WE START WITH YES.

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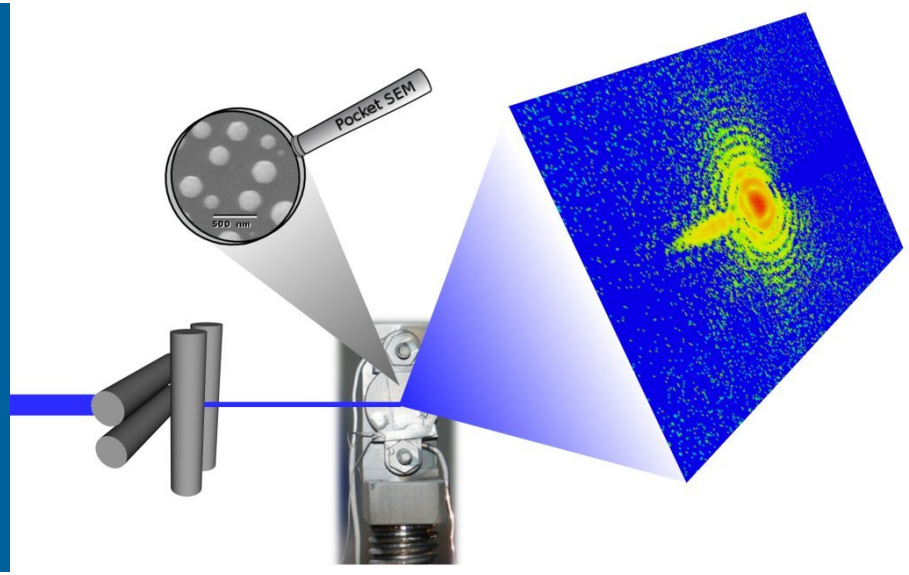
DEVELOPMENT OF IN-SITU BROADBAND LAUE MICRODIFFRACTION FOR DETERMINATION OF CRYSTALLOGRAPHIC ORIENTATION AT THE 34-ID-C BRAGG COHERENT DIFFRACTION IMAGING INSTRUMENT



DR. ROSS HARDER
Advanced Photon Source
Argonne National Laboratory

Beamline 34-ID-C

MULTI INSTITUTION PARTNERSHIP

PUP began in 2017 with LANL

Richard Sandberg, Brigham Young University

Saryu Fensin, Los Alamos National Laboratory

Reeju Pokharel, Los Alamos National Laboratory

Jonathan Gigax, Los Alamos National Laboratory

Ross Harder, Advanced Photon Source

Wonsuk Cha, Advanced Photon Source

Jon Tischler, Advanced Photon Source

Wenjun Liu, Advanced Photon Source

Tony Rollett, Carnegie Mellon University

Anastasios Pateras, Carnegie Mellon University

Matthew Wilkin, Carnegie Mellon University

Yueheng Zhang, Carnegie Mellon University

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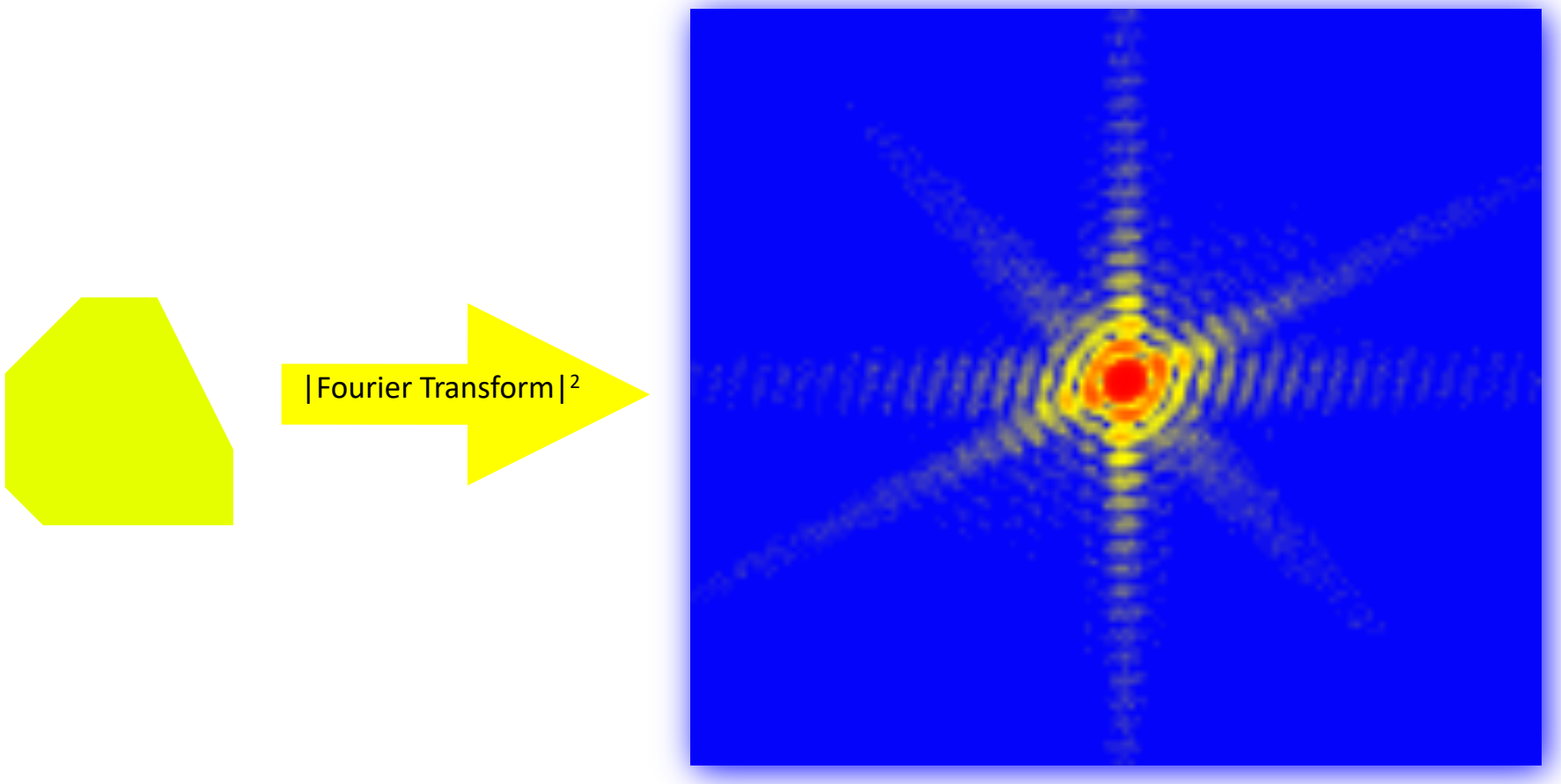
Los Alamos NATIONAL LABORATORY

