

Coherent Diffraction Methods in Corrosion Science

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London Centre for Nanotechnology
& Diamond Light Source

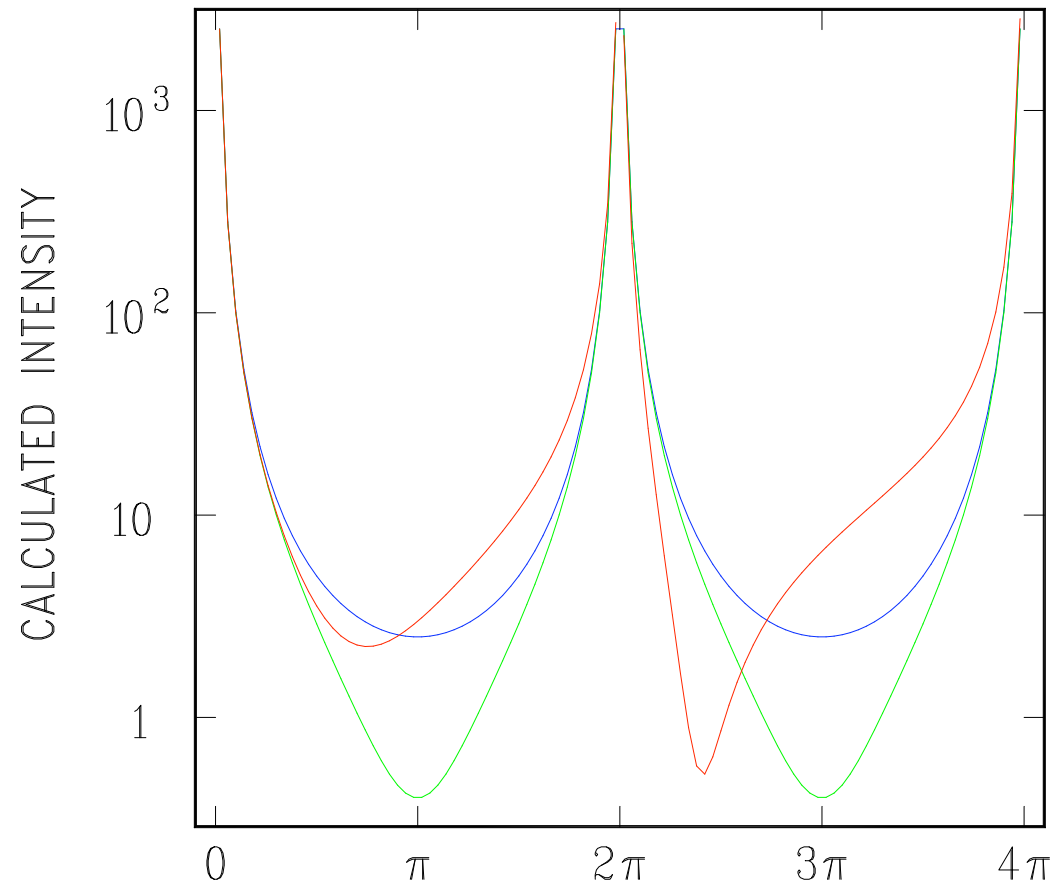
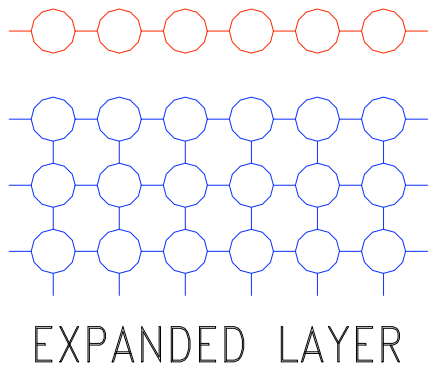
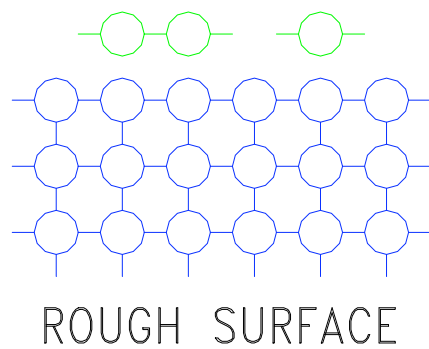
Corrosion Chemistry in Pits, Crevices and Cracks

Mansfield College, Oxford, 20th July 2009

Techniques available at Diamond

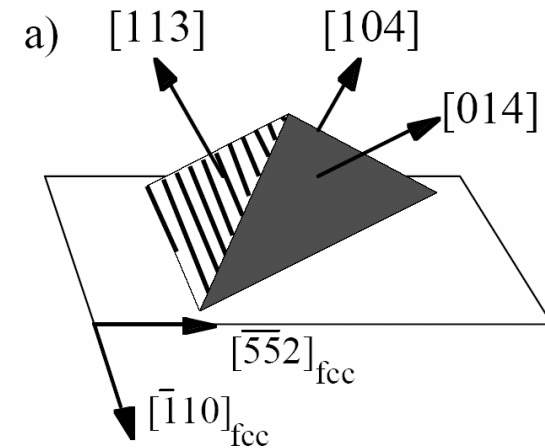
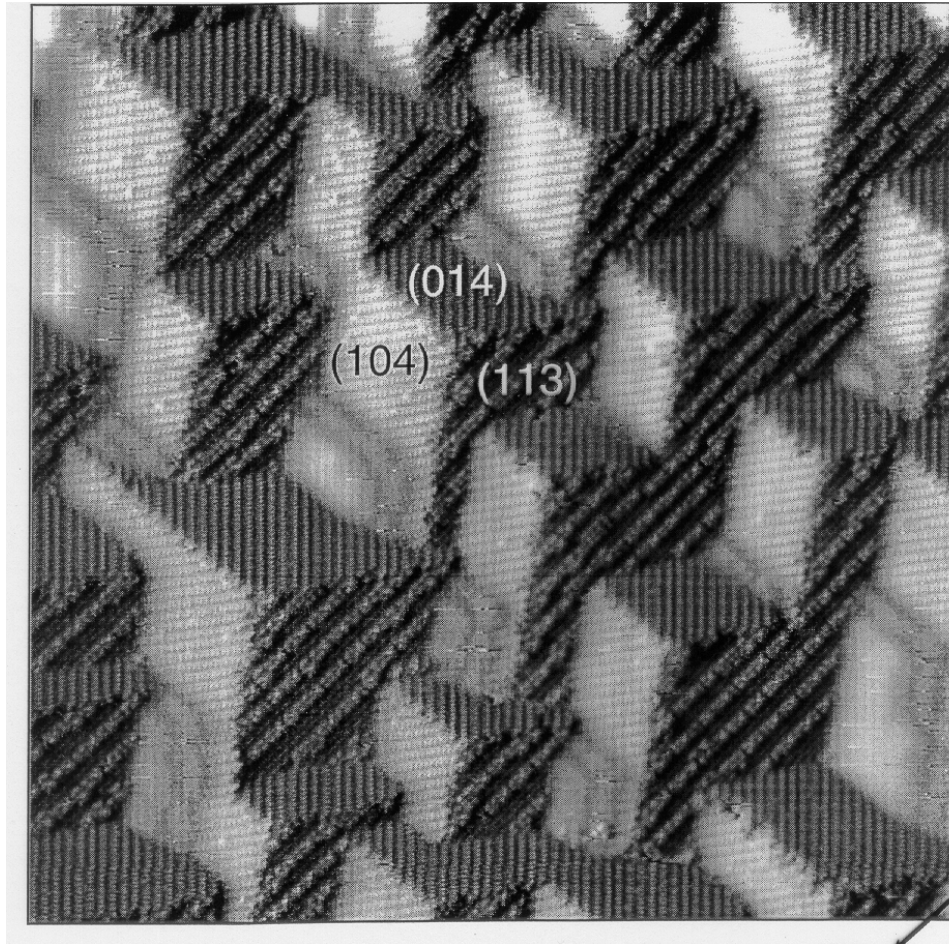
- Surface X-ray diffraction, CTR
 - in-situ electrochemical cells
 - I-07 starting Oct 2009
 - Chris Nicklin, PBS
- Transmission X-ray Microscopy
 - nanoscale tomography for 3D images
 - Karen Chen & David Dunand, NU
- Coherent X-ray Diffraction and Imaging
 - I-13 starting Nov 2011
 - Christoph Rau, PBS

