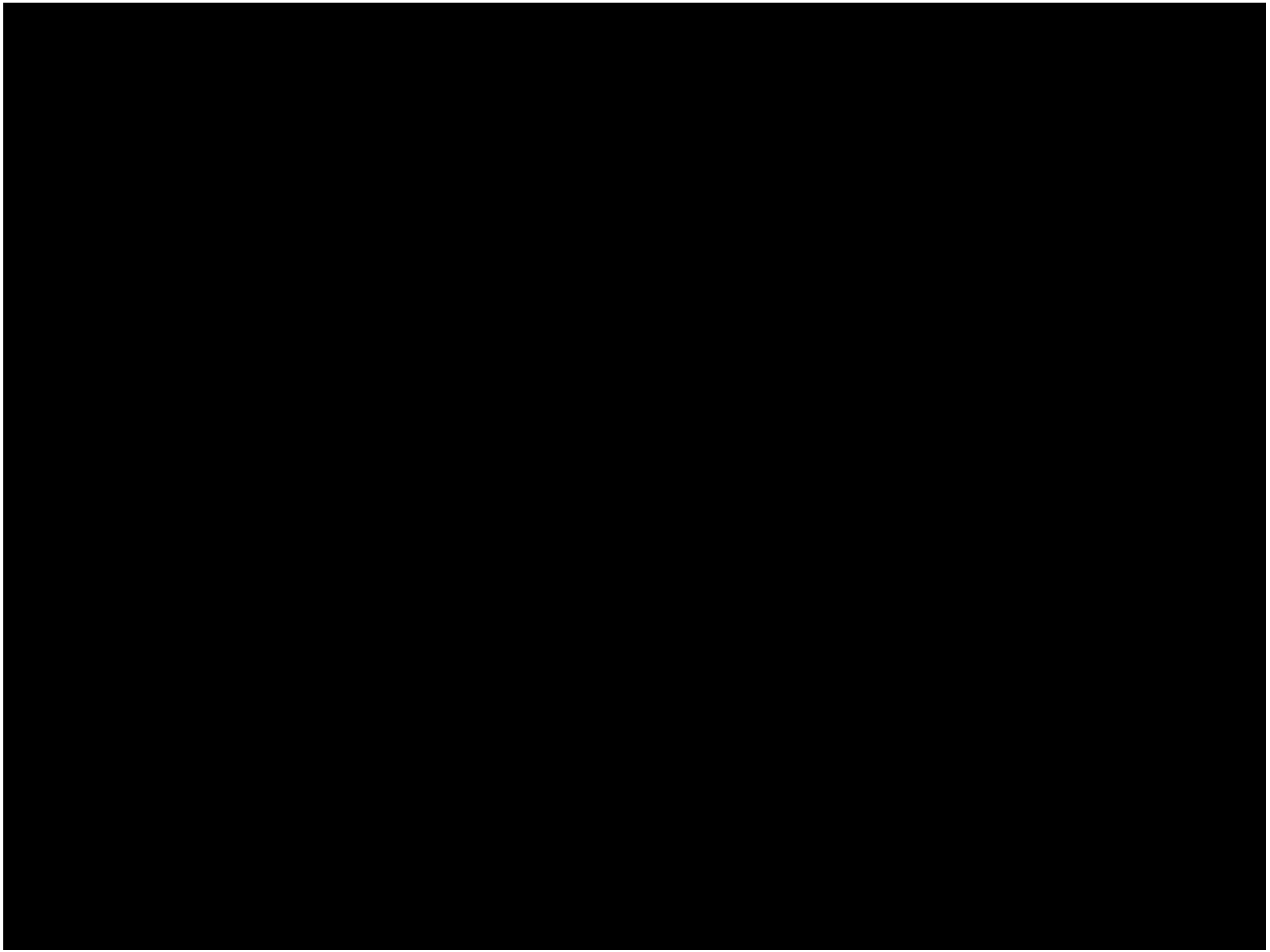





B
100 nm

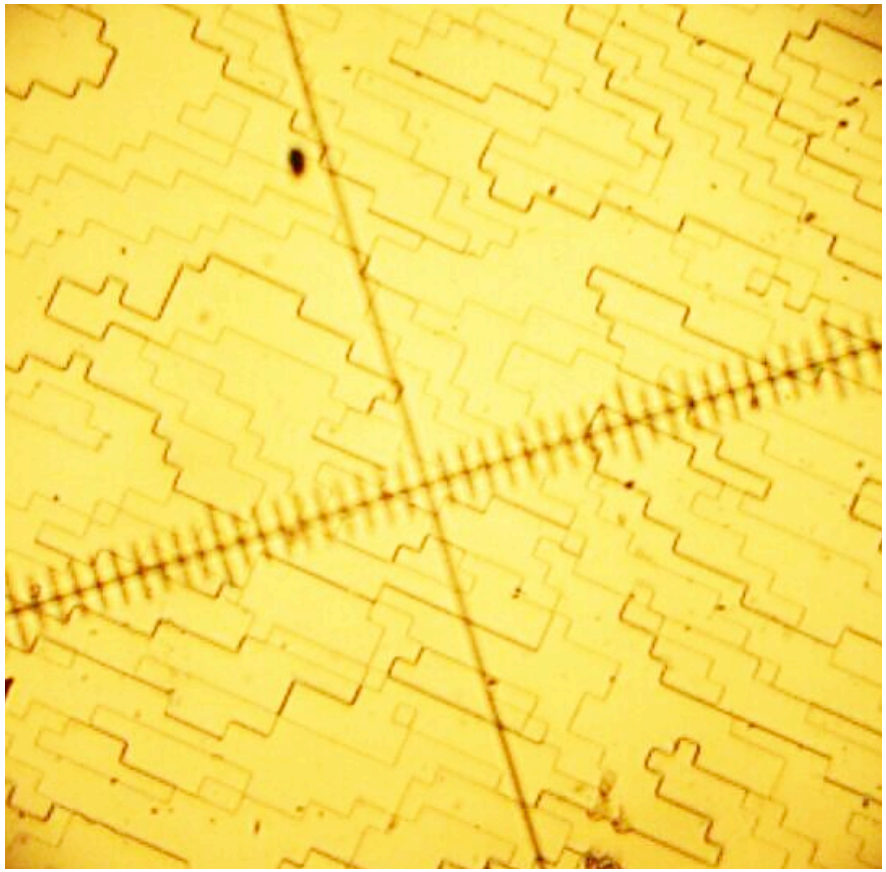


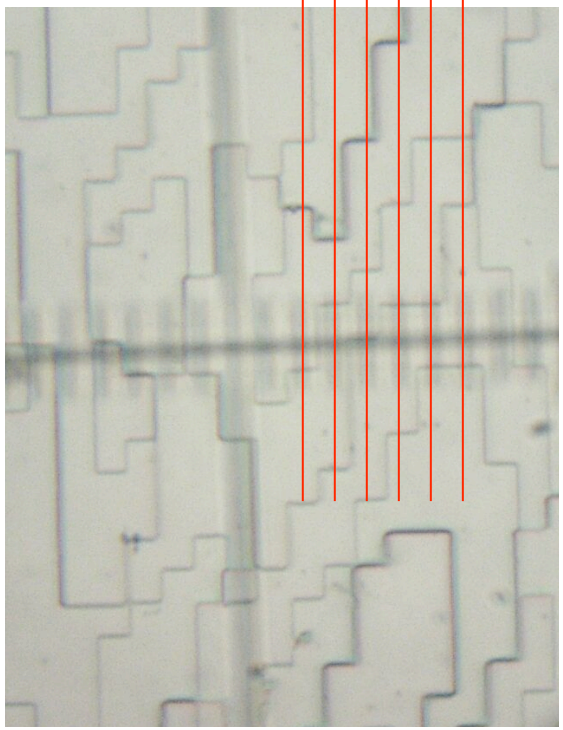

scalar0
-1.60 -1.14 -0.686 -0.229 0.229 0.686 1.14 1.60