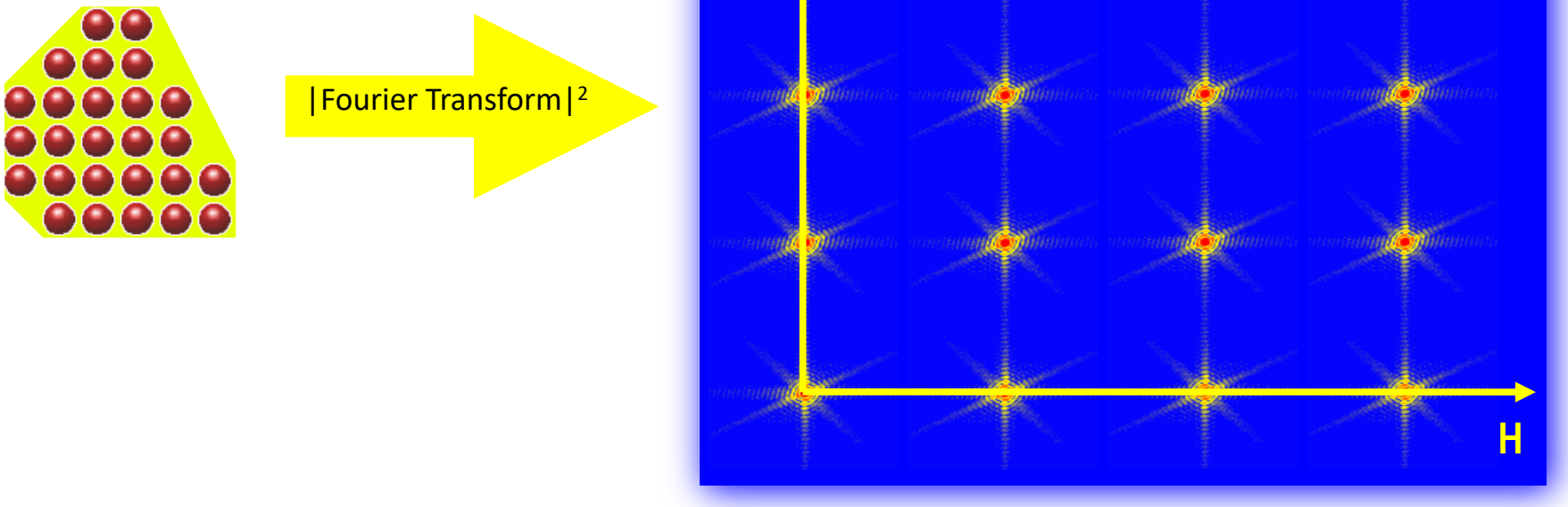
Carnegie Mellon University

BRIGHAM YOUNG UNIVERSITY FOUNDED BYU 1875 PROVO, UTAH

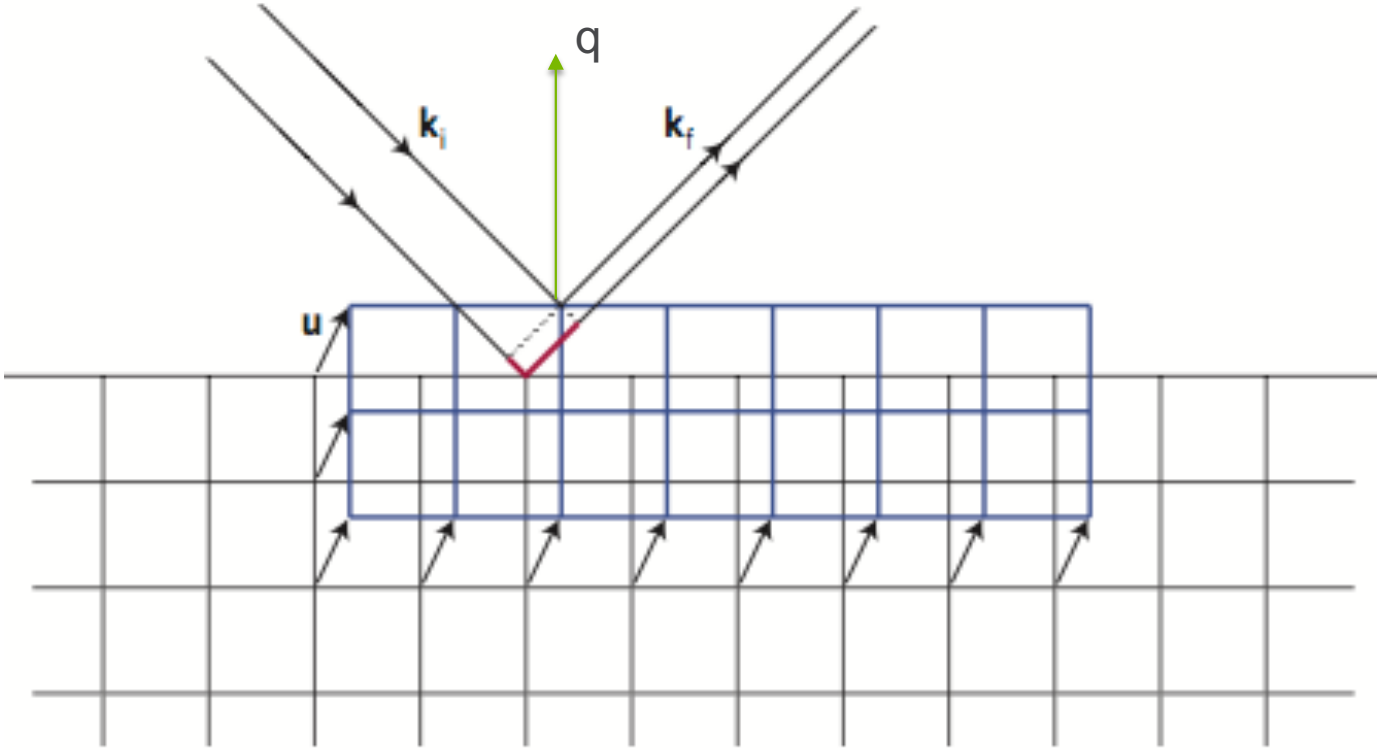
Coherent Diffraction from Crystals



Coherent Diffraction from Crystals