CTR is Sensitive to Surface Structure



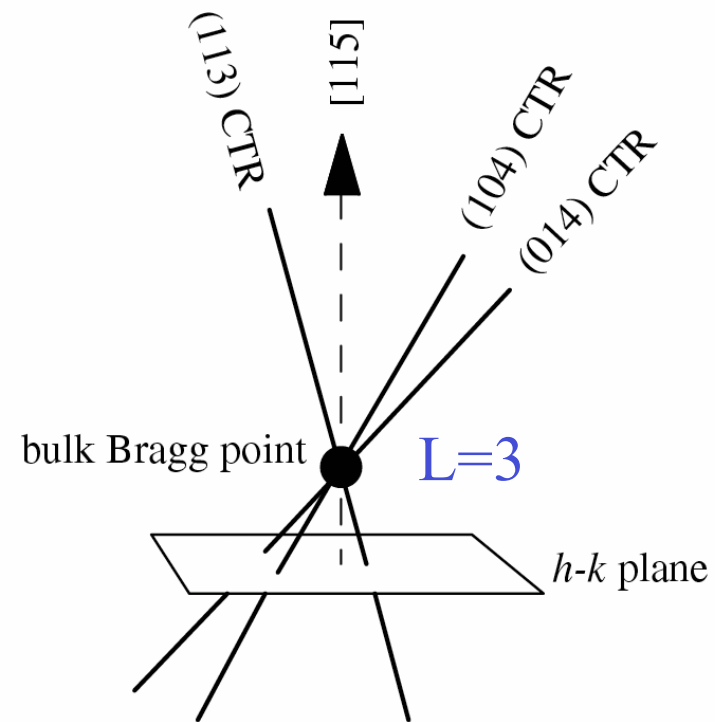
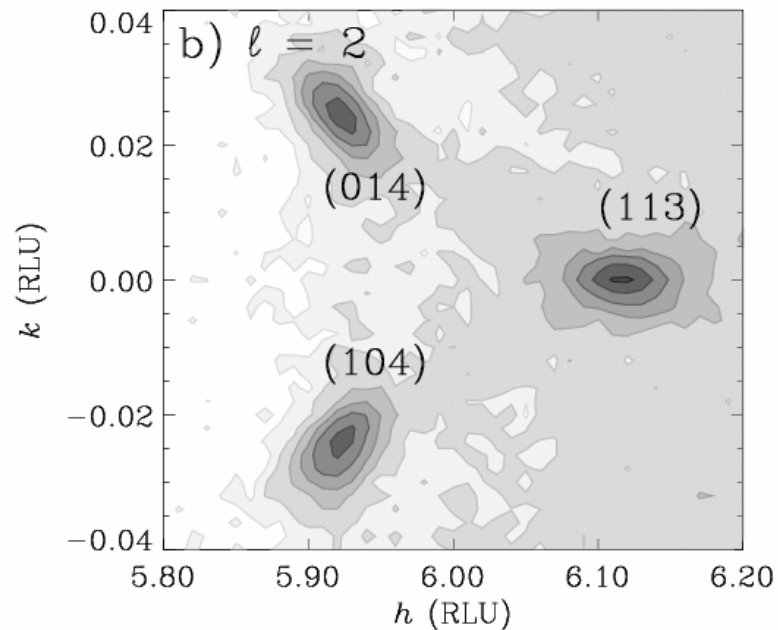
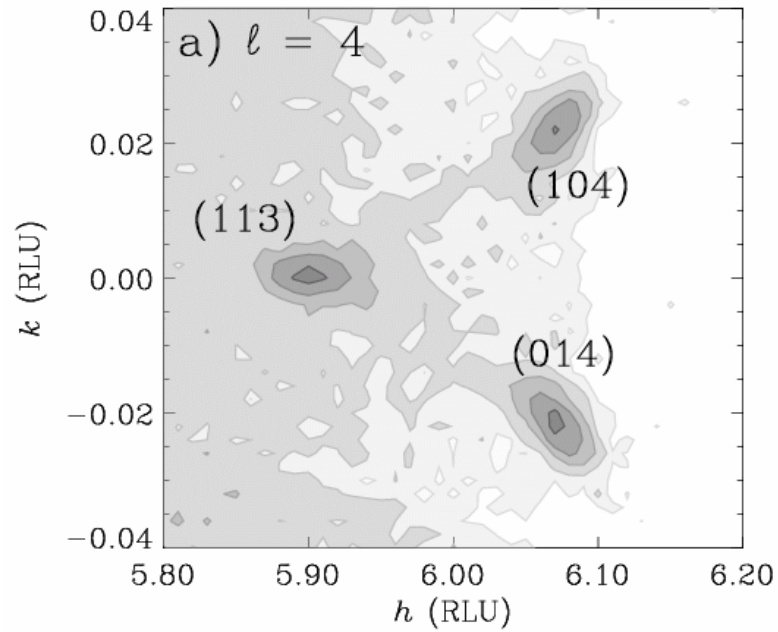
Cu(115) after Oxidation: STM

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

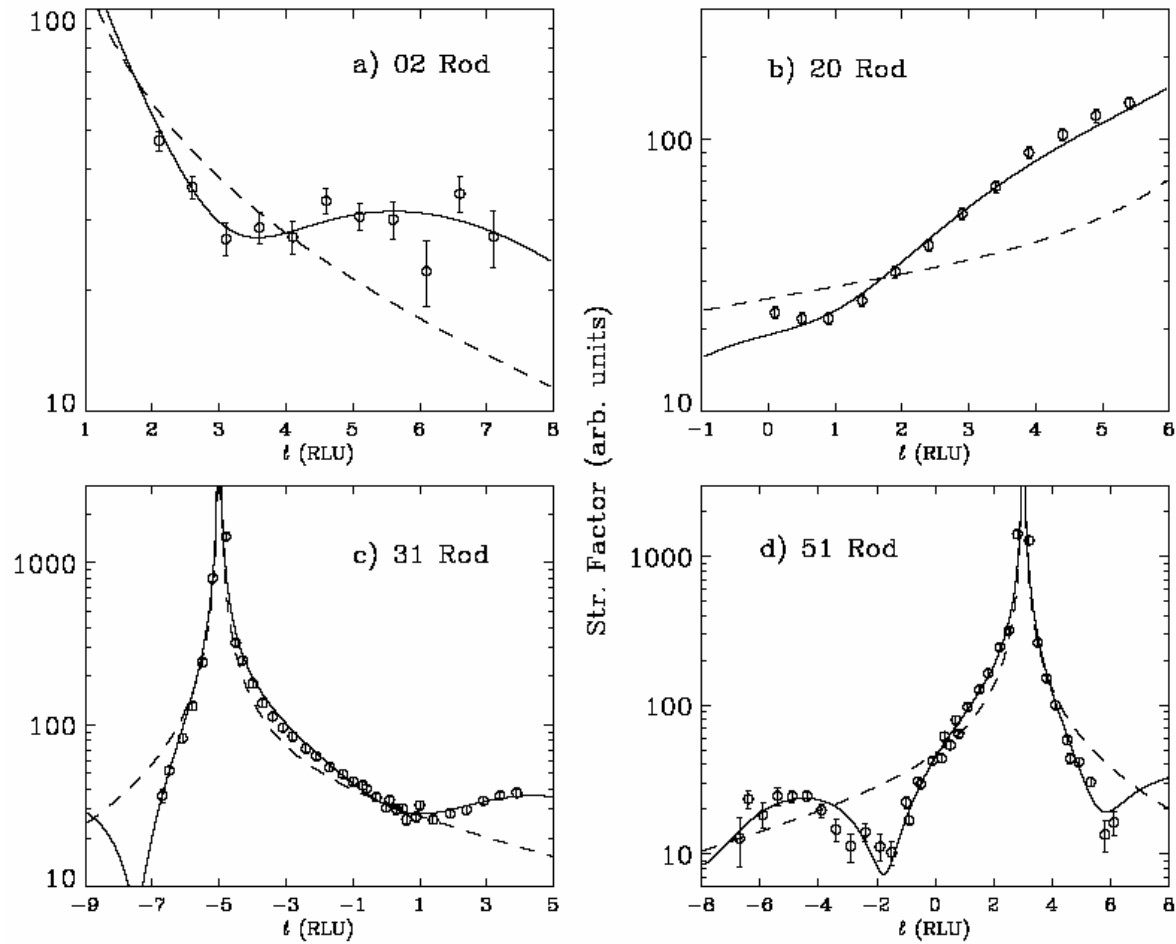


Cu(115) after Oxidation: NSLS

Don Walko, UIUC PhD
Dissertation (2000)



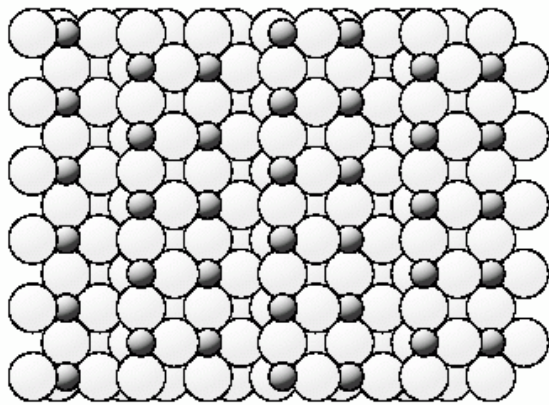
Re-index CTRs for Cu(104) facets



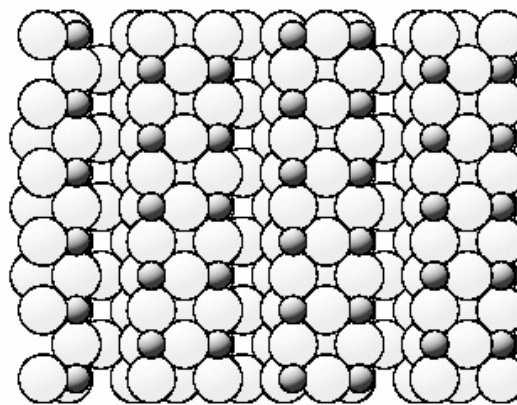
Surface Structures of Cu(104)-O

E. Vlieg, S. M. Driver, P. Goettkindt, P. J. Knight, W. Liu, J. Luedecke, K. A. Mitchell, V. Murashov, I. K. Robinson, S. A. de Vries and D. P. Woodruff, *Surface Science* **516** 16-32 (2002)

