Coherent Diffraction Methods in electrochemistry

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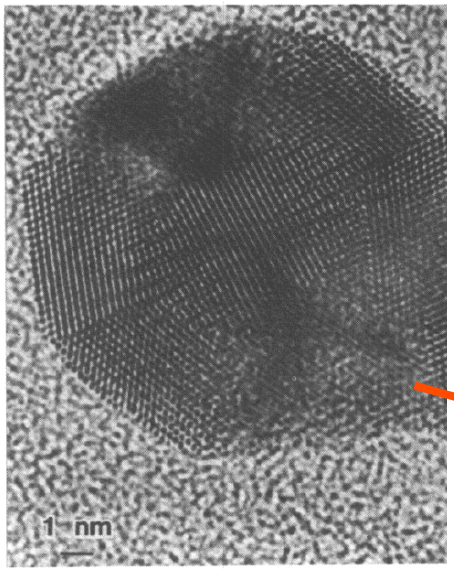
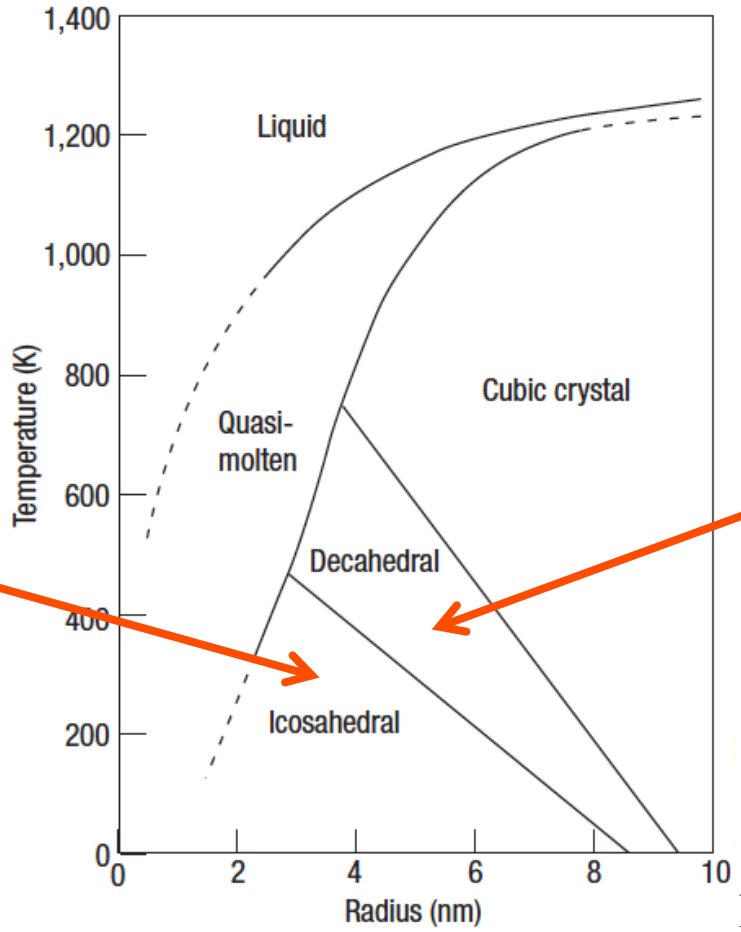
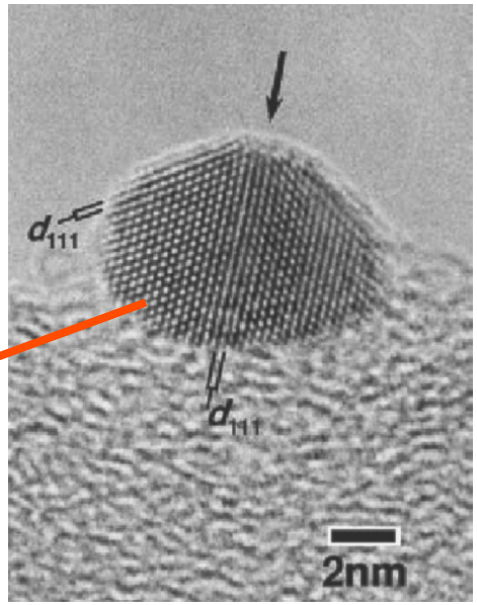
Outline

- Nanocrystal structures
- Coherent x-ray diffraction
- Crystal strain as complex density
- Strain induced by surface reaction
- Structural change during alloying
- Time resolve *in-situ* imaging