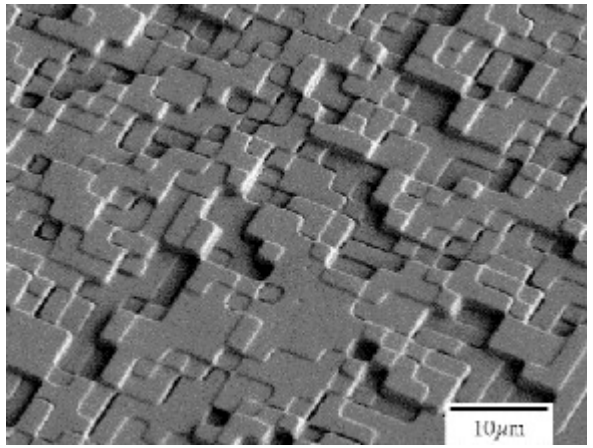


Microscope Images of Gratings

Digital Optics Corp: phase grating

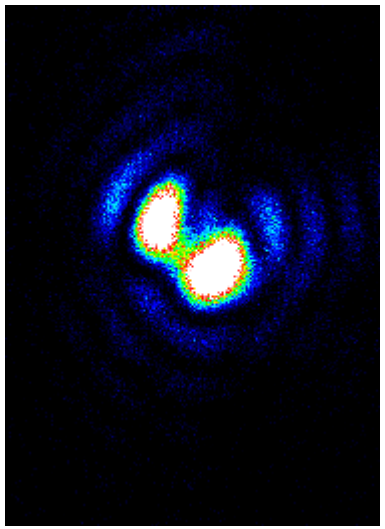