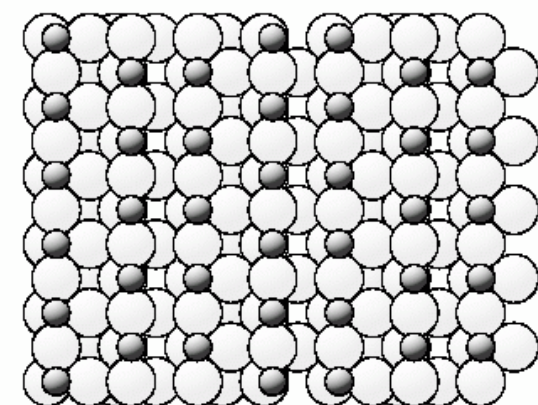
Overlayer



Missing Row 4



Missing Row 2



Perderau and Rhead



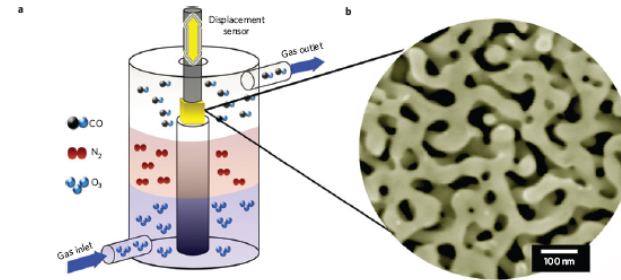
Robinson et al



Woodruff et al

Nanoporous Gold

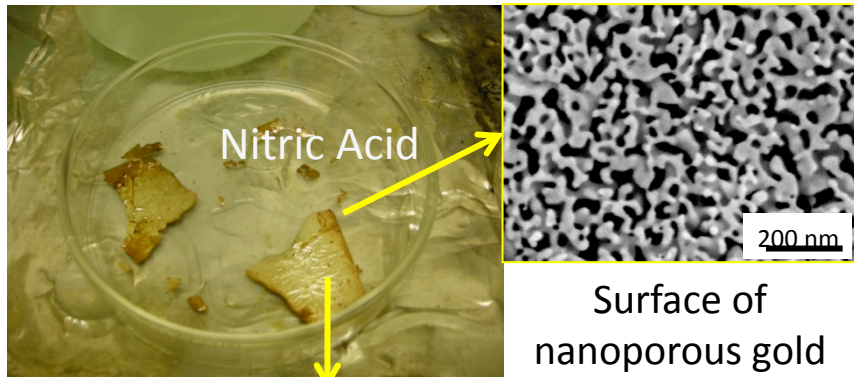
Karen Chen, NU, APS



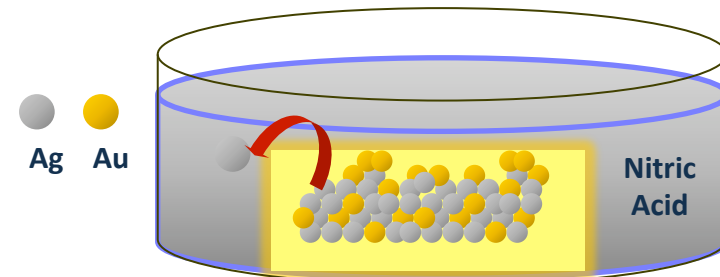
J. Biener et. al., Nature Material (2008)
Surface-chemistry-driven actuation

Dealloying:

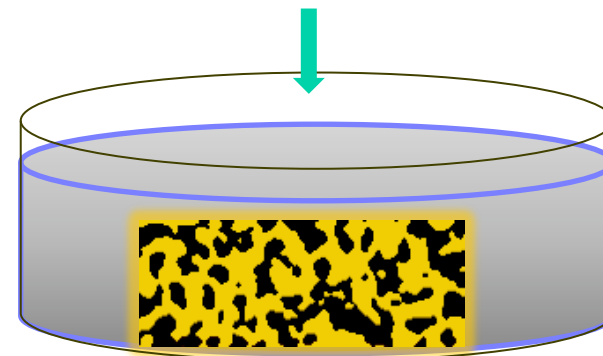
Nitric Acid dissolves silver atoms from silver-gold alloy → porous gold structure



Silver-gold alloy (Ag-30at% Au)



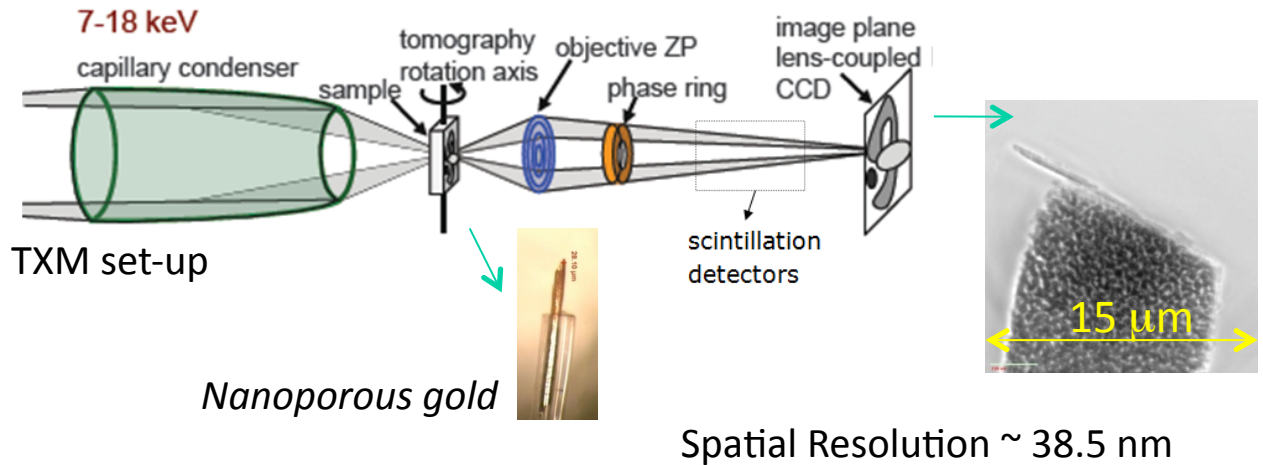
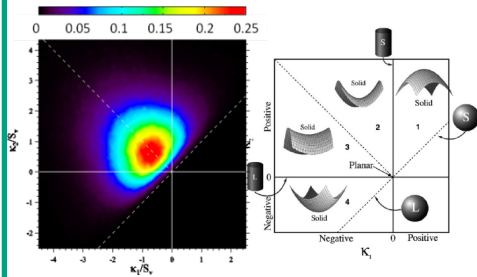
Dealloying happens not just on the surface but throughout the bulk sample