Structure of Gold vs Size



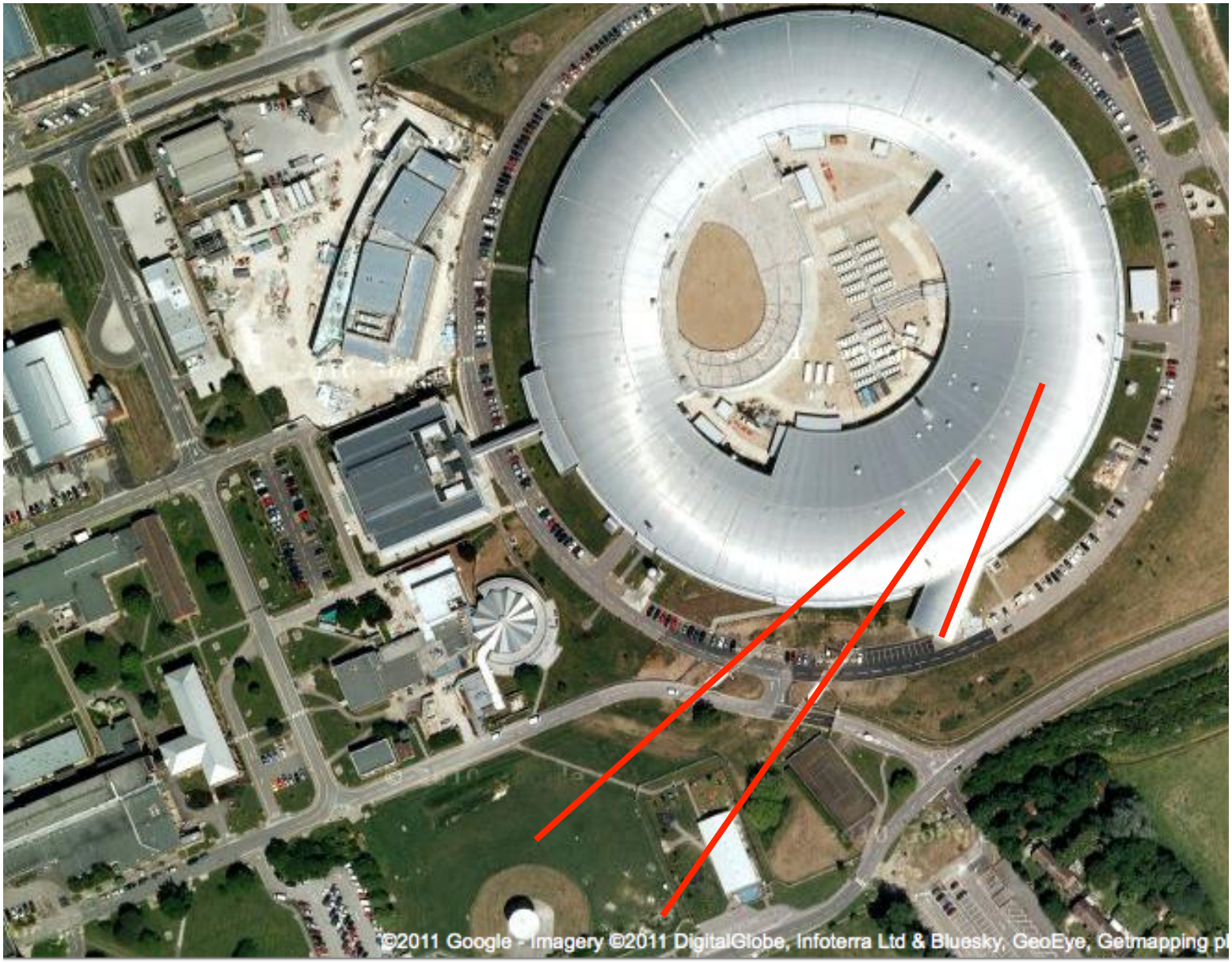




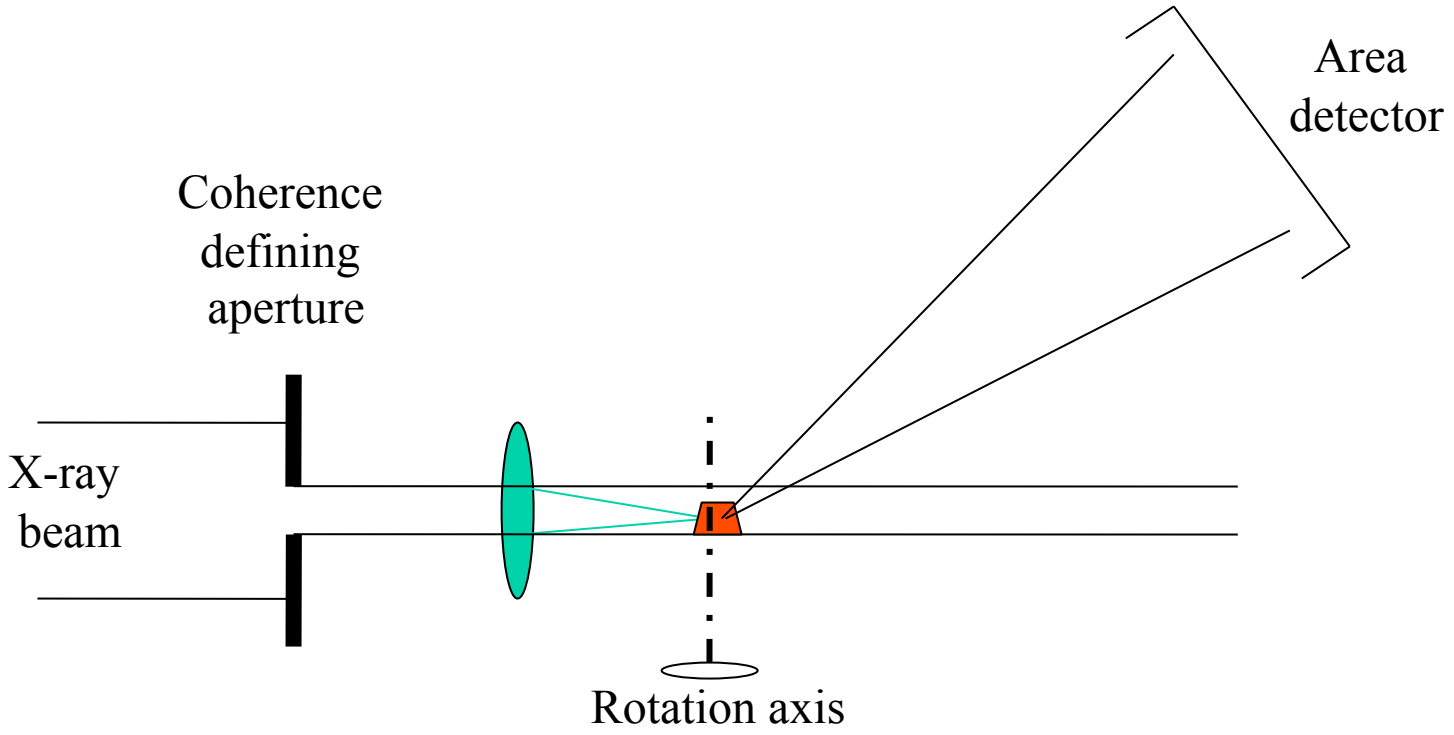
L. D. Marks, RPP (1994)

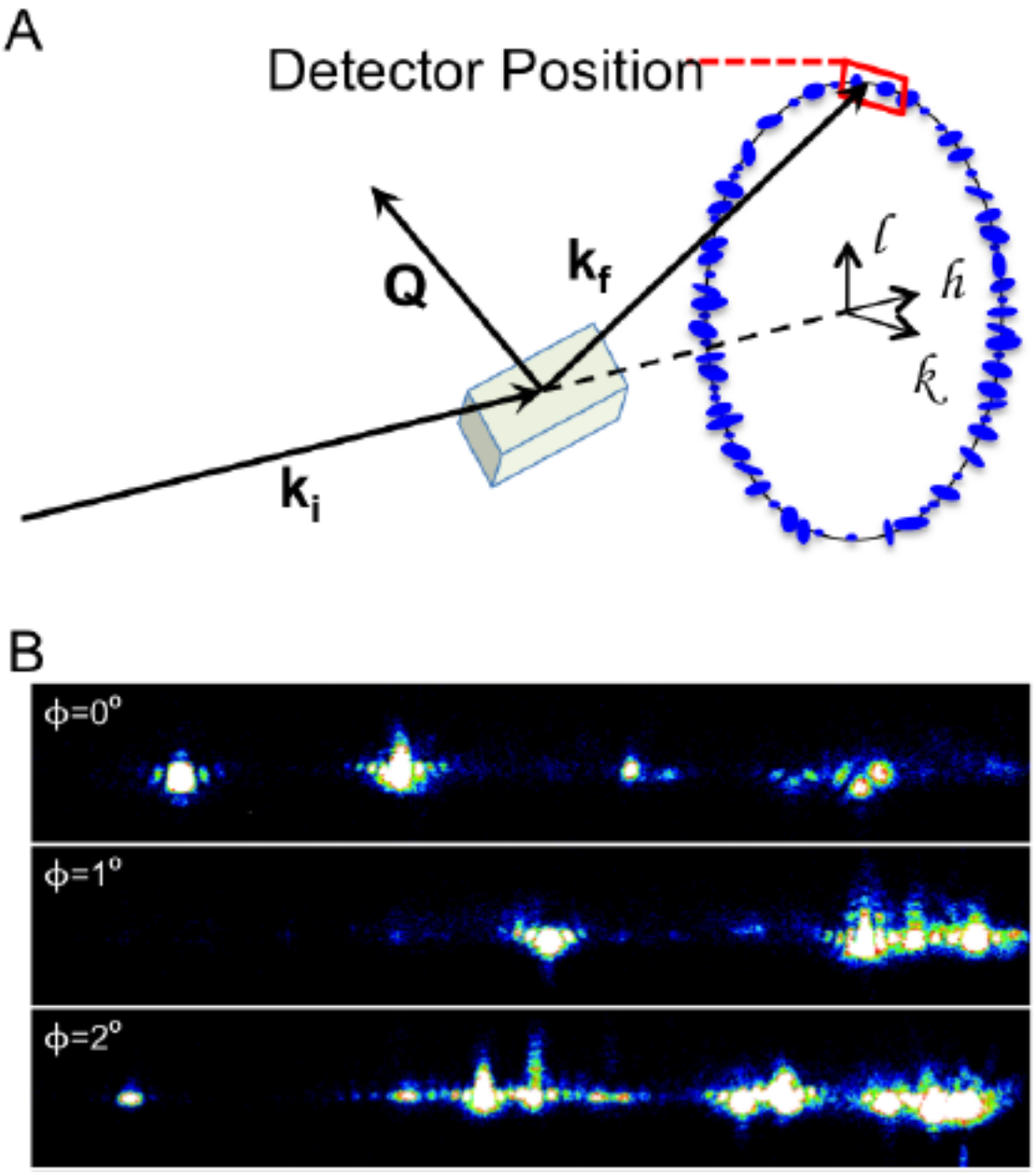
Koga and Sugawara (2003)