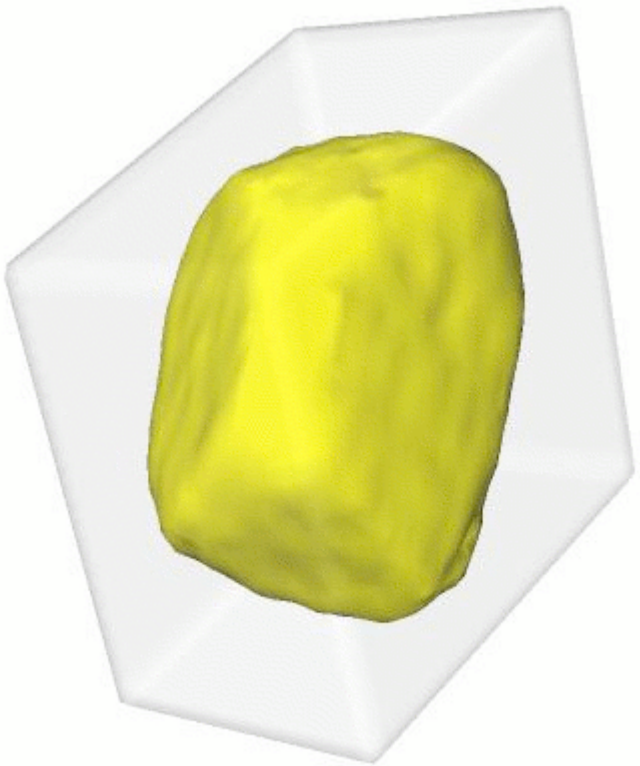




Gold nanocrystal reconstruction

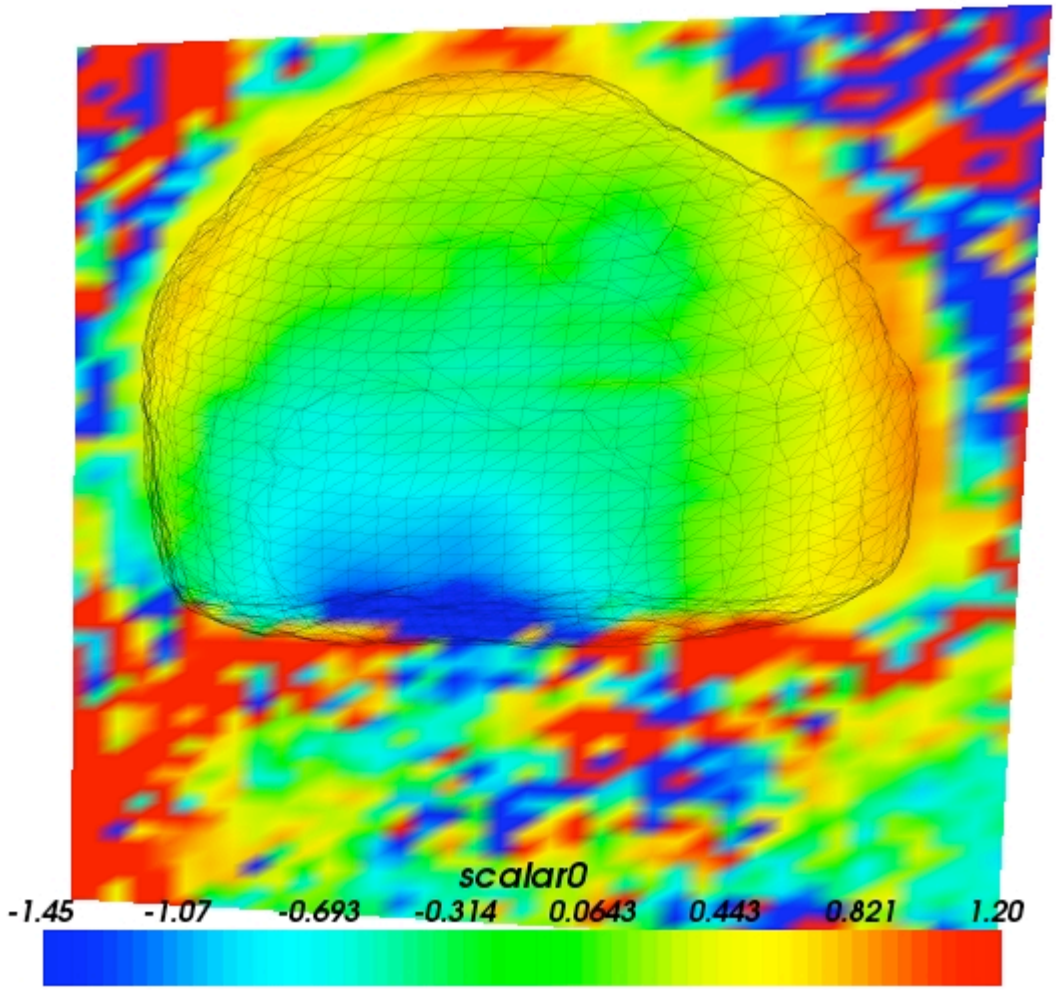
showing support used for 20 HIO followed by 10 ER