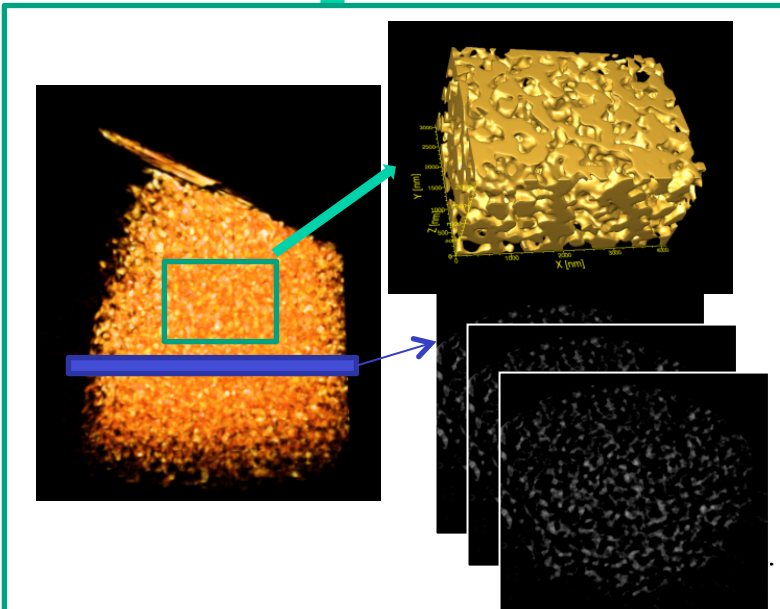
Porous structure

Transmission X-ray Microscopy Tomography

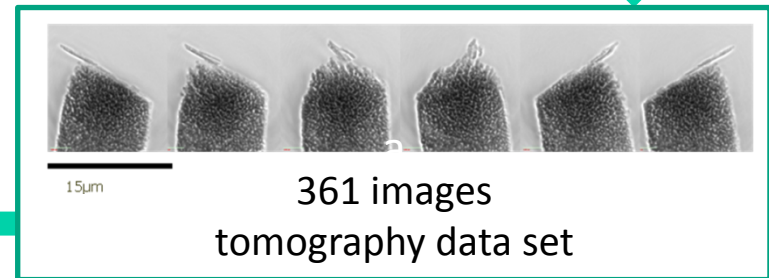
Eg. Principle curvatures distribution



Analysis



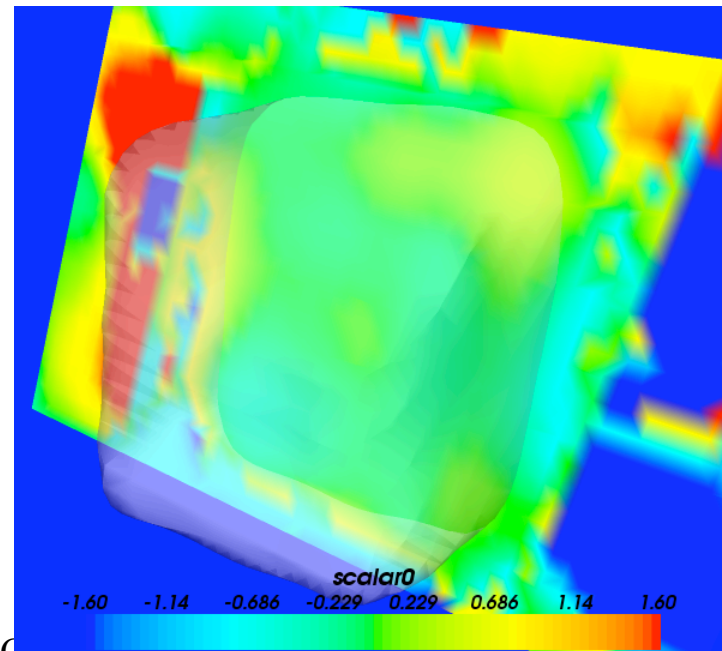
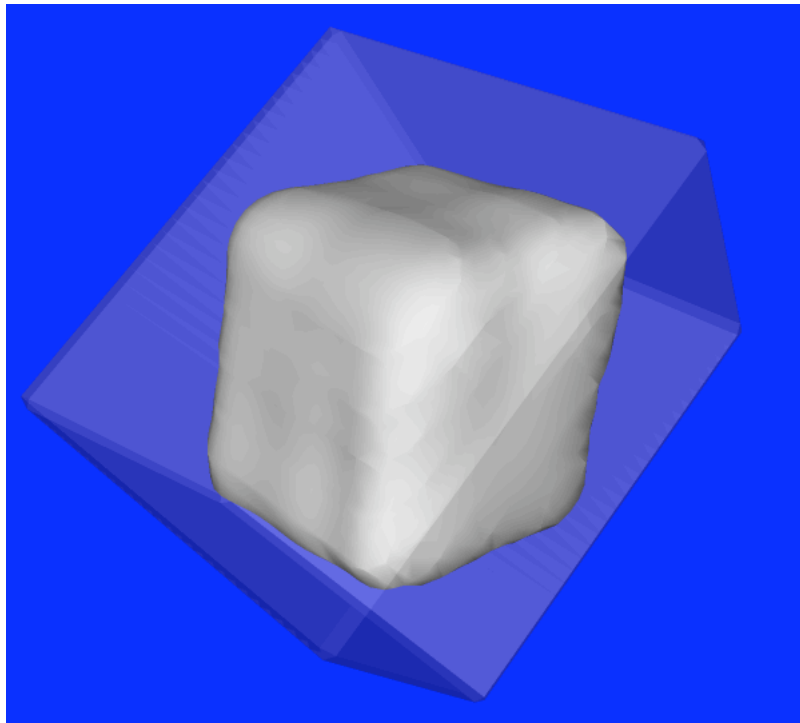
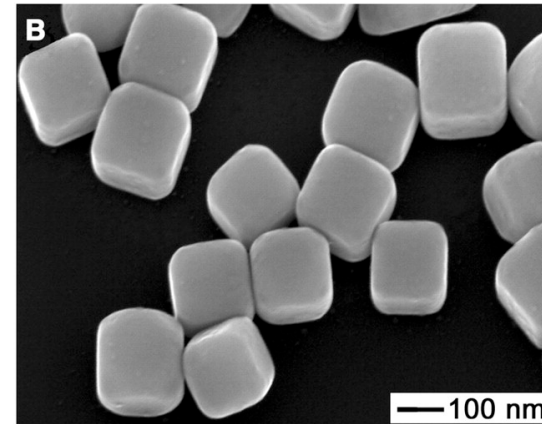
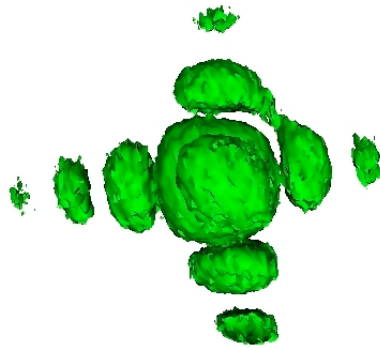
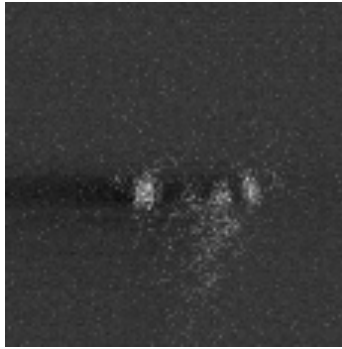
Tomography
Rotate 180 degrees
Images taken
at each 0.5 degree



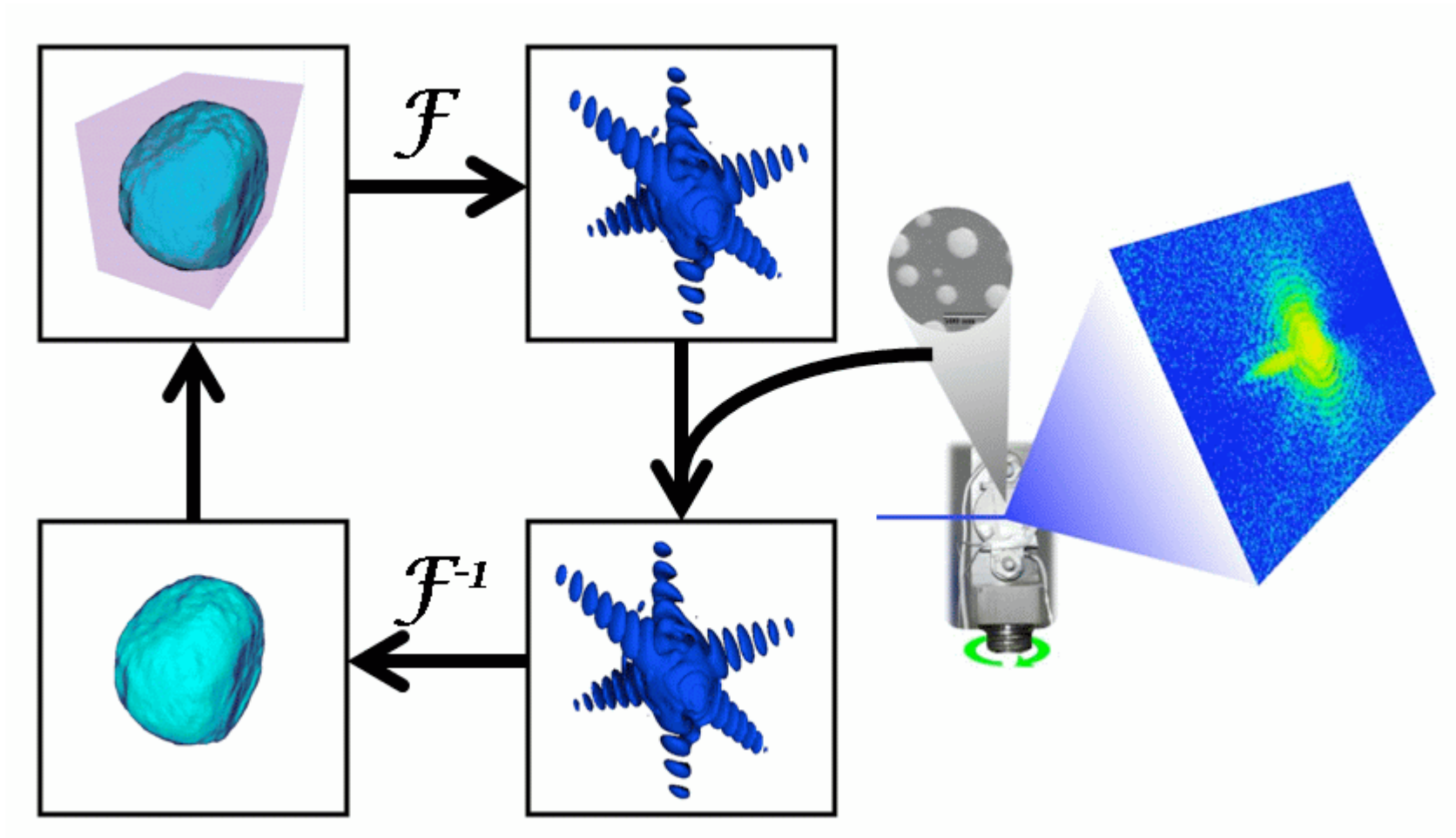
Reconstruction

Yu-chen Karen Chen, JaeMock Yi, Yong Chu, Wah Keat Lee, Qun Shen, Peter Voorhees, Ian McNulty and David Dunand