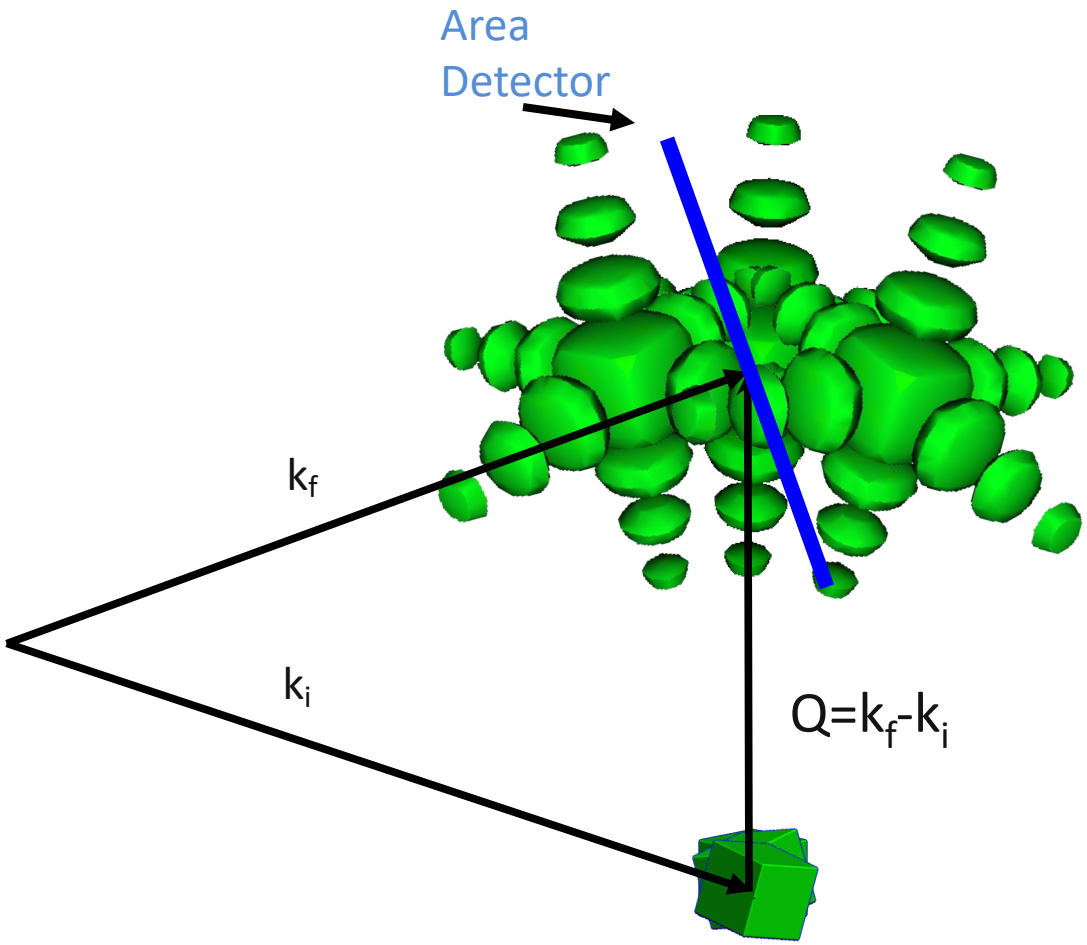
STRUCTURAL SENSITIVITY WITH BRAGG CDI



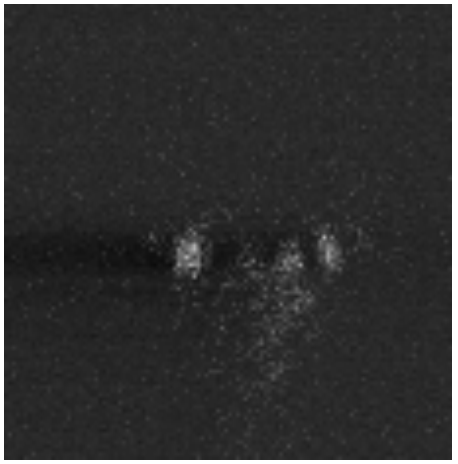
Measured intensity affected by atomic displacement fields with picometer sensitivity