Phase Maps of strain in Pb nanocrystal

including correction for refraction by crystal



Advanced Photon Source (ANL)

Urbana

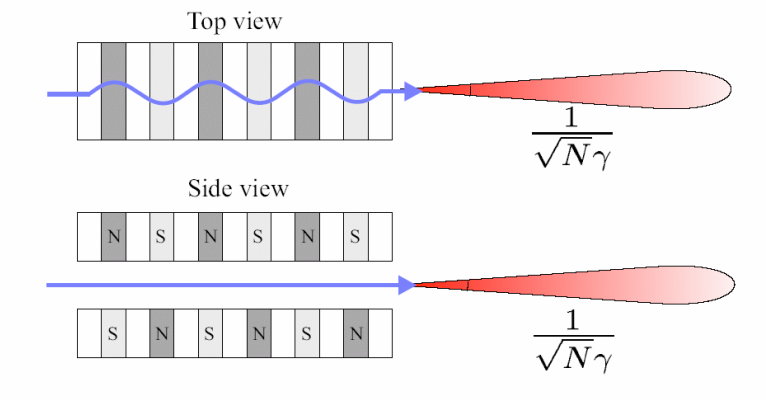
34-ID-C

Diamond Light Source (RAL)

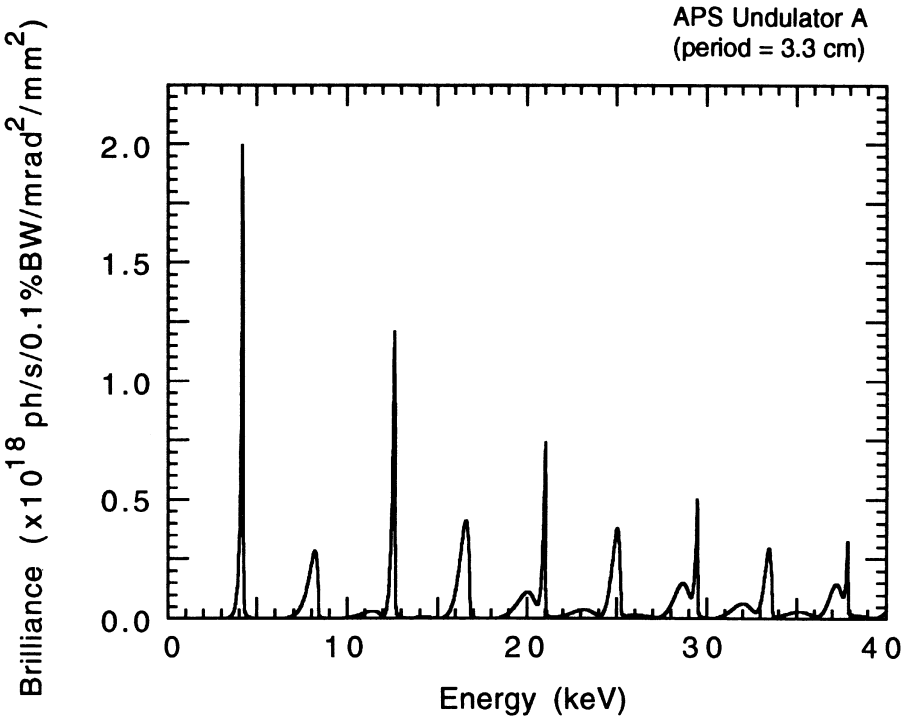
Diamond in-vacuum X-ray Undulator

X-ray Undulator Principle

Vitalii Ginzburg, Novosibirsk (1947)