CXD, Ross Harder, APS



Generic “Error Reduction” method



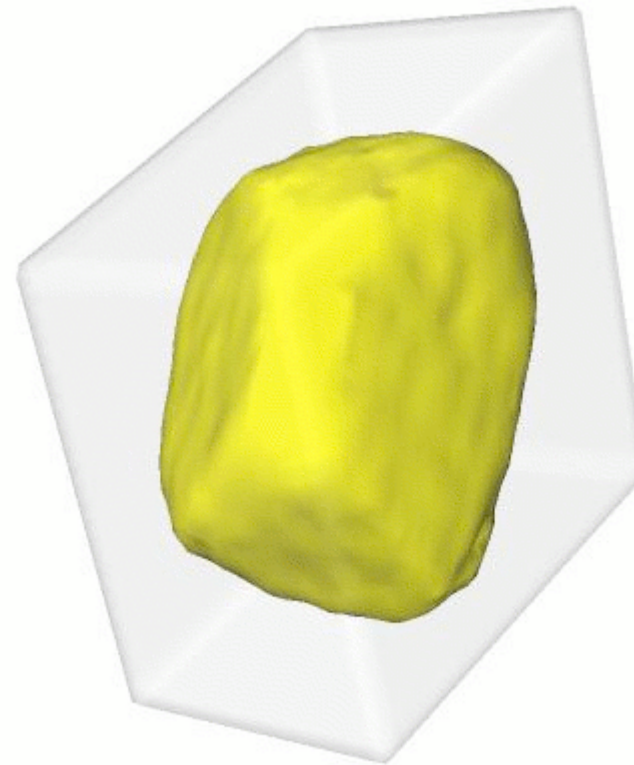
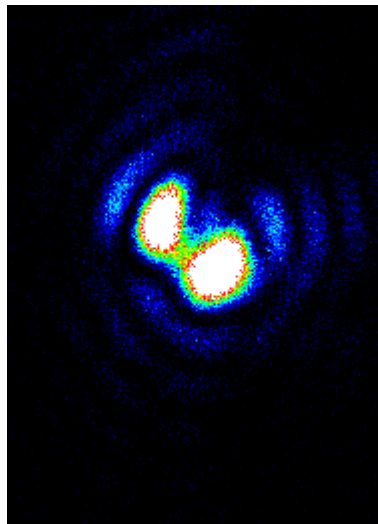
J. R. Fienup *Appl. Opt.* 21 2758 (1982)

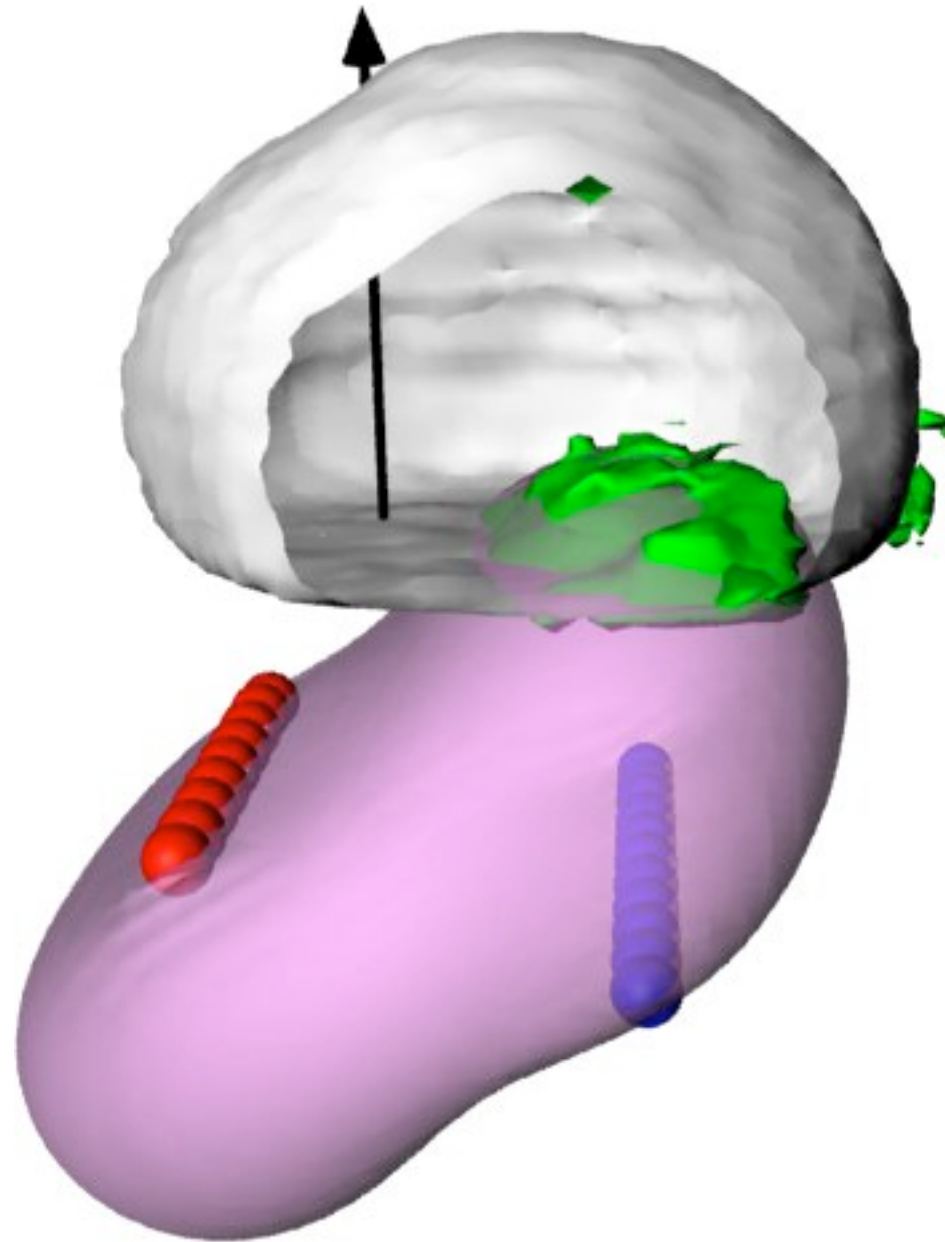
R. W. Gerchberg and W. O. Saxton *Optik* 35 237 (1972)

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Gold nanocrystal reconstruction

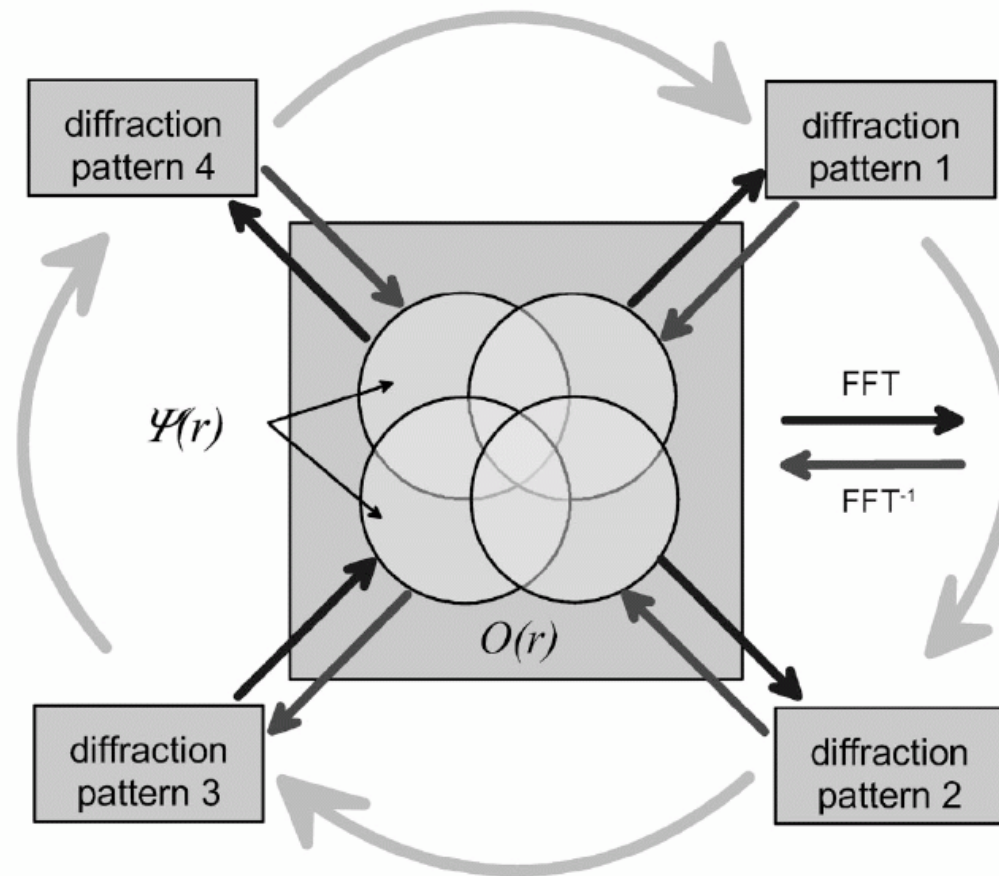
showing support used for 20 HIO followed by 10 ER