Diamond Light Source, 2009

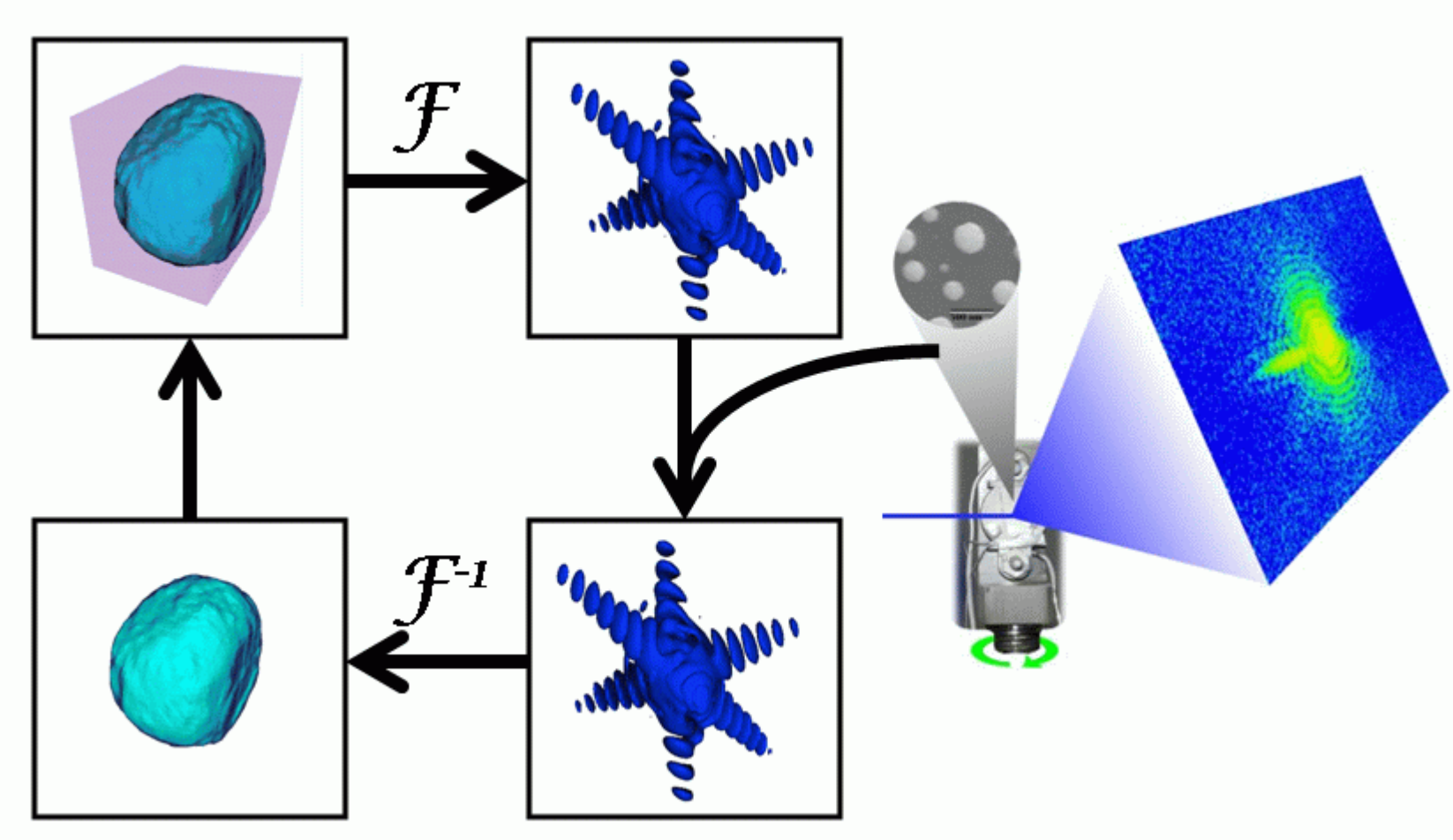


Lensless X-ray Microscope, 2003




A
Detector Position
Q
k_f
k_i
l
h
k
B
ϕ=0°
ϕ=1°
ϕ=2°

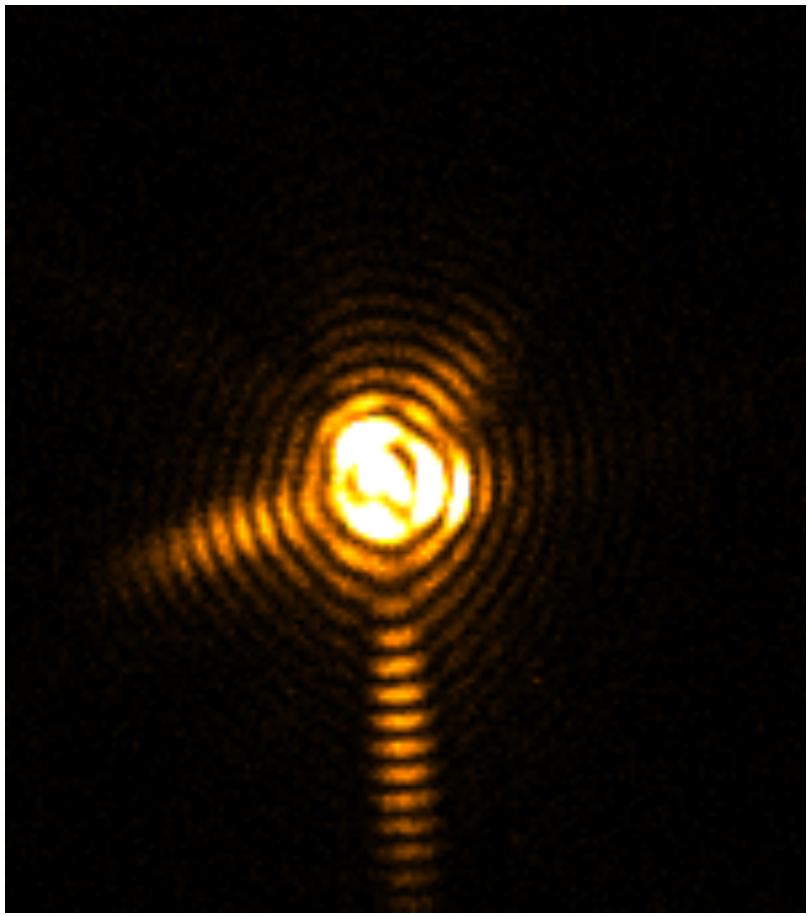
Generic "Error Reduction" method

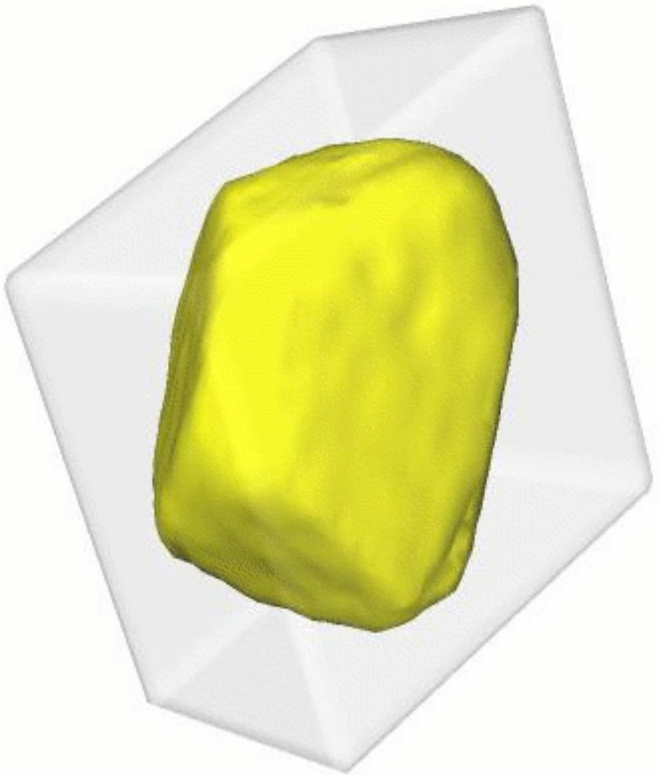


J. R. Fienup Appl. Opt. 21 2758 (1982)
R. W. Gerchberg and W. O. Saxton Optik 35 237 (1972)

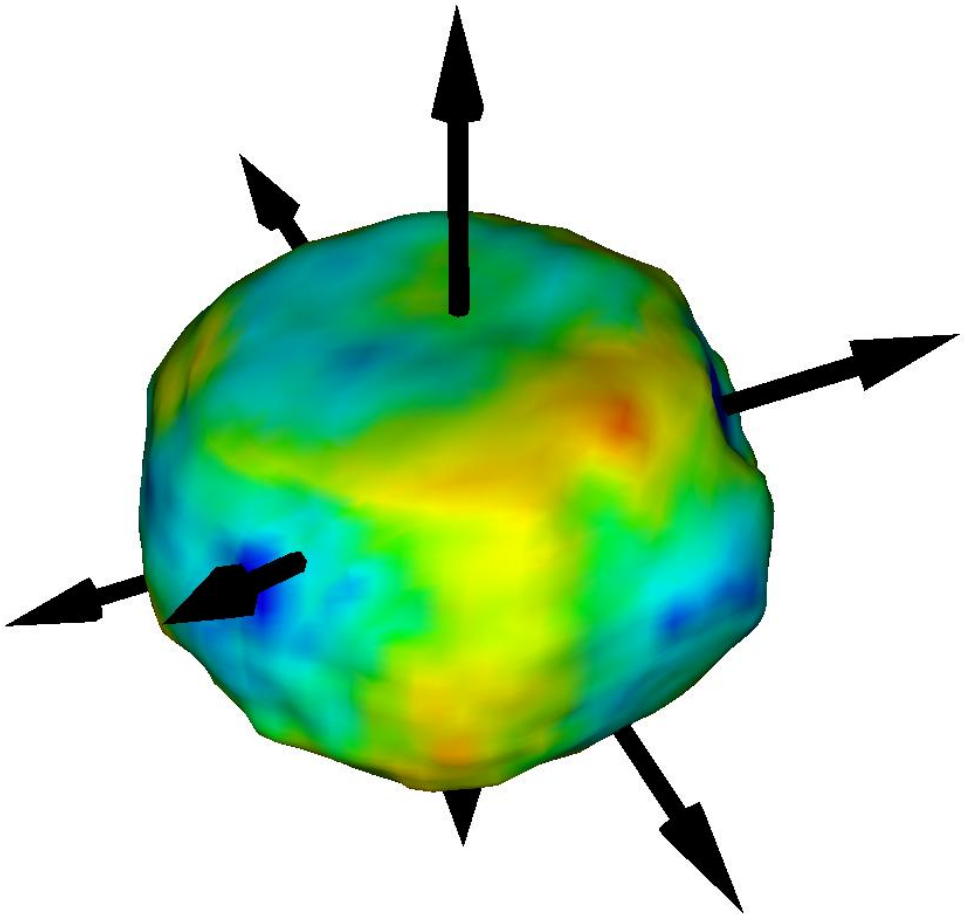
Gold nanocrystal reconstruction