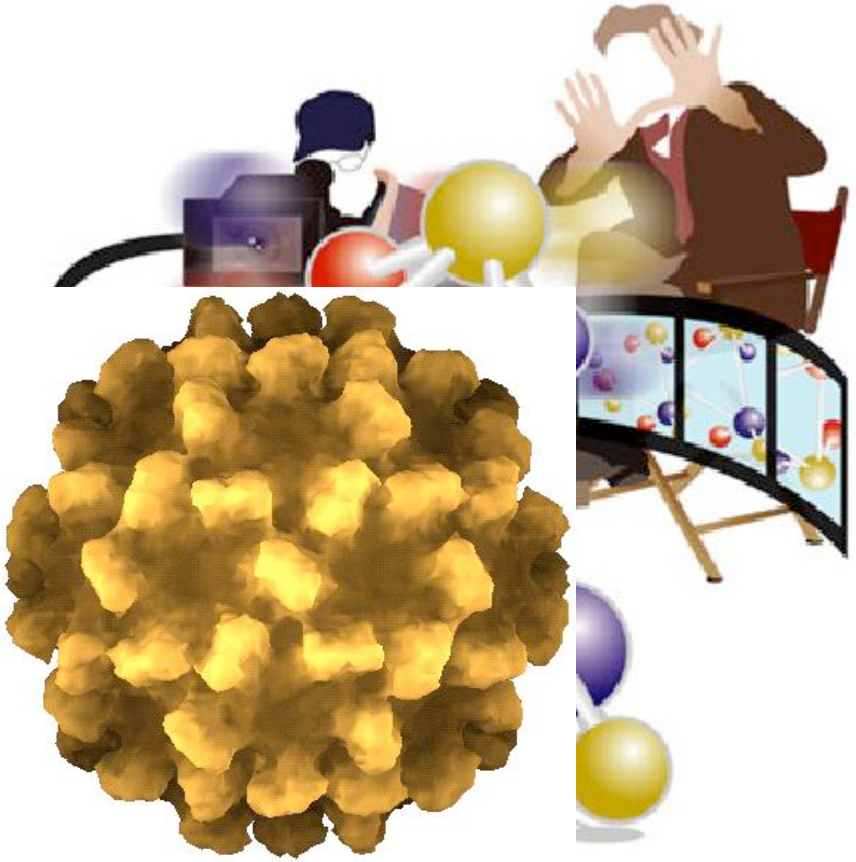


$$\lambda_X = \frac{\lambda_U}{2\gamma^2}\left\{1 + \frac{K^2}{2} + (\gamma\theta)^2\right\}$$



Diamond beamlines I-22 and I-16

Molecular Movies using XFEL



New routes to biological structure?

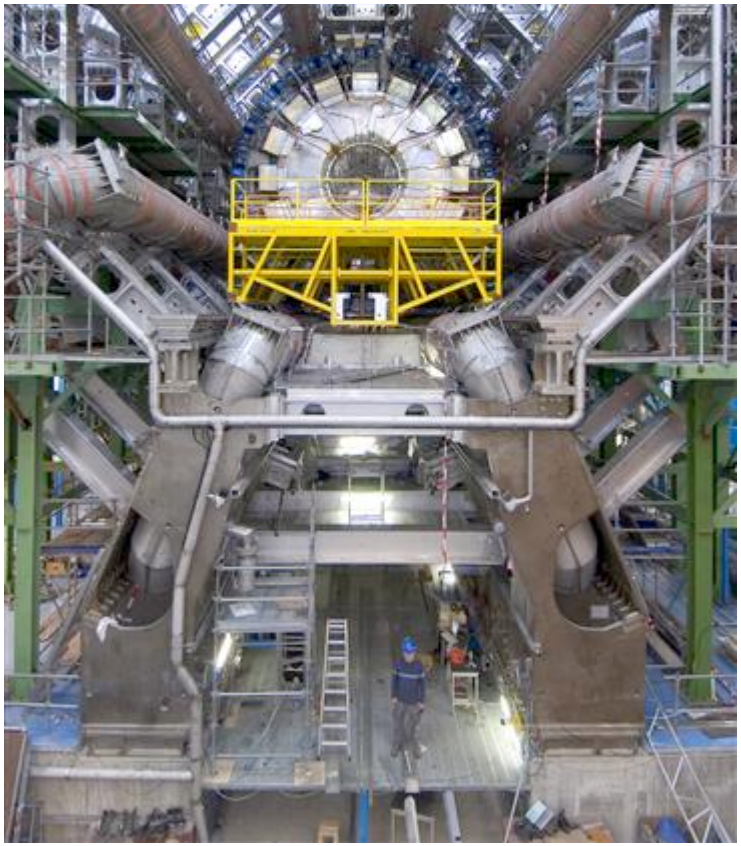
- Cryo-EM
  - real-space image, but not dose-efficient
- Inversion of diffraction
  - dose-efficient, especially for phase contrast
  - about equally efficient for photons or electrons
- How to beat the dose limit?