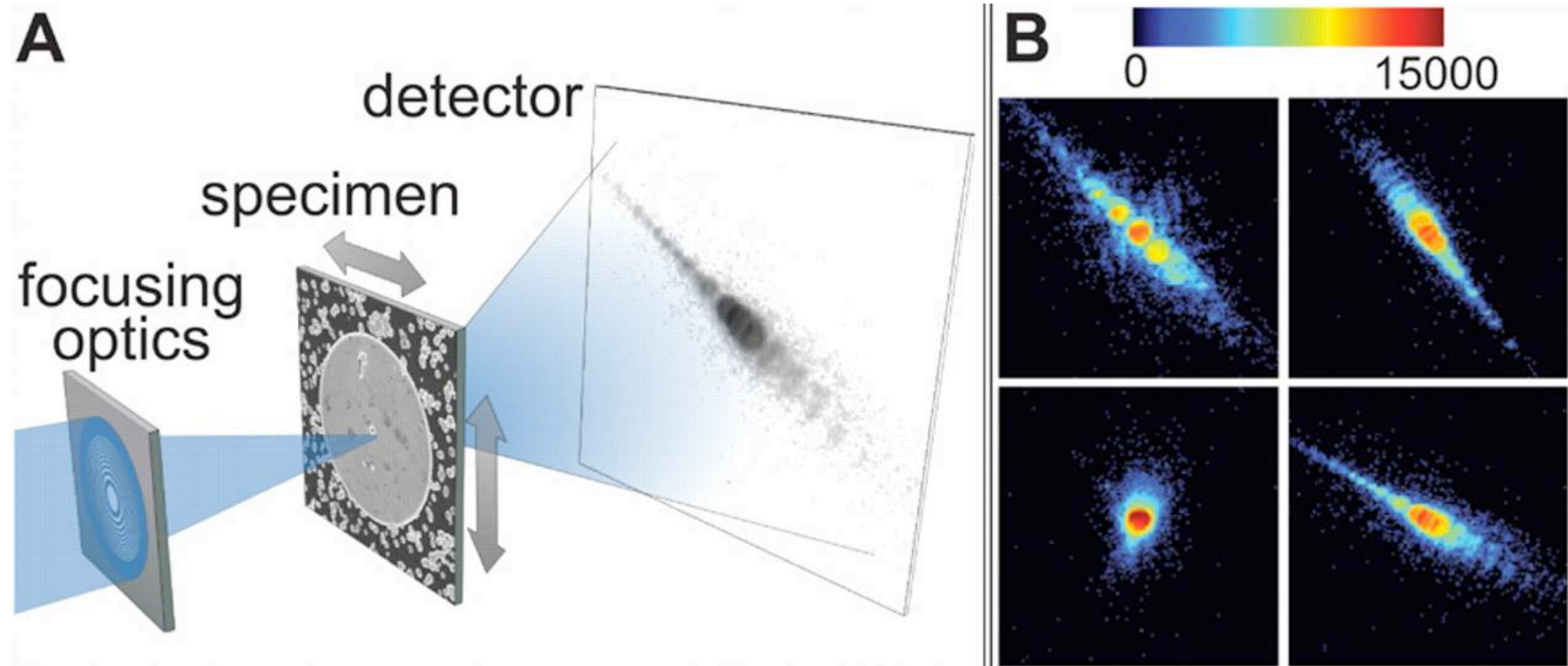
X-ray Ptychography

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



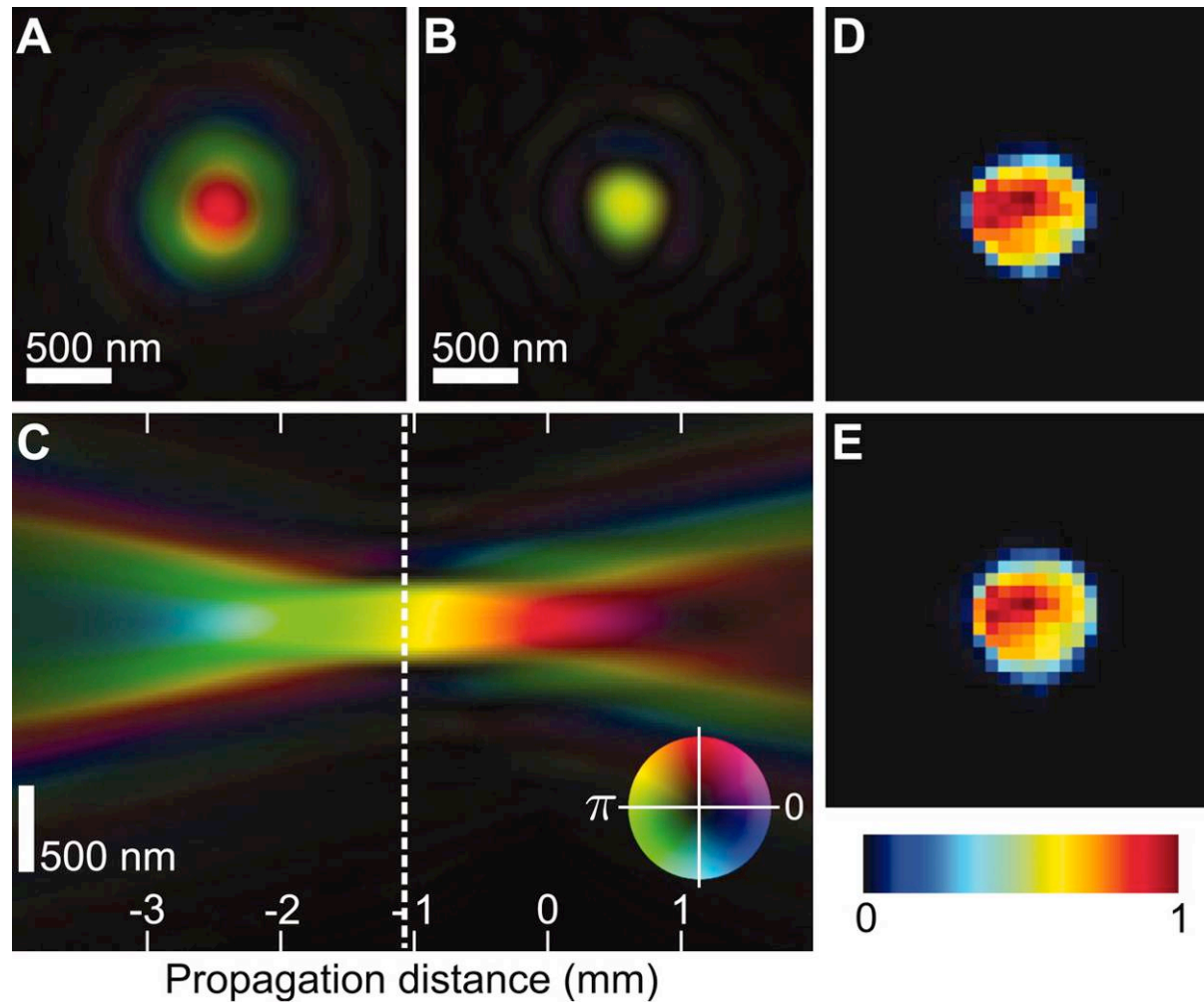
X-ray Ptychography

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



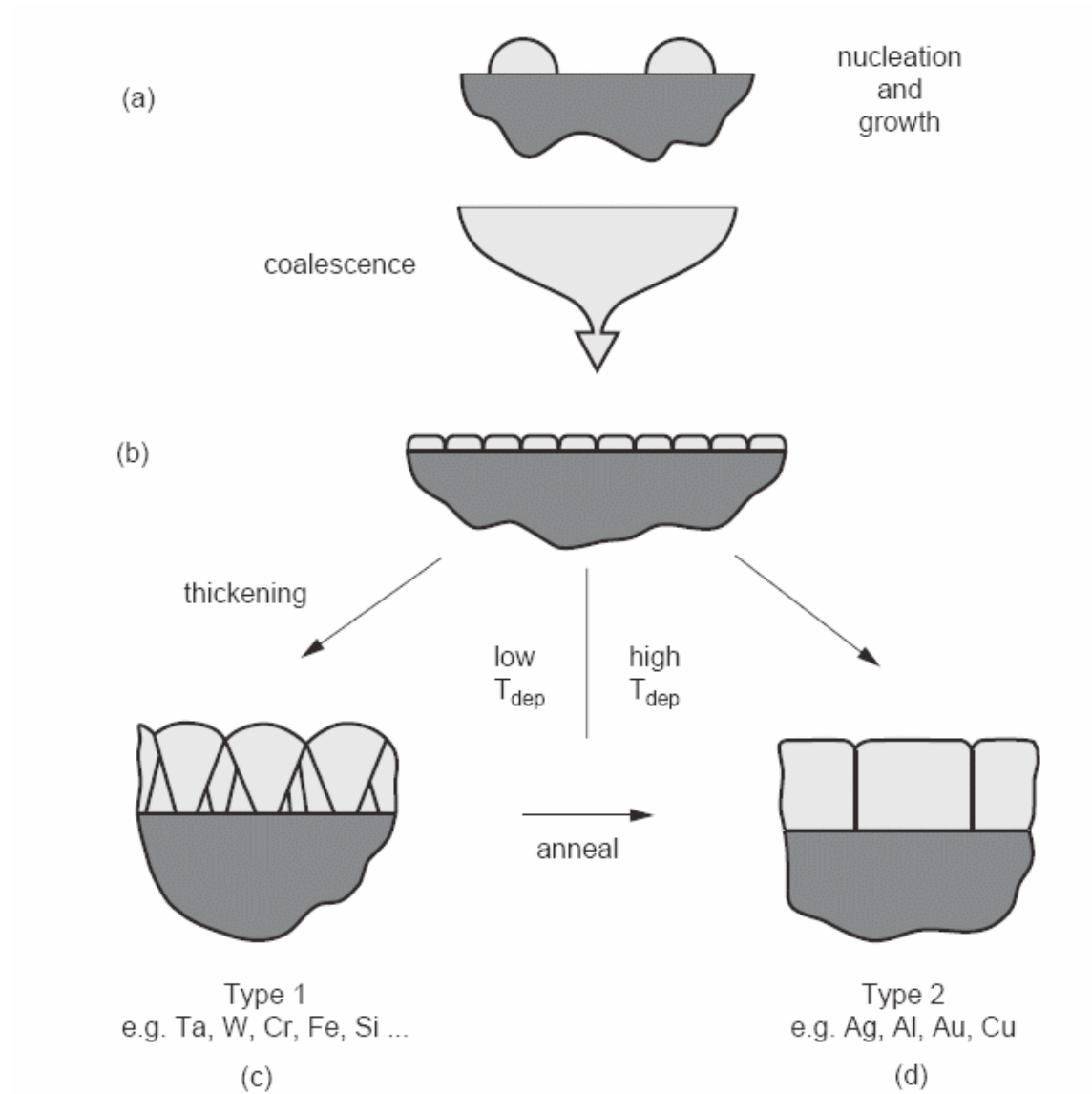
Reconstruction of Probe

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

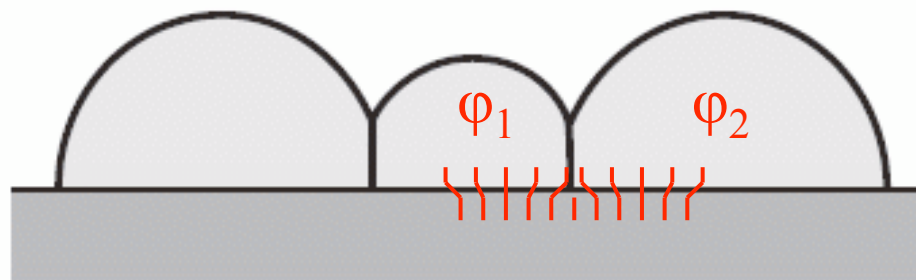
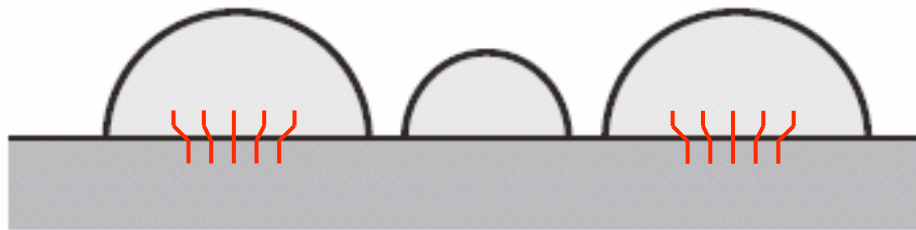