showing support used for 20 HIO followed by 10 ER



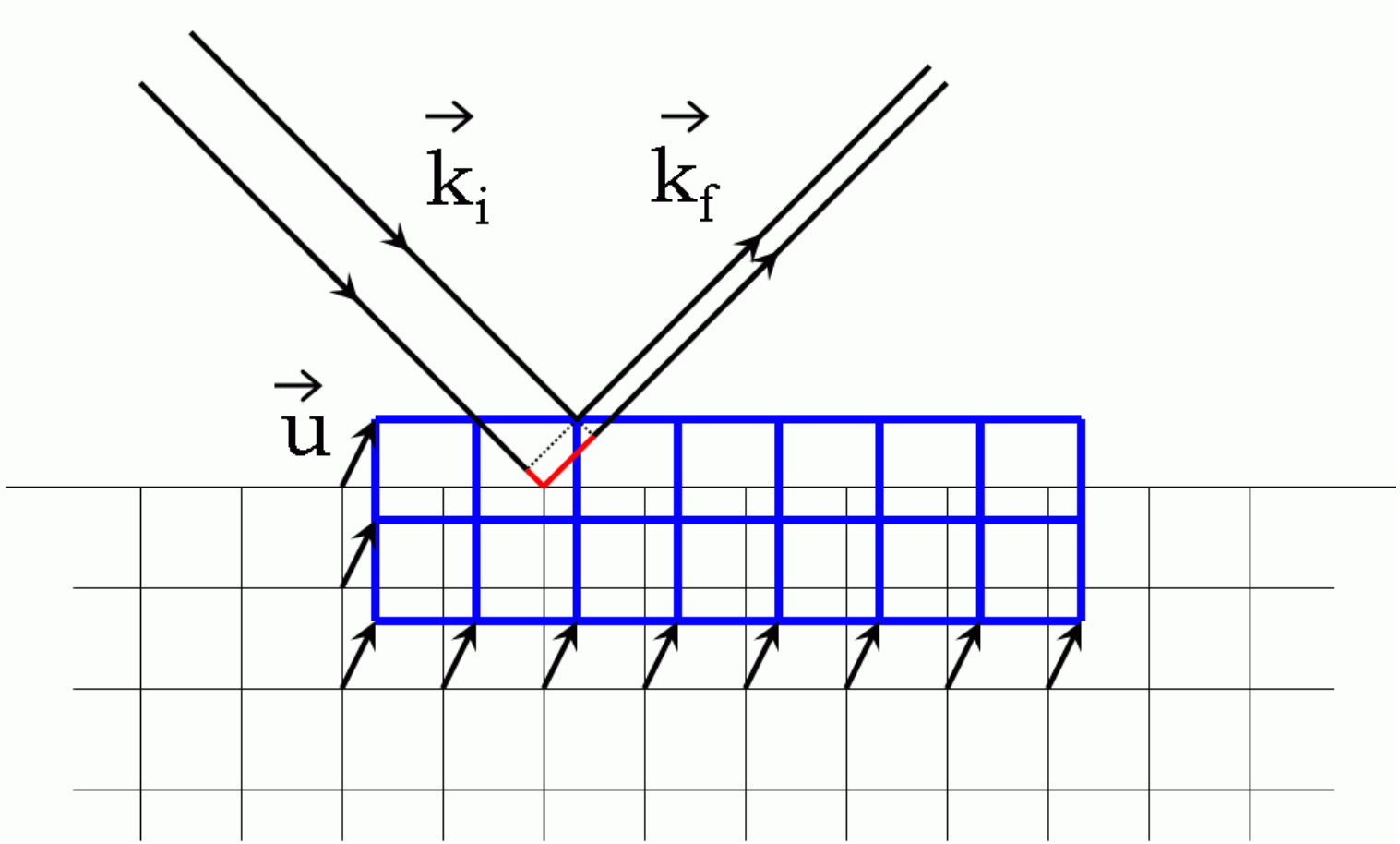


Phase isosurface of residual strain



Sensitivity to strain

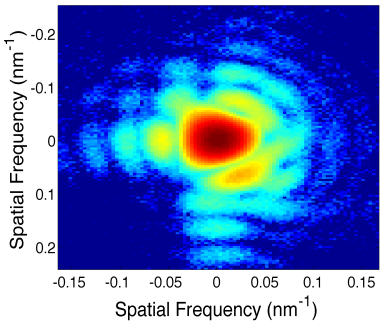
$$\Delta\varphi = \mathbf{k}_f \cdot \mathbf{u} - \mathbf{k}_i \cdot \mathbf{u} = \mathbf{Q} \cdot \mathbf{u}$$



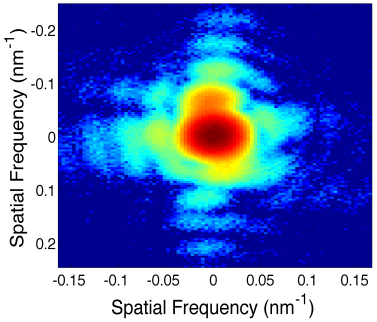
Formation of Al-Au alloy

Heating of a Au nanocrystal coated with Al (Jesse Clark)