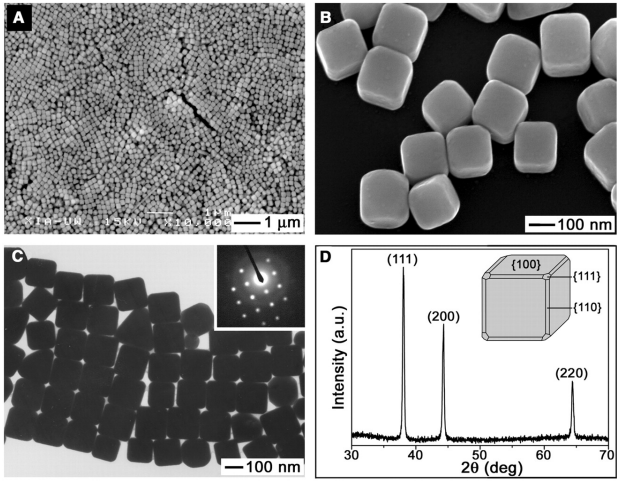
Robinson & Harder, Nat. Mater., 8 (291), 2009

Measuring 3D CXD



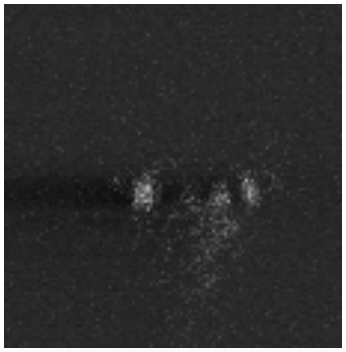
Silver Nano Cube (111)

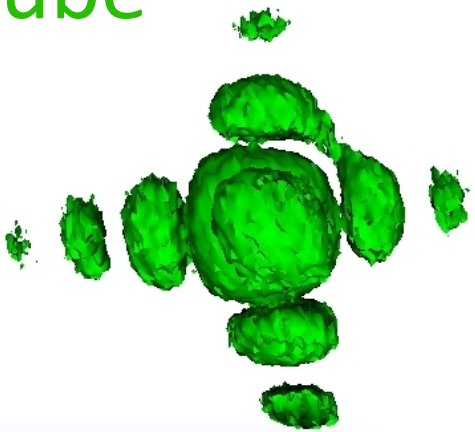


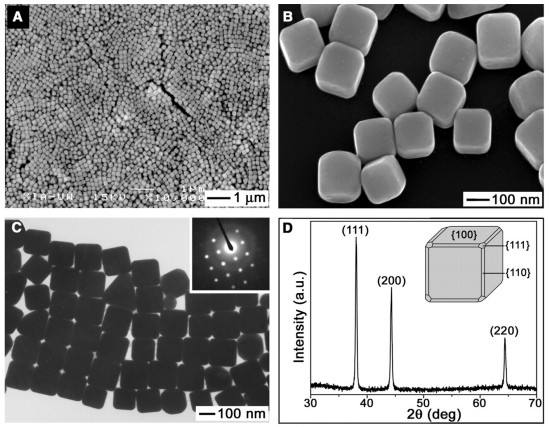


Yugang Sun and Younan Xia, Science 298 2177 (2003)

