Coherent X-ray Diffraction imaging of strain fields in Nanocrystals

Ian Robinson
Ross Harder
Steven Leake
Marcus Newton
Xiaowen Shi
Gang Xiong
Loren Beitra

London Centre for Nanotechnology
Harwell Research Complex

East Midlands Materials Society, Leicester, February 3, 2011
Outline

• Coherent x-ray diffraction
• CXD can solve the phase problem
• Nanocrystal structures
• Crystal strain as complex density
• Nanowire structures
• Full strain tensor
London Centre for Nanotechnology

- Clean Rooms
- Low-T STM
- Lithography
- 3-beam FIB
- Visualisation
- CLS?
Nanocantilevers
Dr Rachel McKendrie
Size-dependent Melting of Au Particles
Structure of Gold vs Size

L. D. Marks, RPP (1994)  
Koga and Sugawara (2003)
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
Diamond Light Source (RAL)
Diamond
in-vacuum
X-ray
Undulator
Longitudinal Coherence

$2L_L = N \lambda$

Als-Nielsen and McMorrow (2001)

$L_L = \frac{1}{2} \frac{\lambda^2}{\Delta \lambda}$
Lateral (Transverse) Coherence

\[ L_T = \frac{\lambda R}{2D} \]

Als-Nielsen and McMorrow (2001)
Robert Hooke’s microscope, 1665

National Museum of Health and Medicine, Washington DC
Lensless X-ray Microscope, 2003
Generic “Error Reduction” method

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

I. K. Robinson,  EMMS 2011
Chemically Synthesized Silver Nanocubes

Yugang Sun and Younan Xia,
Rocking scan of Ag cubes with 0.01° steps
In situ growth of Pb crystals
Coherent Diffraction from Crystals

I. K. Robinson, EMMS 2011
Sensitivity to strain
\[ \Delta \varphi = k_f \cdot u - k_i \cdot u = Q \cdot u \]
Good statistics, 3D diffraction data

Figure 4.12: Center slices from 3D CXD pattern from Pb sample, on a log scale. Data file 296 from 10/03.
Modeling of 3D Phase Bump
3D phase map sections
Refraction effects in Lead at 8.9keV

Phase accumulation due to refraction along scattering path

\[ n = 1 - \delta + i\beta \]

\[ \delta = 2.23 \times 10^{-5} \]
\[ \beta = 2.19 \times 10^{-6} \]
Refraction corrected phase map

Max phase = 1.15 rad
  = 0.052 nm

Phase on the (111) facet:
  = 0.47 rad
  = 0.02 nm
Gold nanocrystal reconstruction
showing support used for 20 HIO followed by 10 ER
Phase isosurface of residual strain

I. K. Robinson, EMMS 2011
Single Au nanocrystal synthesis

I. K. Robinson, EMMS 2011
Two views of strain in Au NC
Au409B-52 (11-1) and Au409B-60 (200)
Vector displacement field

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

I. K. Robinson, EMMS 2011
Confocal Alignment Microscope
Reconstruction of InP nanowire
CVD on Si, Suneel Kodambaka, UCLA
GaAs Nanowire “Barcode”
Vincent Favre-Nicolin, Joel Eymery (CEA), Rienk Algra (Philips), Ross Harder
ZnO Sample Preparation

Dimensions: 4-5µm

I. K. Robinson, EMMS 2011
Six Independent Bragg Peaks

(0,1,1)  (0,-1,1)  (1,0,1)  (-1,0,1)  (-1,1,1)  (1,-1,1)
Separate images 6 Bragg Peaks

I. K. Robinson, EMMS 2011
**Full Strain Tensor**

\[
\epsilon_{ij} = \frac{1}{2} \left( \frac{\partial u_j}{\partial x_i} + \frac{\partial u_i}{\partial x_j} \right), \quad \tau_{ij} = \left( \frac{\partial u_j}{\partial x_i} - \frac{\partial u_i}{\partial x_j} \right)
\]

I. K. Robinson, EMMS 2011
ZnO Rigid-body Rotations

\[ \tau_{xy} \quad \tau_{xz} \quad \tau_{yz} \]

I. K. Robinson, EMMS 2011
Mosaic domains on SOI film

- Pitch Orientation in mRad
- Roll Orientation in mRad

Position in X of SOI sample in Micron
Coherent or incoherent effect?
Conclusions

• CXD is a new branch of Crystallography
• 3D imaging practical for nanocrystals
• Phasing by computation instead of lens
• Strain fields imaged from asymmetric patterns
• Applications to semiconductors and metals