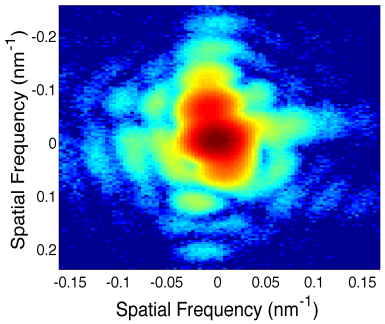
T=30°c



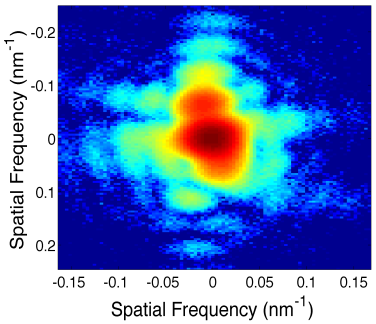
T=80°c



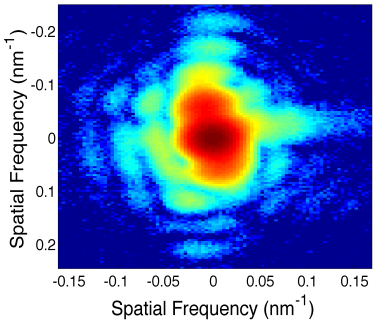
T=150°c



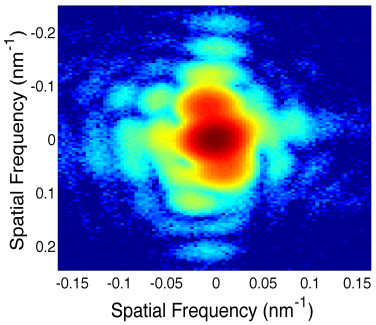
T=200°c



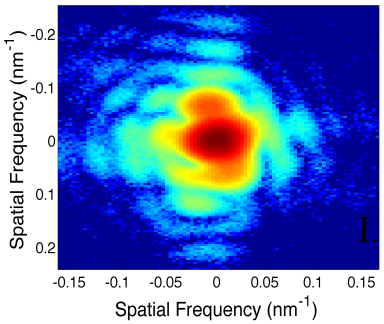
T=250°c



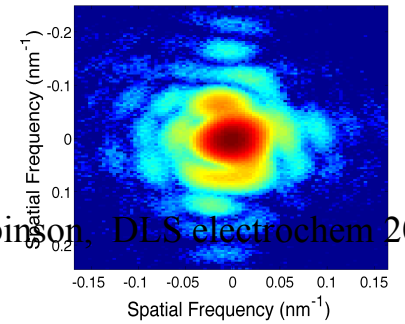
T=300°c



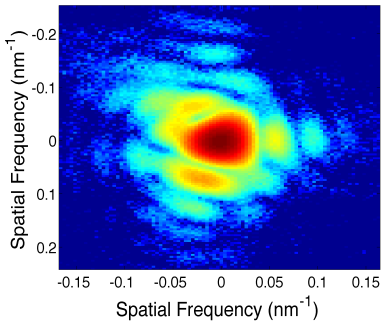
T=350°c



T=400°c



T=440°c



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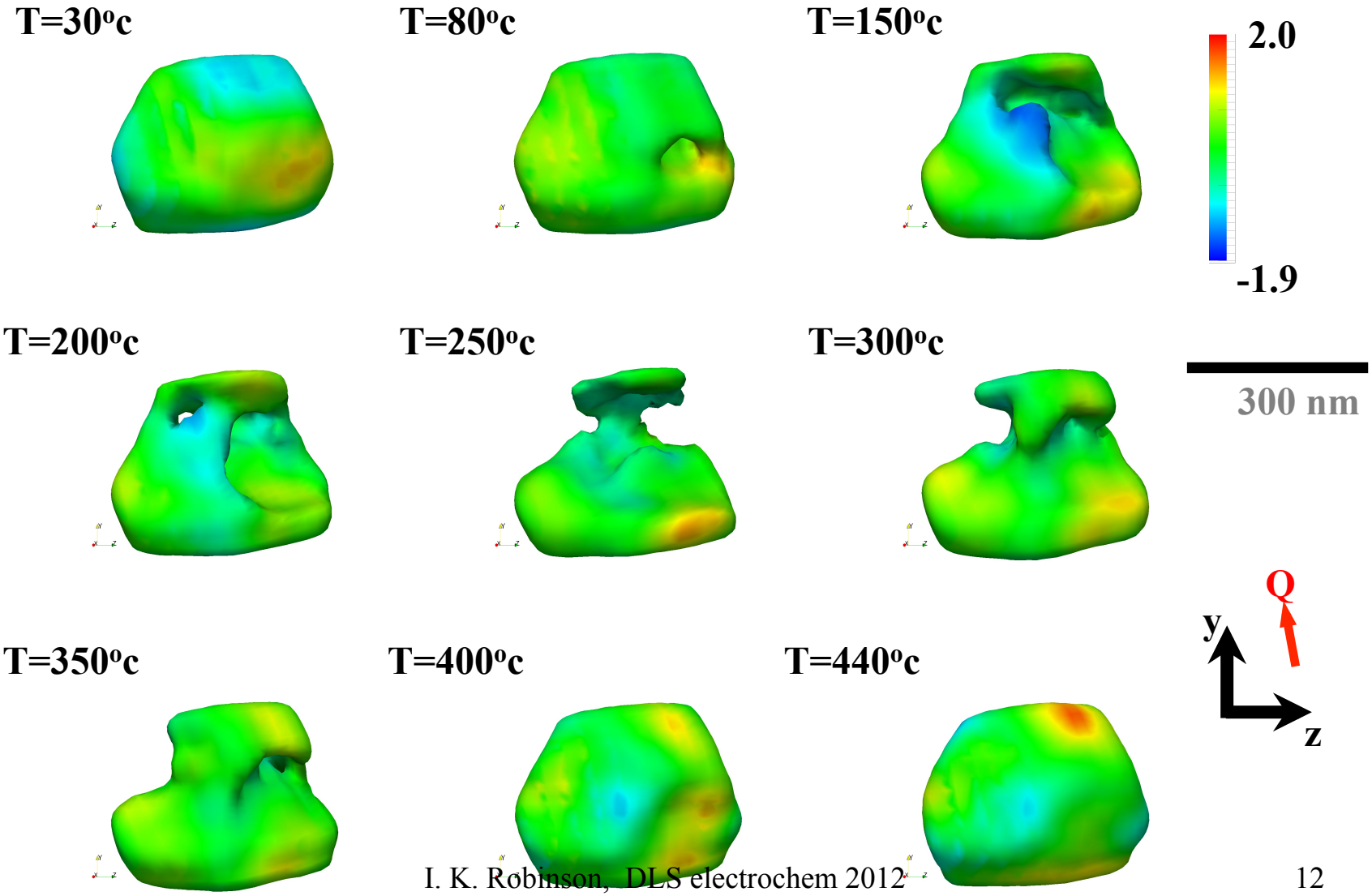

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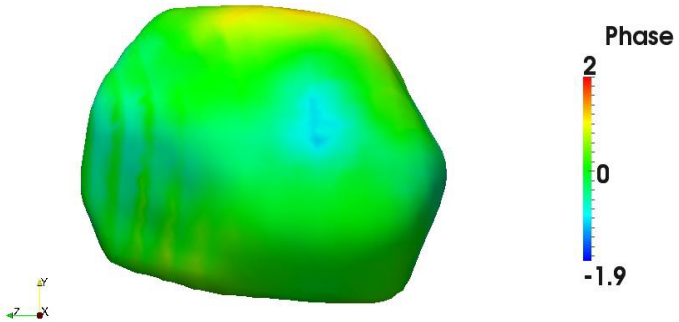
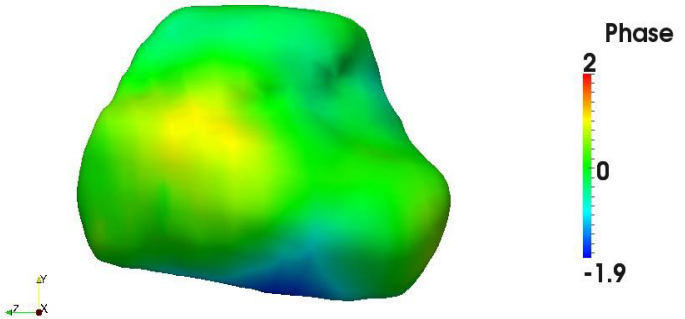
Heating of a Au nanocrystal coated with Al (Jesse Clark)