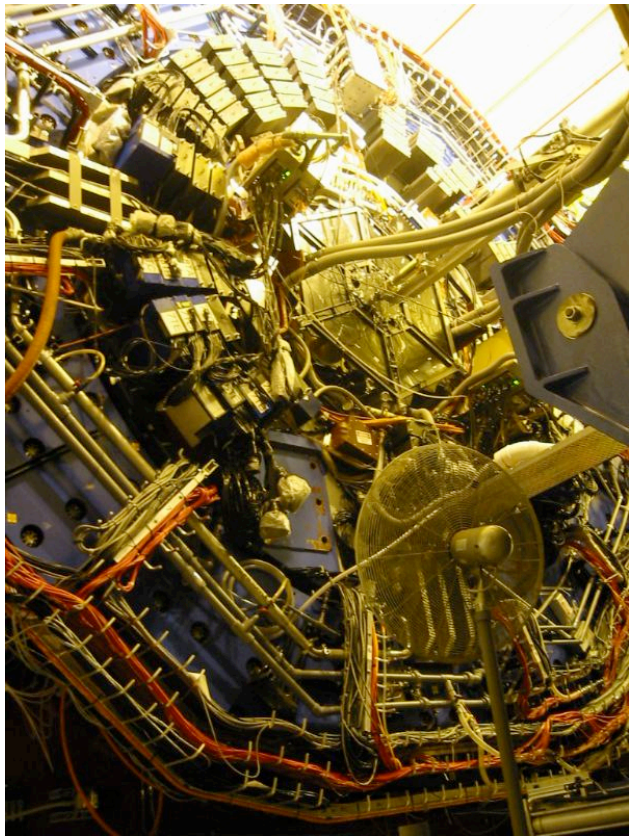
Do it faster!

How fast must we go?

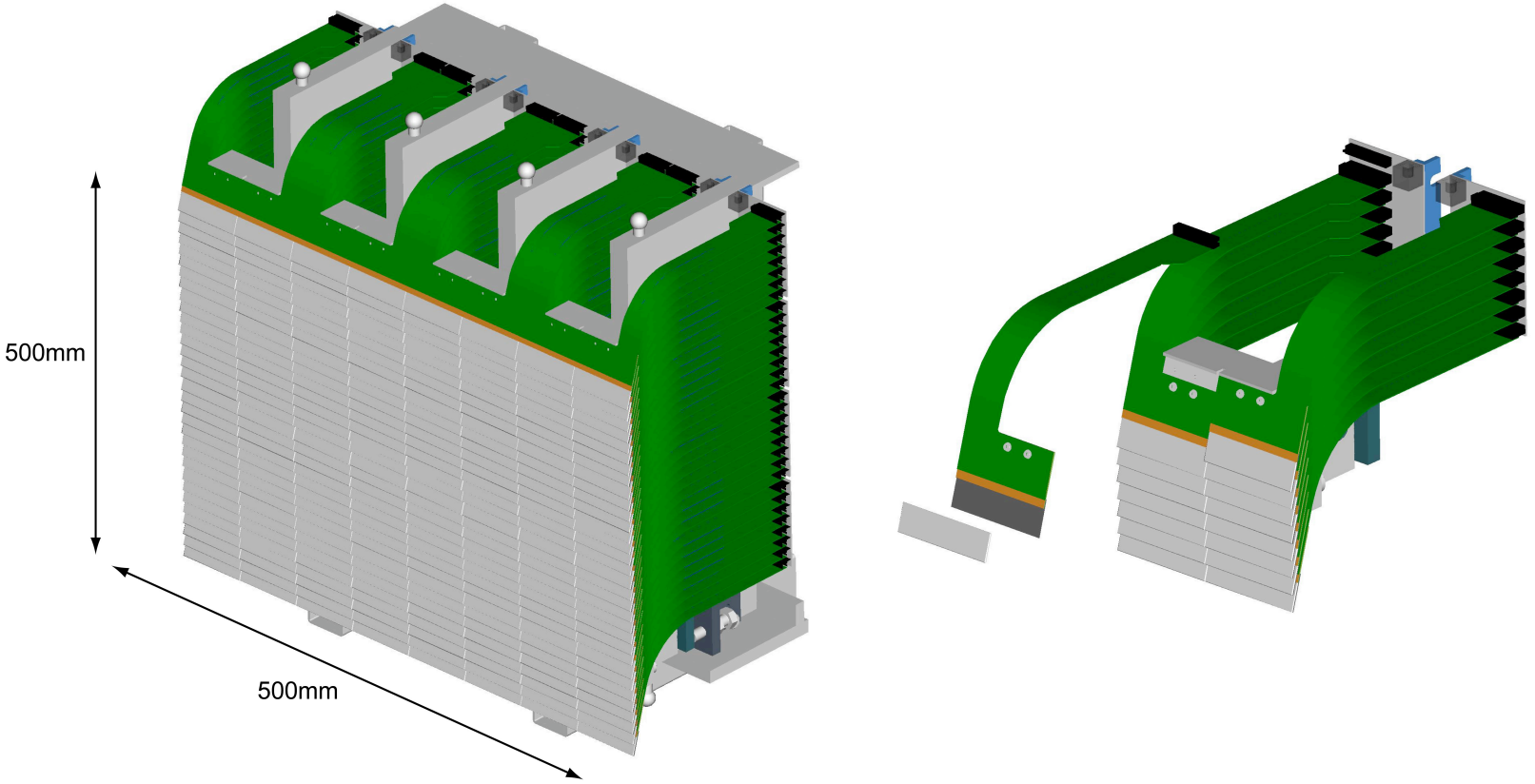
- Radiation breaks bonds and ionizes matter
- Inertial response of nucleus to broken bond
- Structure is ‘lost’ when an atom moves by one bond-length
- Phonon vibration time: 10^{14} Hz = 10fs
- Photons better than electrons (space charge)
- Possible with X-ray free-electron laser (XFEL)