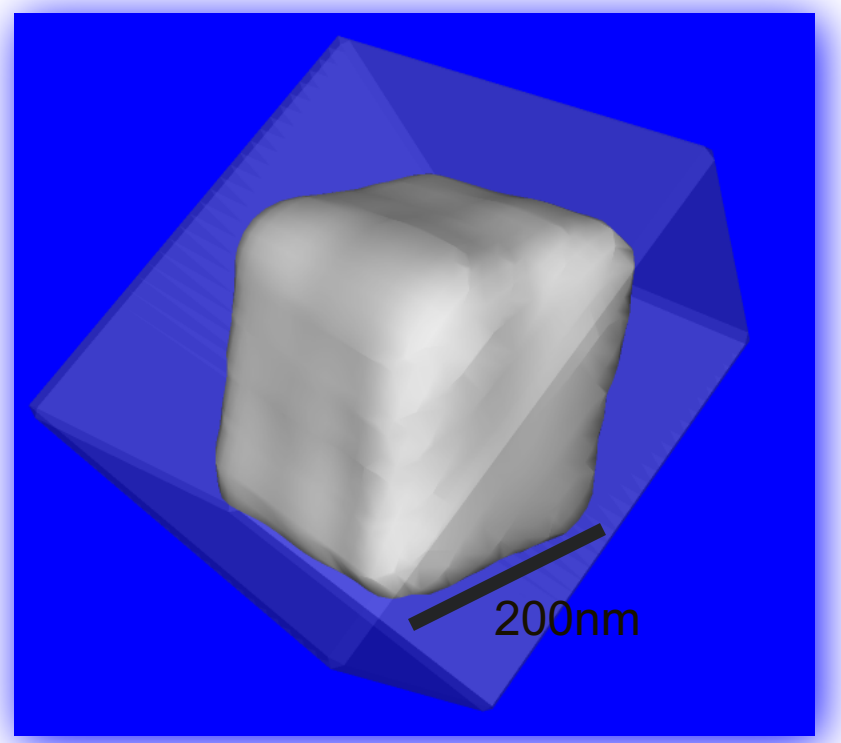
3D Ag Nano Cube

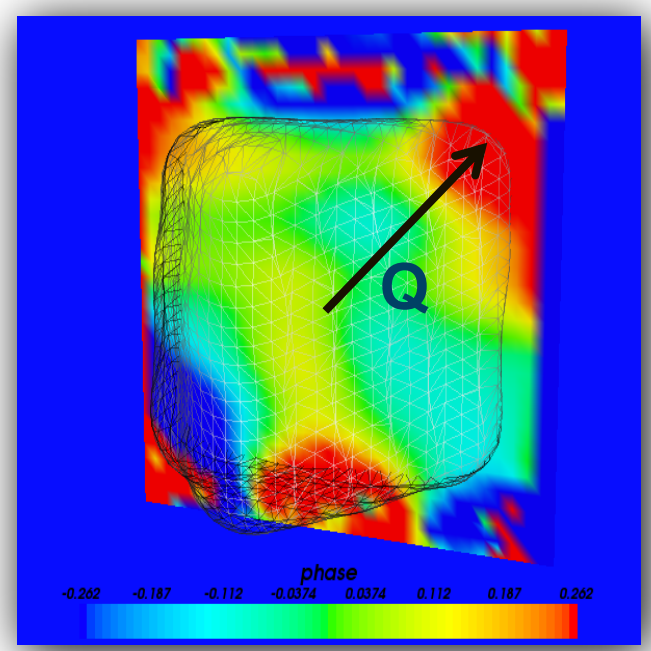






Yugang Sun and Younan Xia, Science 298 2177 (2003)





Harder, R., et al. (2010). *New Journal of Physics*. **12**, 035019.

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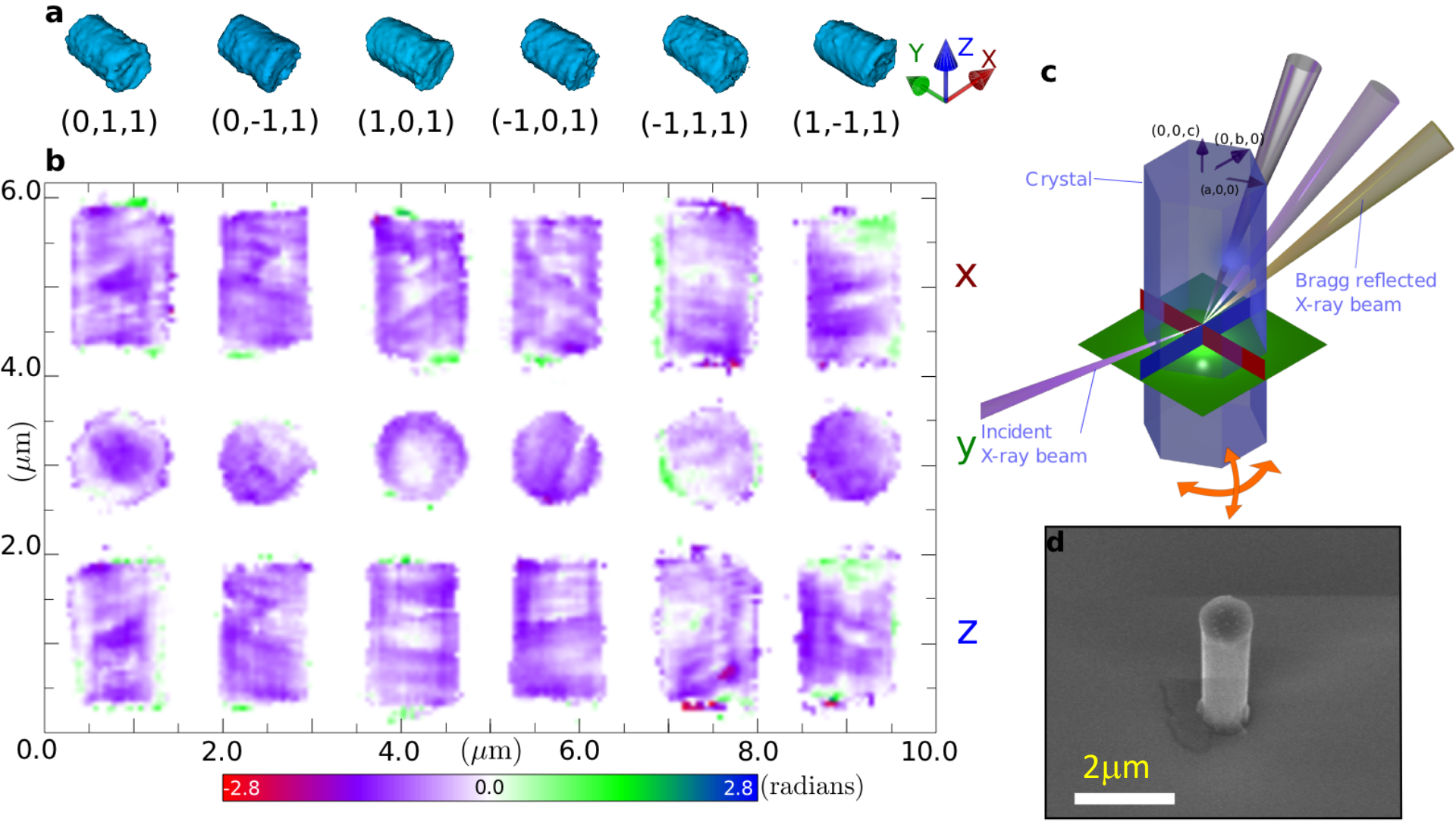
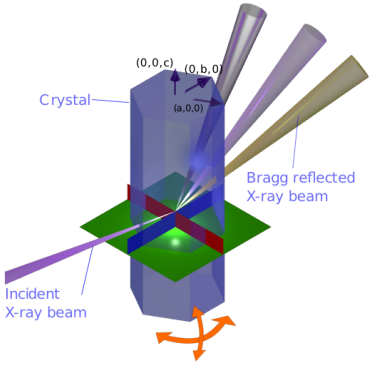
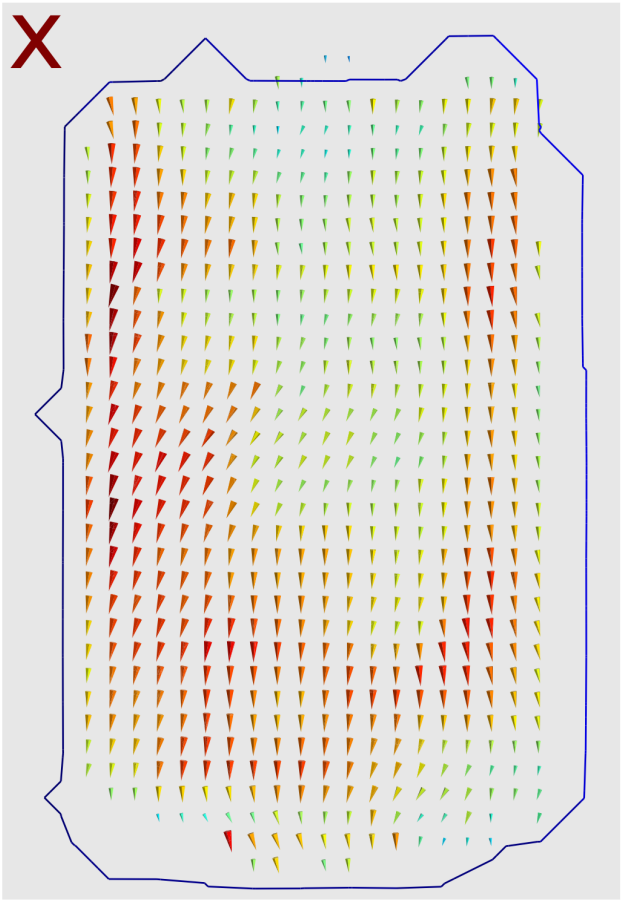
3D Strain Map in ZnO