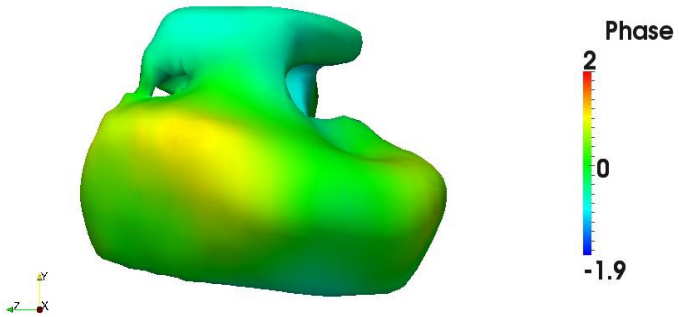
T=30°c



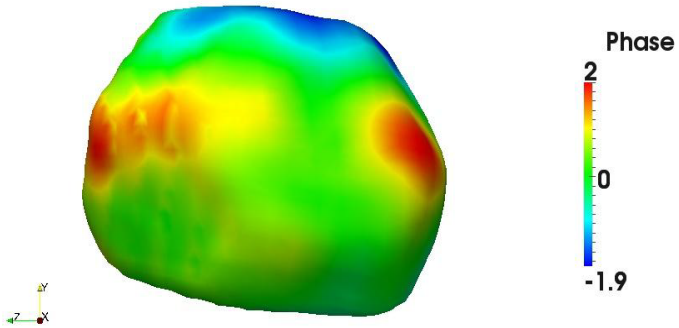
T=150°c



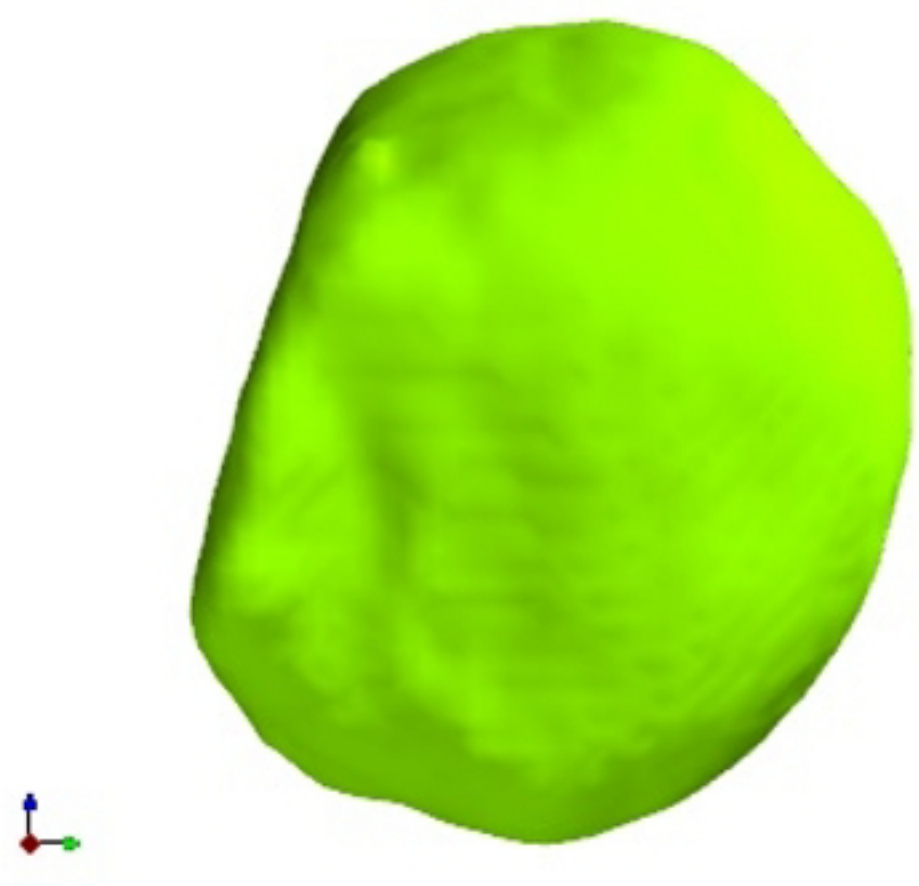
T=300°c



T=440°c

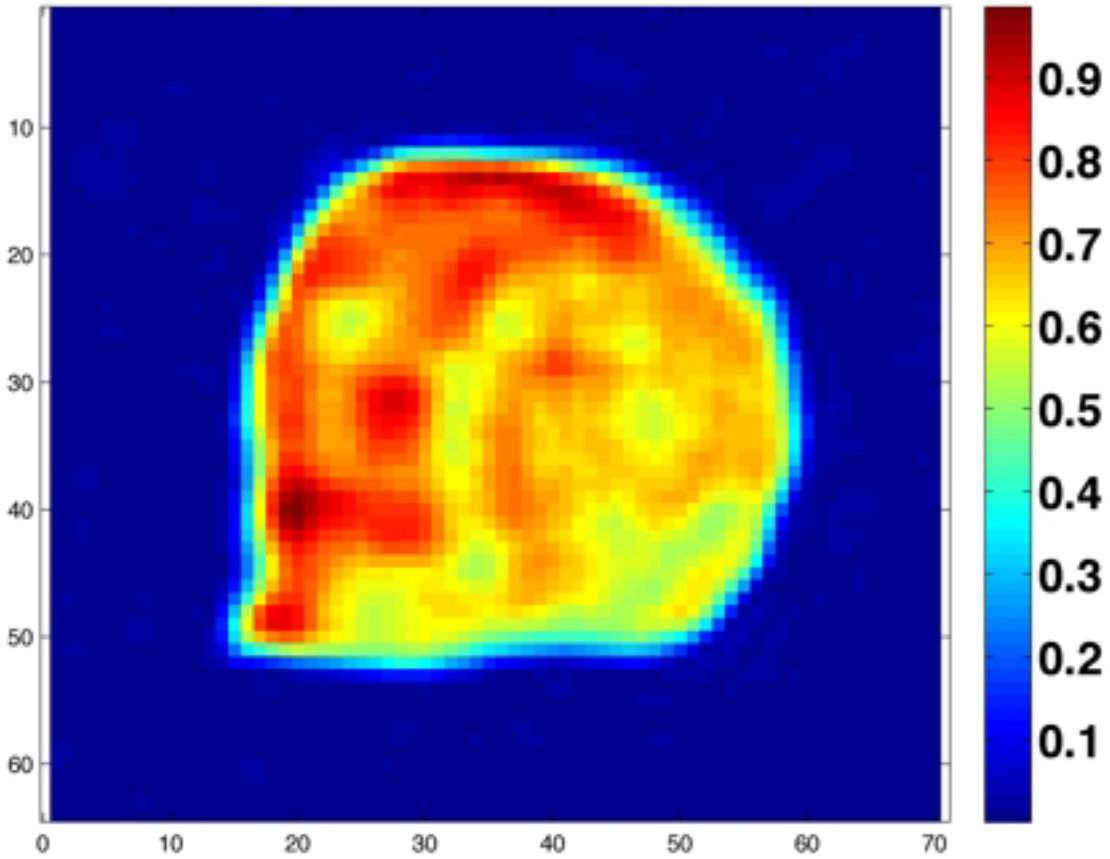


In-situ deposition of Cu onto Au nanocrystal at 300C (Gang Xiong)