Thin film growth after deposition

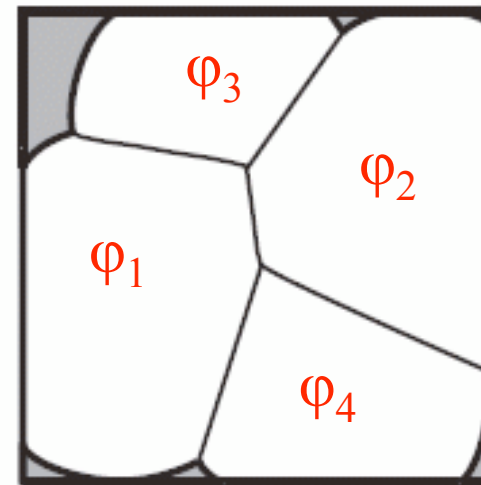
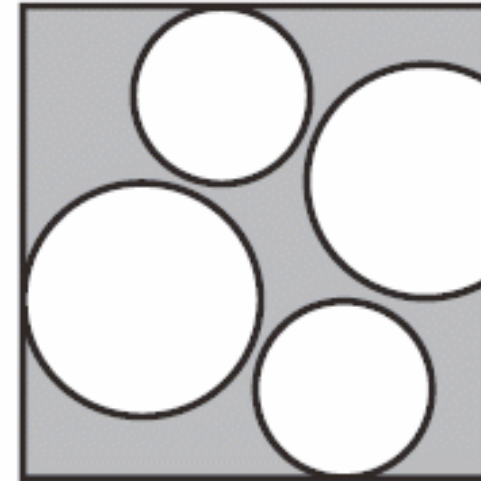
C. V. Thompson, Annu. Rev. Mater. Sci. 2000. 30:159–90



Epitaxial growth effects



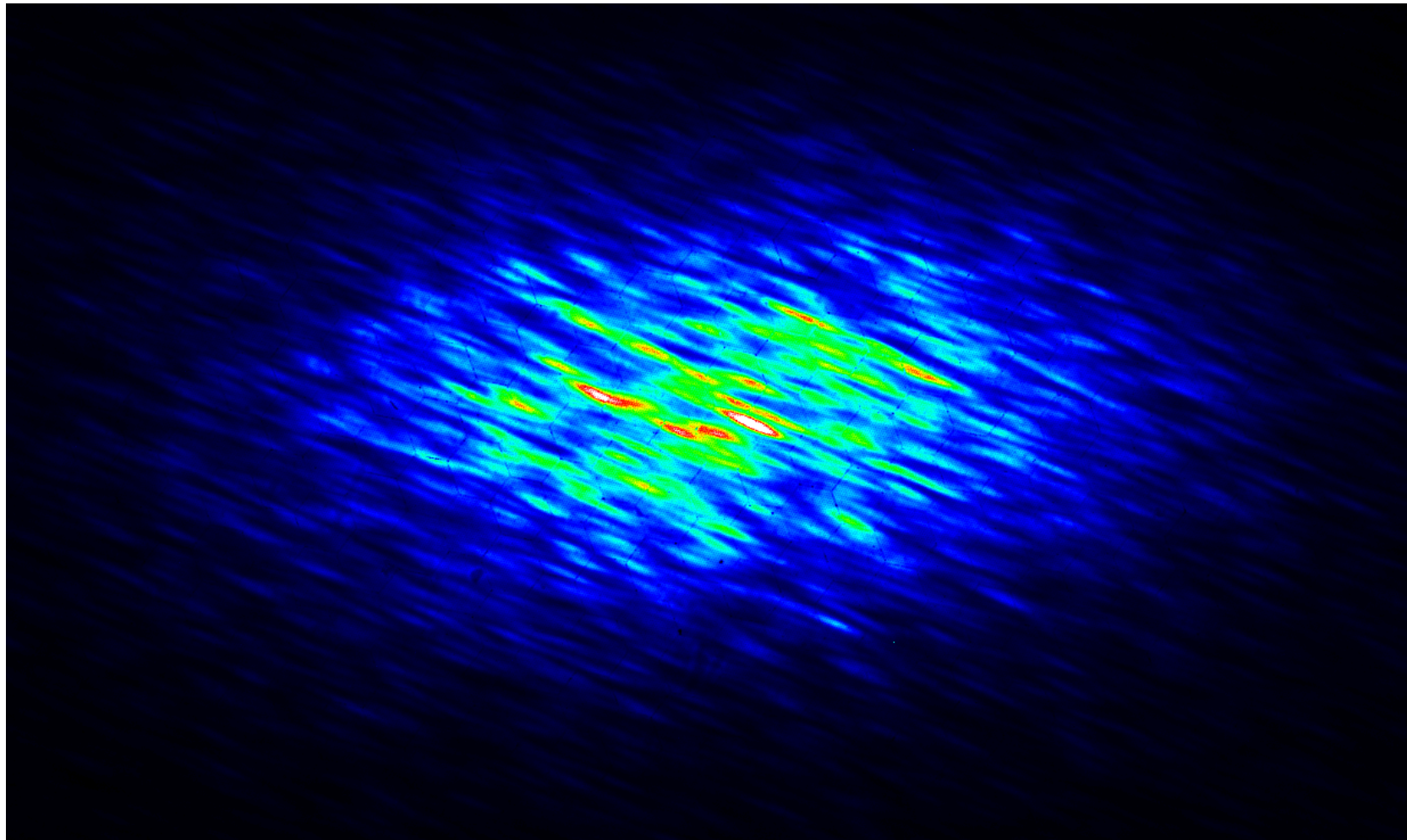
(a)



(b)

Niobium (110) Thin Film Grains

Richard Bean, I-16, Nb110-35 Jan 2009



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FeAl antiphase domains (001)

Lorenz Stadler, PhD dissertation, TU Wien (2005)