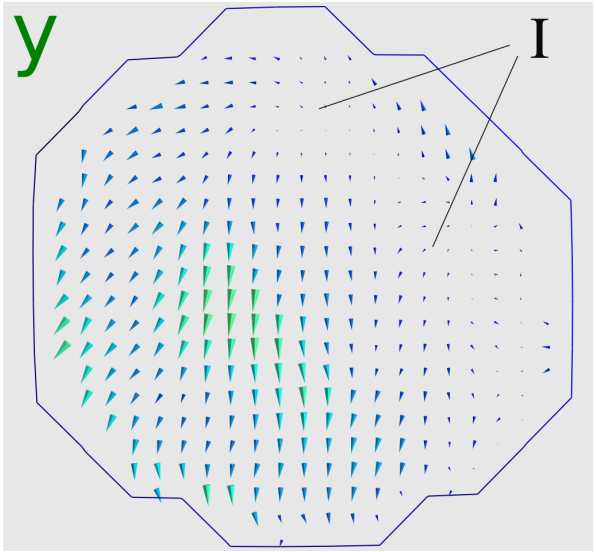
Nature Materials ***9, 120 - 124 (2010)***

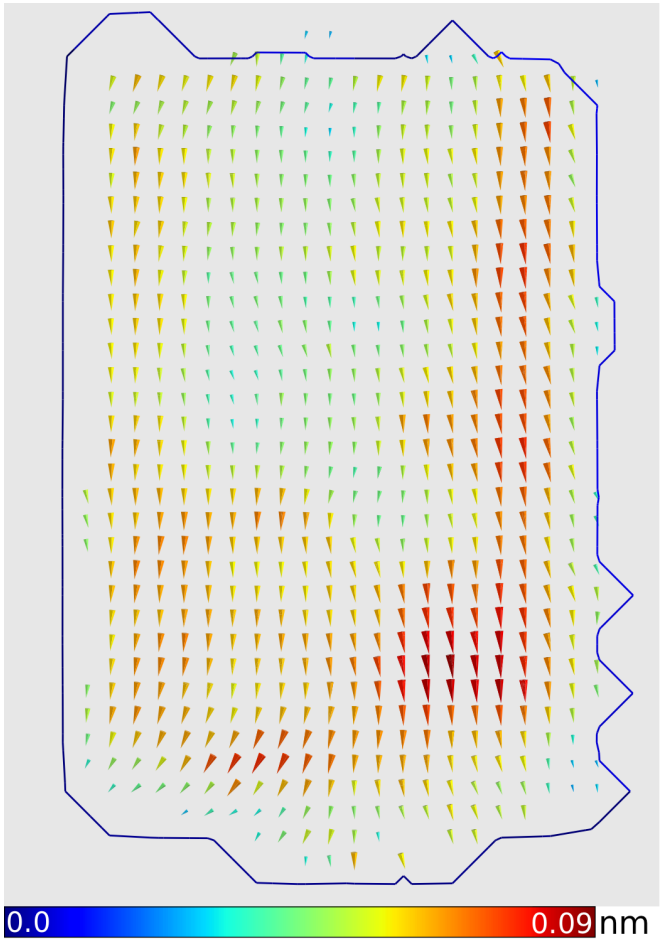
3D Strain Map in ZnO





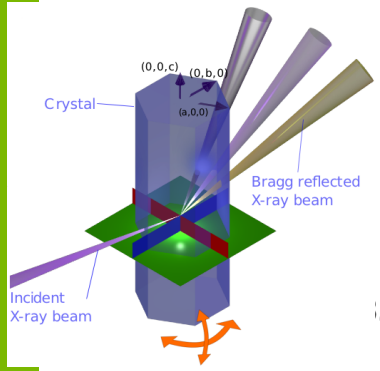
$$u_j = \xi_{ji} q_{ki} \phi_k; \qquad \xi_{ji} = (q_{kj} q_{ki})^{-1}$$