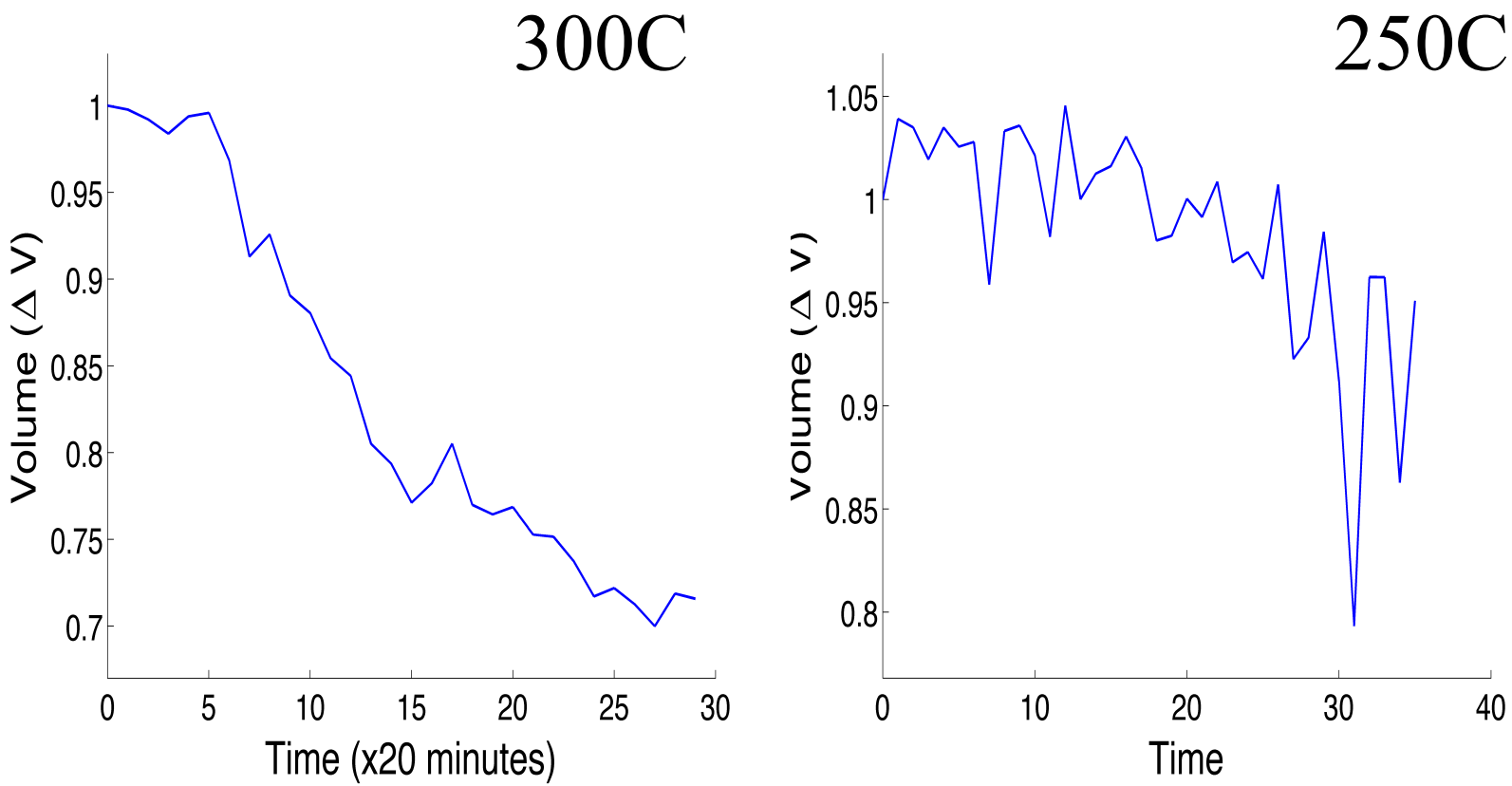


Amplitude cross section

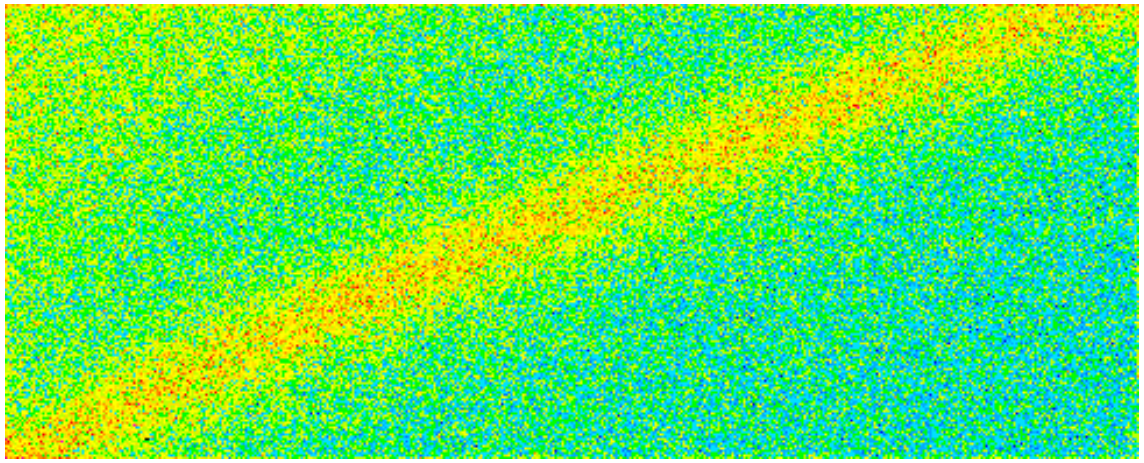
~20 frames, ~20 minutes per 3D dataset at I-07



Retraction of crystal volume during deposition

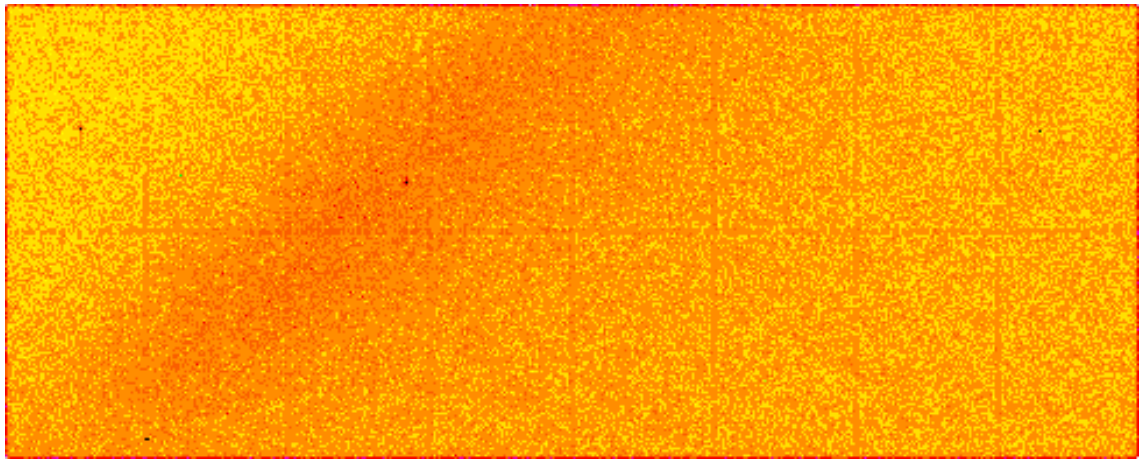


Post-mortem study of Alloy



Cu{111} ring
300C x 500min

Cu_3Au{100} ring



Imaging of Aluminium Epoxy Marine Coating (Bo Chen)