The HEP approach: detectors drive the science

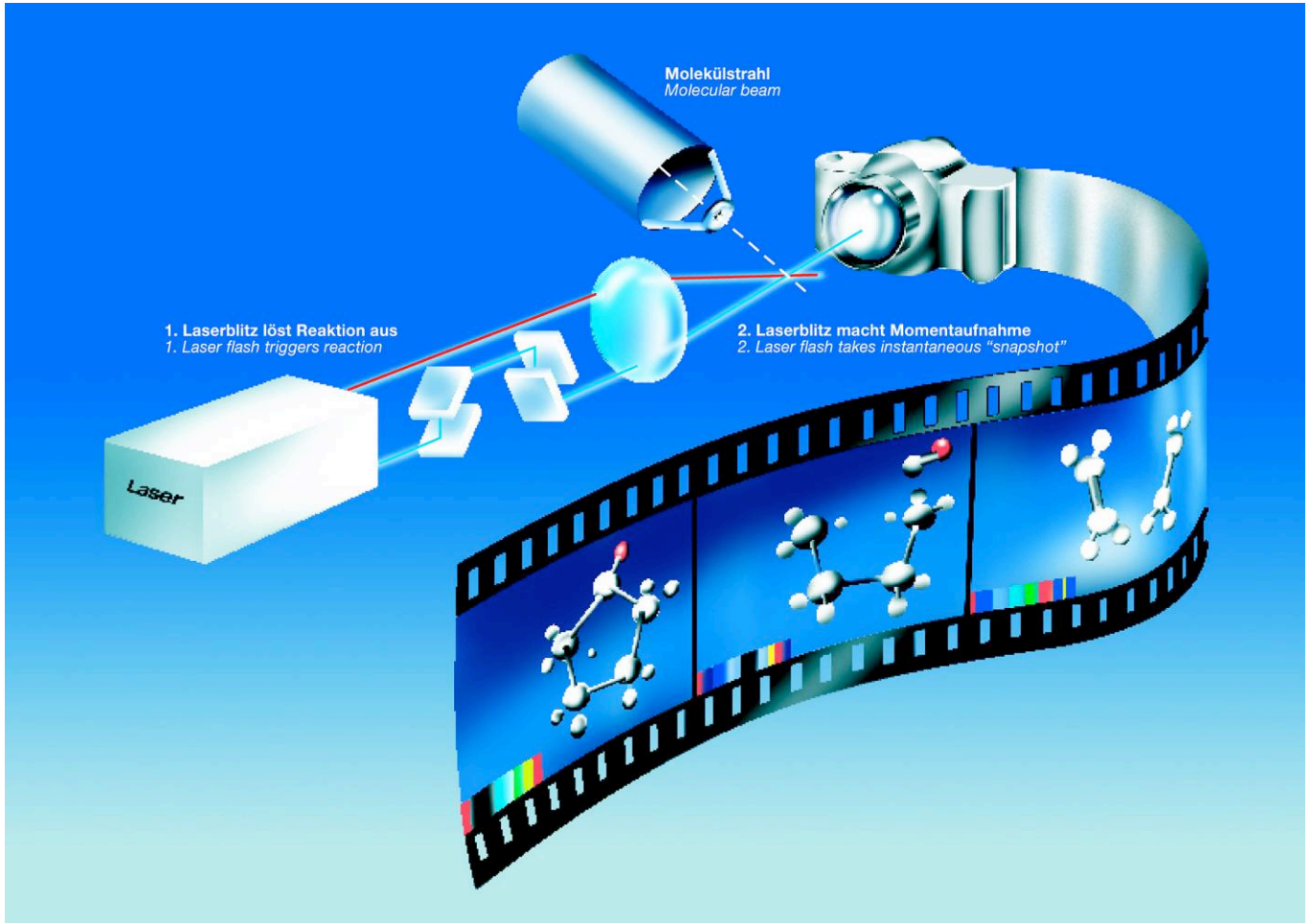




UK “Large Pixel” Detector Consortium (Marcus French, RAL)