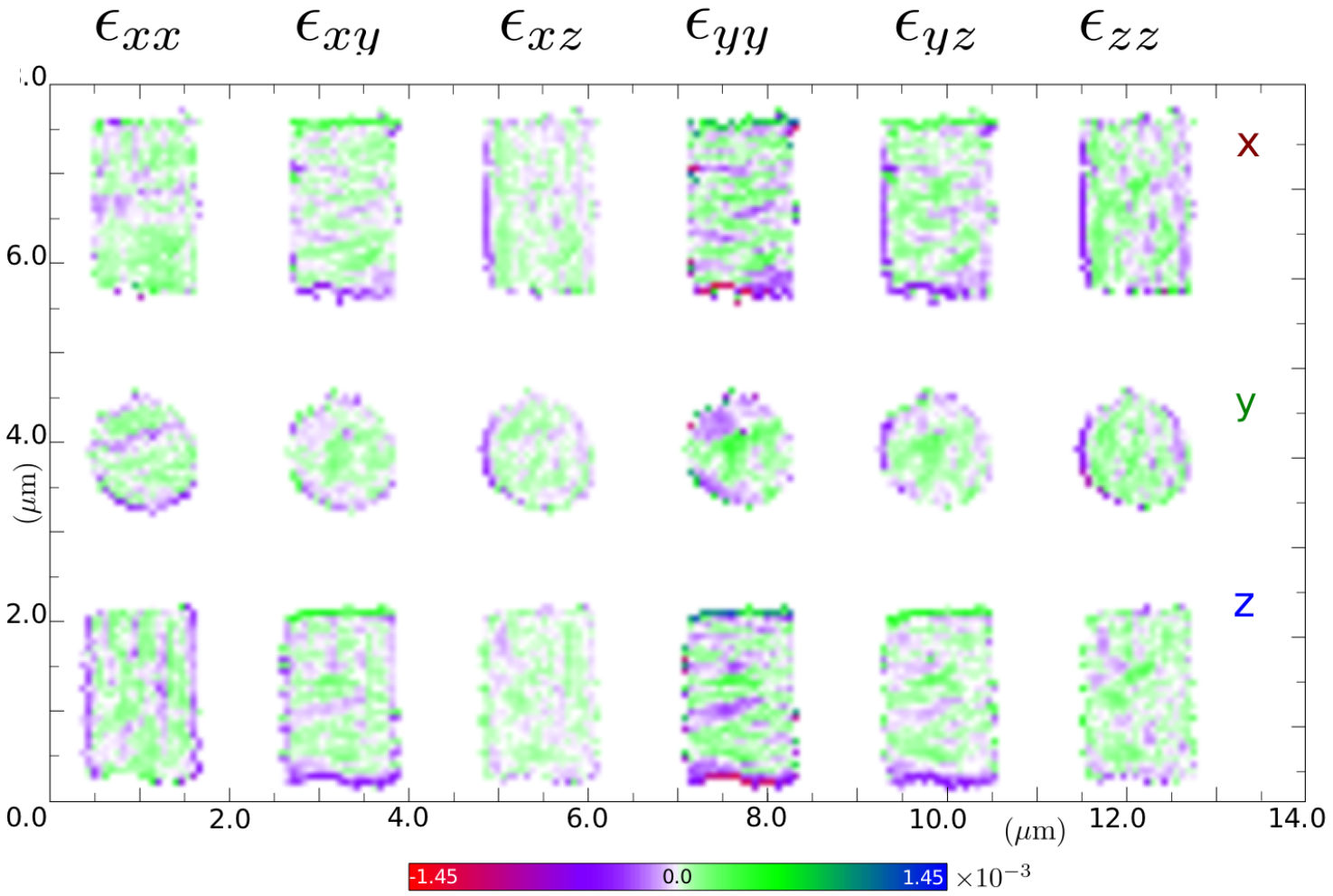


Nature Materials ***9, 120 - 124 (2010)***

3D Strain Map in ZnO

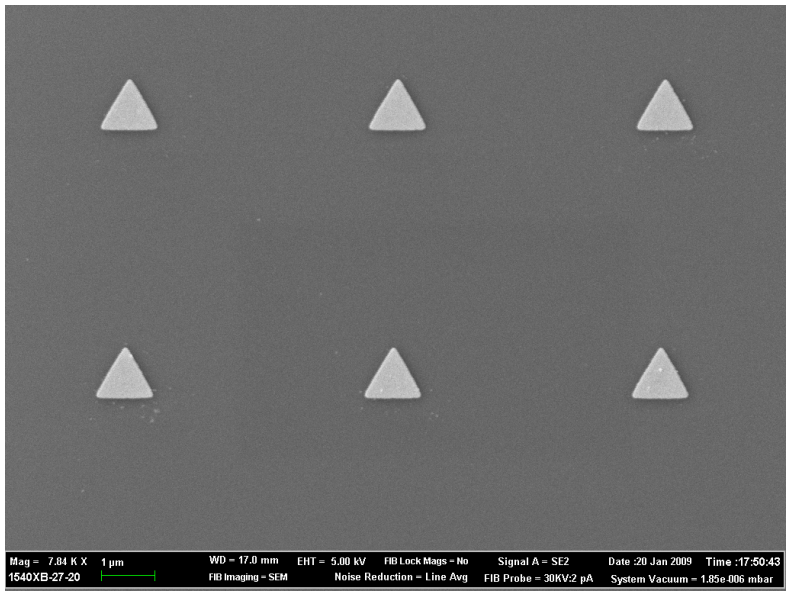


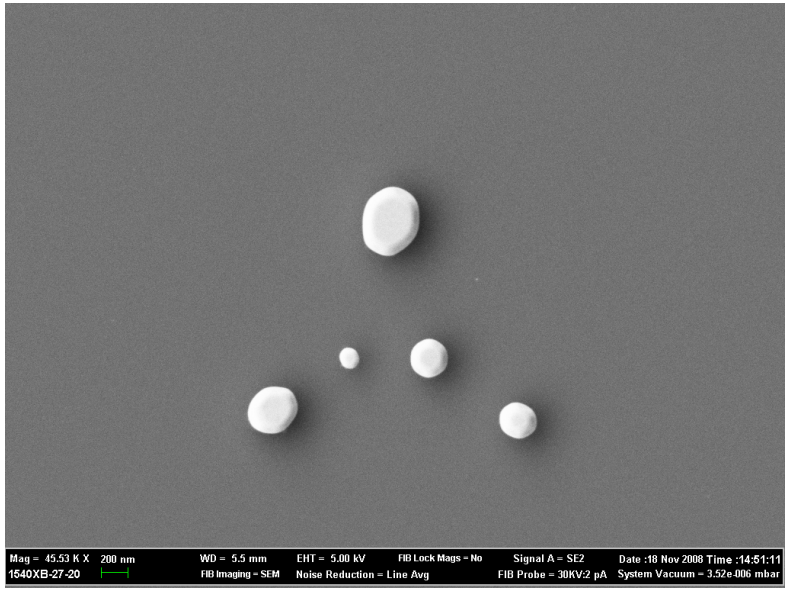
$$\epsilon_{ij} = \frac{1}{2}\left(\frac{\partial