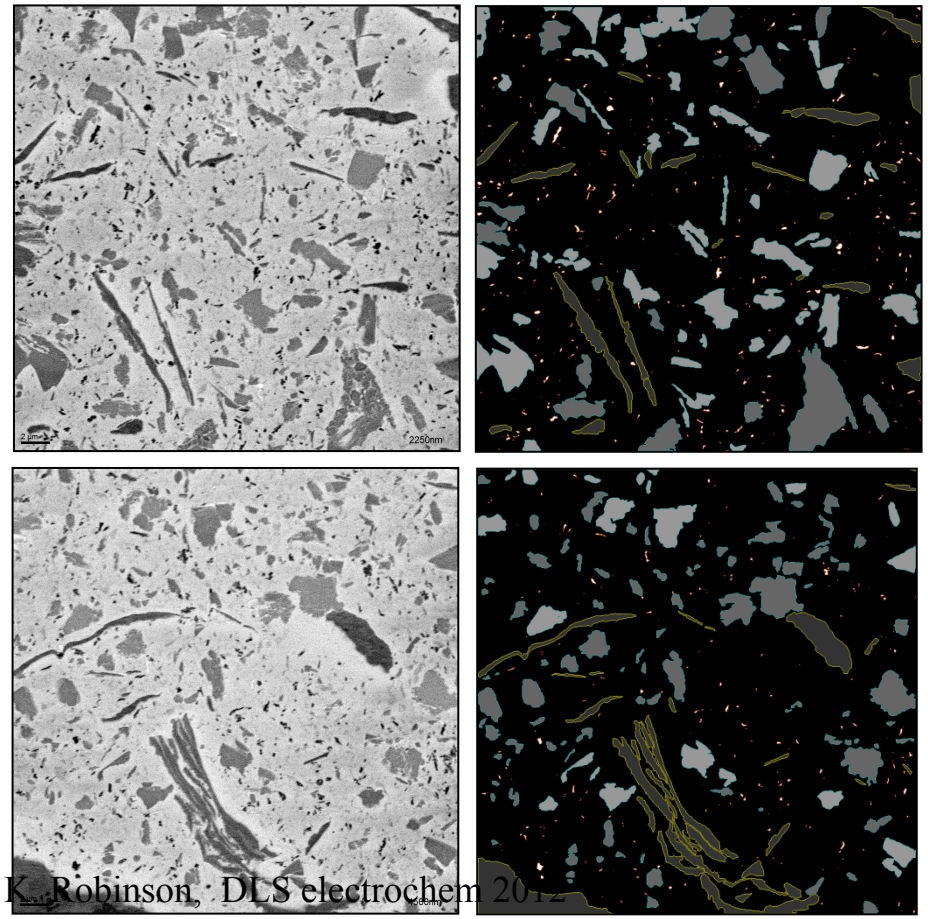
Serial Block Face Scanning Electron Microscopy (SBFSEM)

Size: 30*30 micron square;

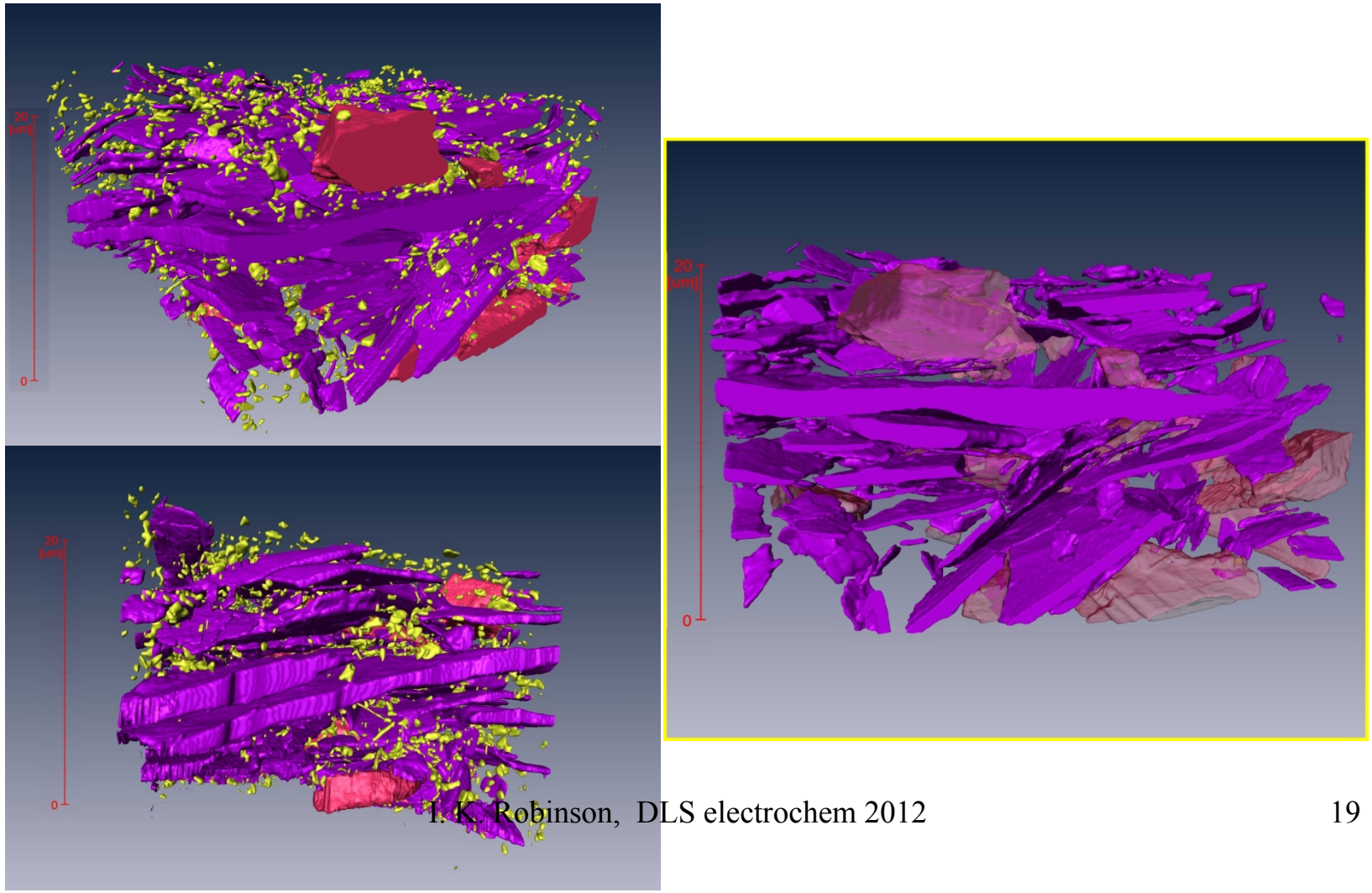
Pixel size: 30 nanometers;

Right: Original section slices;

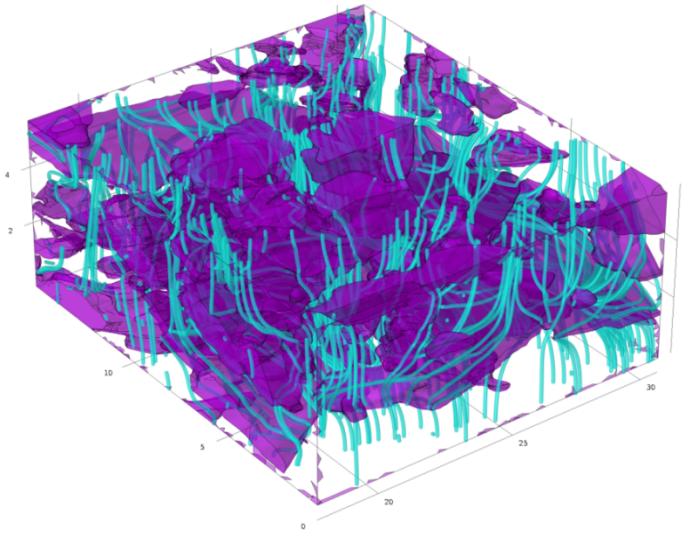
Left: Segmented section slices

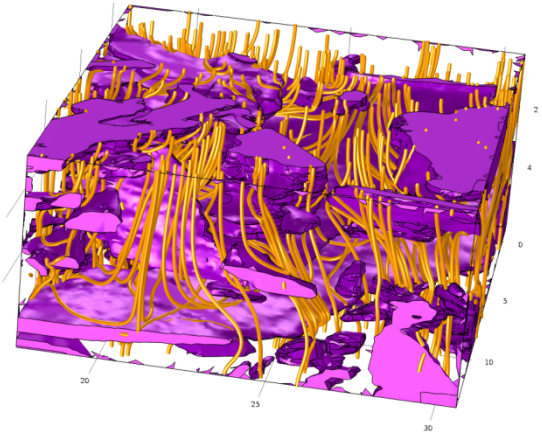


3D rendering of degassed coating



Results of electric conductivity simulation on SBFSEM results:





$E = \sigma_{perpendicular} / \sigma_{parallel}$ **Relative Errors**

0.516 **0.0079**

Results of electric conductivity simulation on X-ray results

$E = \sigma_{perpendicular} / \sigma_{parallel}$ **Relative Errors**

0.358 **0.0084**

Coherent x-ray diffraction (CXD)

- Complex density can image strain
- Strain associated with nano-shape
- Alloying penetrates from exterior
- Nano-porous transient structure
- Porous Al coating impedes corrosion
- Time resolved *in-situ* 3D imaging