Molecular Movies using XFEL

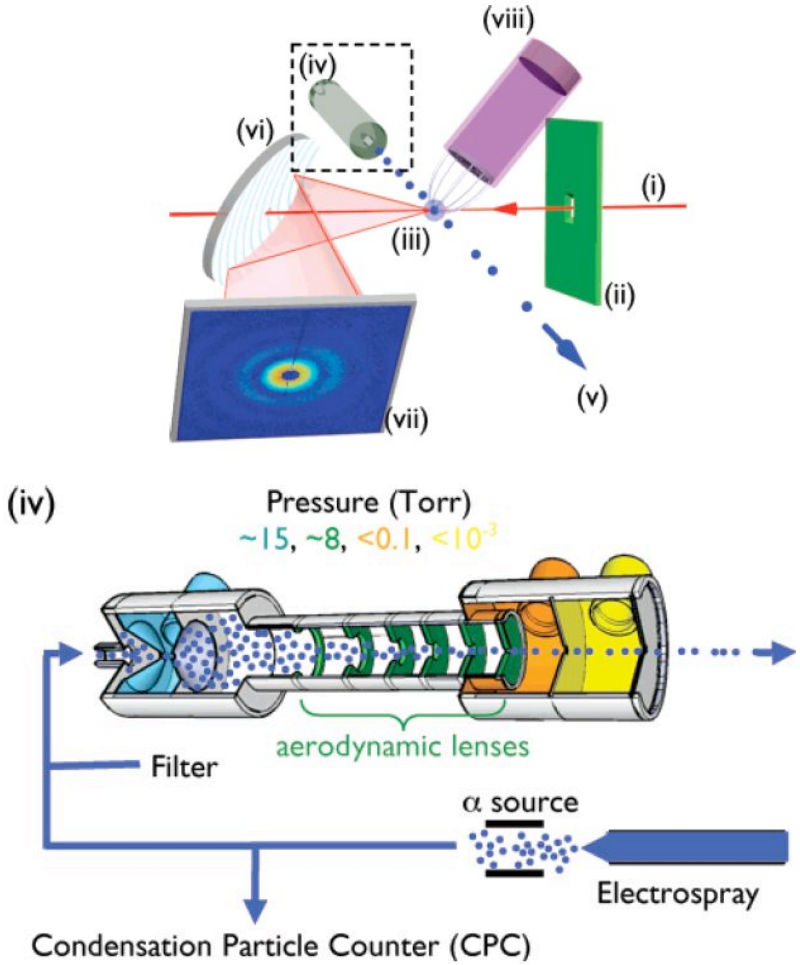


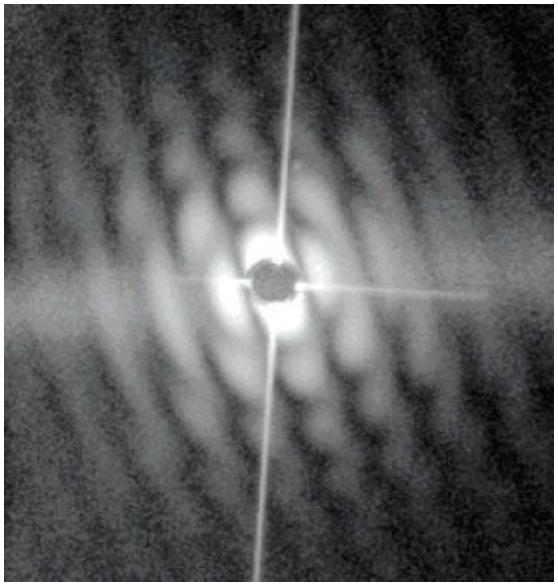
I. K. Robinson UCL Inaugural

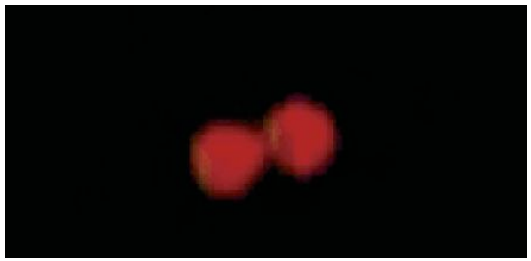
Single Particle X-ray Diffractive Imaging

M. J. Bogan et al Nano Letters 8 310 (2008)

sucrose encapsulated DNA, 248 nm diameter







Surface X-ray Diffraction pioneers

Paul Fuoss	Argonne
Peter Bennett	Arizona
Doon Gibbs	Brookhaven
Ben Ocko	Brookhaven
Peter Eng	Chicago
Robert Feidenhans'l	Copenhagen
Jens Als-Nielsen	Copenhagen
Mike Altman	HKUST
Elias Vlieg	Nijmegen
Sunny Sinha	San Diego
Don Walko	Urbana
Franz Himpsel	Wisconsin

Summary

- Au quantum wires on Stepped Si
- Facetting of Cu(115) seen in CTRs
- Nanocrystal: paradigm of Surface/Bulk
- Future: snapshots of condensed matter

Future of Surface Diffraction

- Nanocrystal Structure CXD
- Buried interfaces, such as solid-liquid CTR
- Quantum dots and wires CXD
- Fluctuating surfaces, capillary waves XPCS
- Nanostructured rough surfaces GISAXS, XPCS
- Continuum models of strain and defects CXD
- Automated techniques CCD