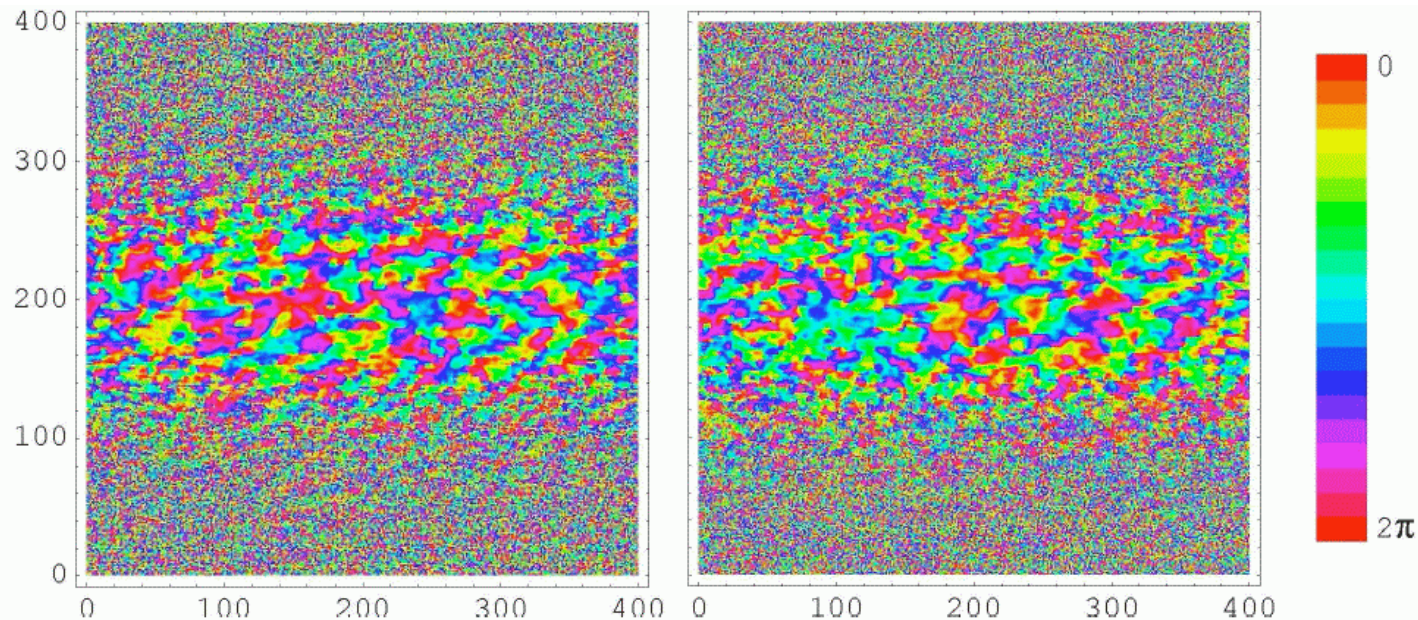
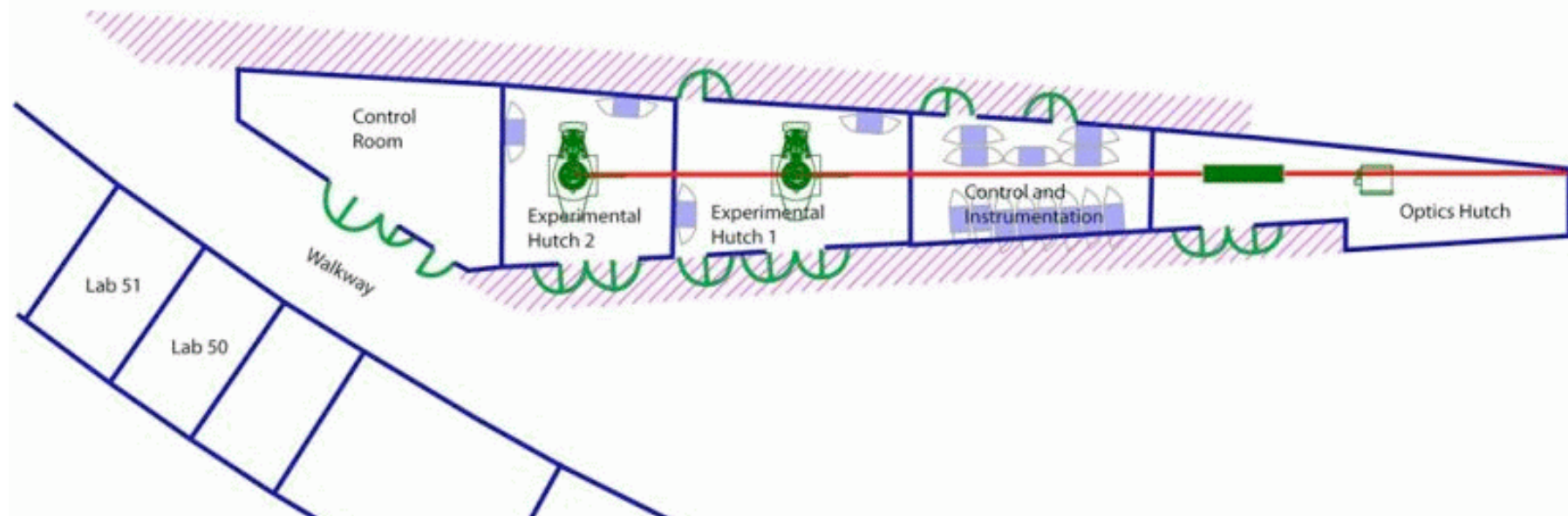


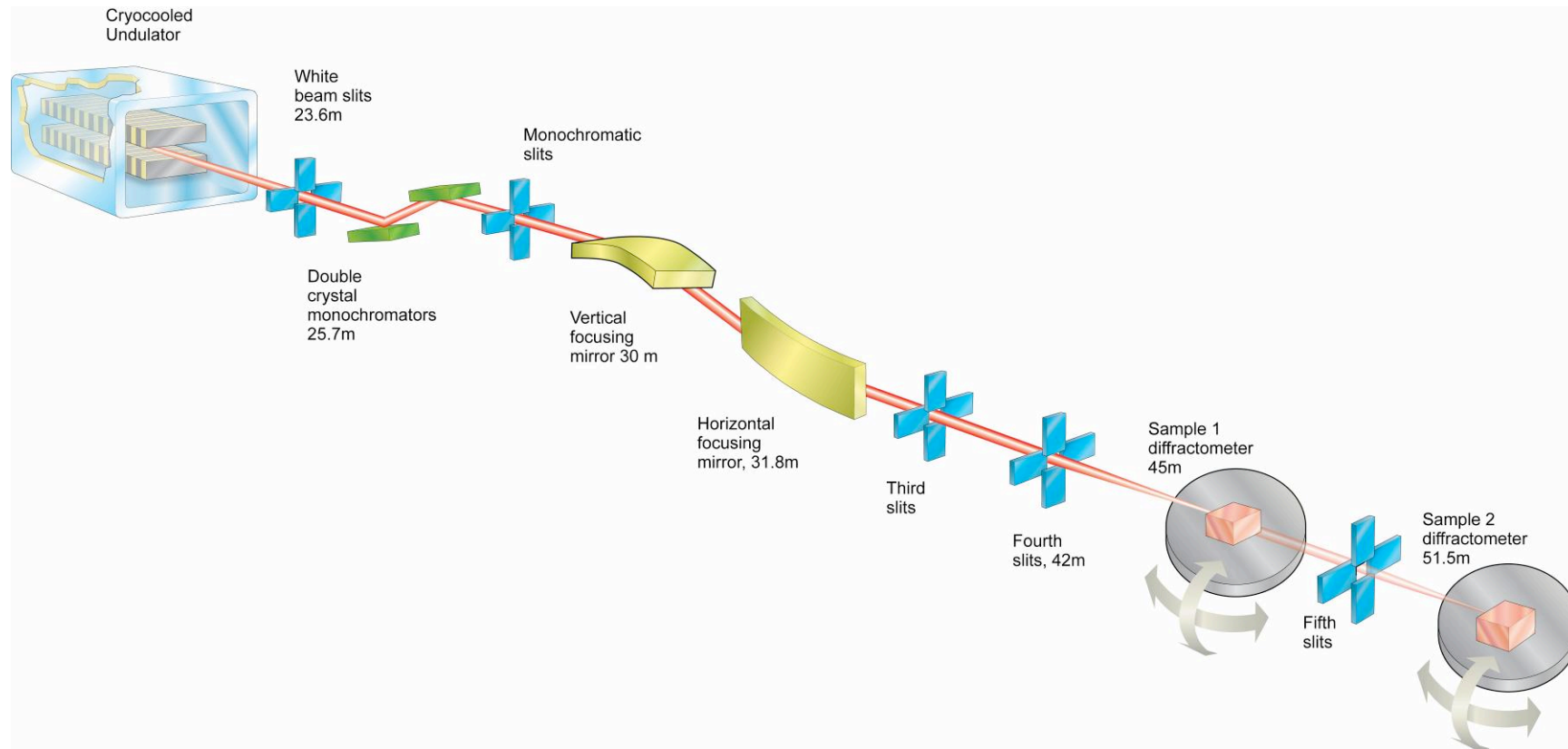
Figure 7.11: Typical reconstructed phases from runs with different combinations of algorithms and supports derived from the 2D Gaussian fit of the illumination function. Numbers in brackets denote how many iterations of the particular algorithm were done each cycle. Graphs on the left are from reconstruc-

I-07 Surface-Interface XRD

Beamline I07 - Layout



I-07 Surface-Interface XRD optics



I-07 experimental Hutch 1

- Hexapod or Eulerian cradle (load capacity: <50kg vertical mounting; <200kg horizontal mounting)
- Horizontal or vertical scattering geometry
- Scattering from liquid surfaces, $Q_z < 1 \text{ \AA}^{-1}$
- Ultrahigh vacuum baby chamber
- Closed cycle cryostat (10 - 800 K)
- Controlled atmosphere environments
- Furnace (300 – 1500K)
- Electrochemical and solid-liquid cells
- Liquid and Langmuir troughs

I-07 experimental Hutch 2

- 2+3 Circle Diffractometer
- Hexapod sample stage
Vertical Scattering Geometry
- Large ultrahigh vacuum sample chamber (removable)
 - Ability to mount other ‘large’ chambers
 - In situ preparation and characterisation using standard surface science techniques.
 - Sample heating up to $\sim 1800\text{K}$

I-07 supporting Lab

- Nima Langmuir Trough
- Nanofilm Imaging Spectroscopic Ellipsometer (incorporating Brewster angle microscopy)
- Kruss Drop Shape Analysis Tensiometer
- Laurell Spin Coater
- UV-Ozone cleaner
- Veeco Atomic Force Microscope
- Offline UHV chamber inc, STM, LEED, XPS, etc.