u_j}{\partial x_i} + \frac{\partial u_i}{\partial x_j}\right), \qquad \tau_{ij} = \left(\frac{\partial u_j}{\partial x_i} - \frac{\partial u_i}{\partial x_j}\right)$$

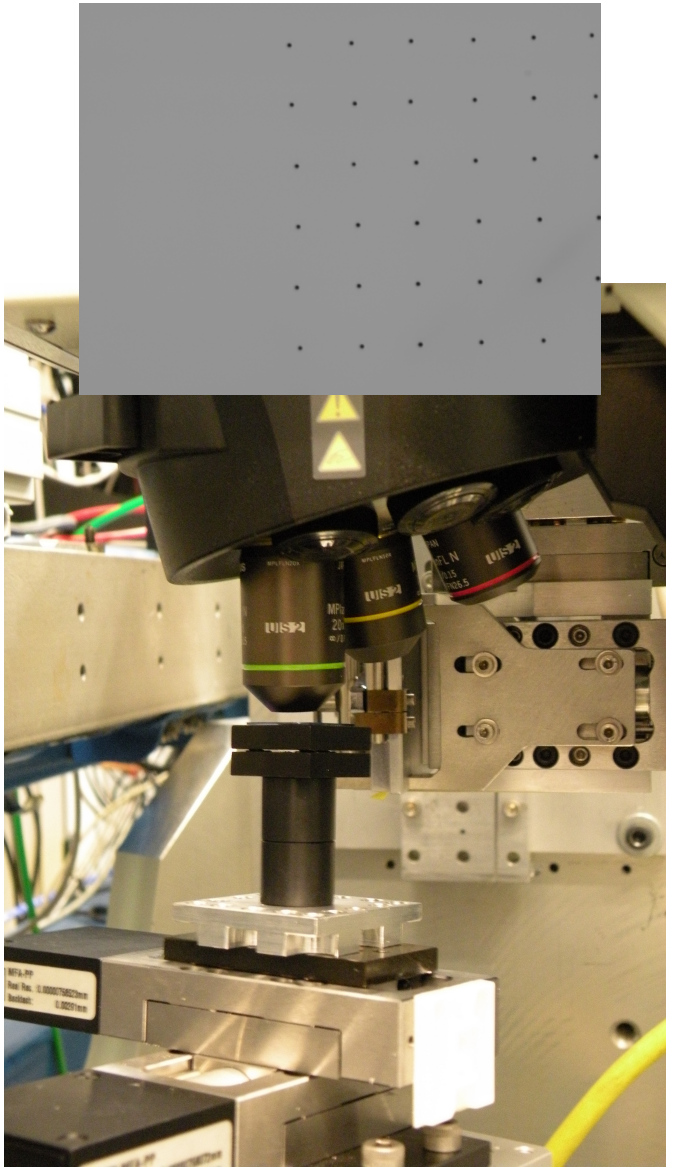


Nature Materials ***9, 120 - 124 (2010)***

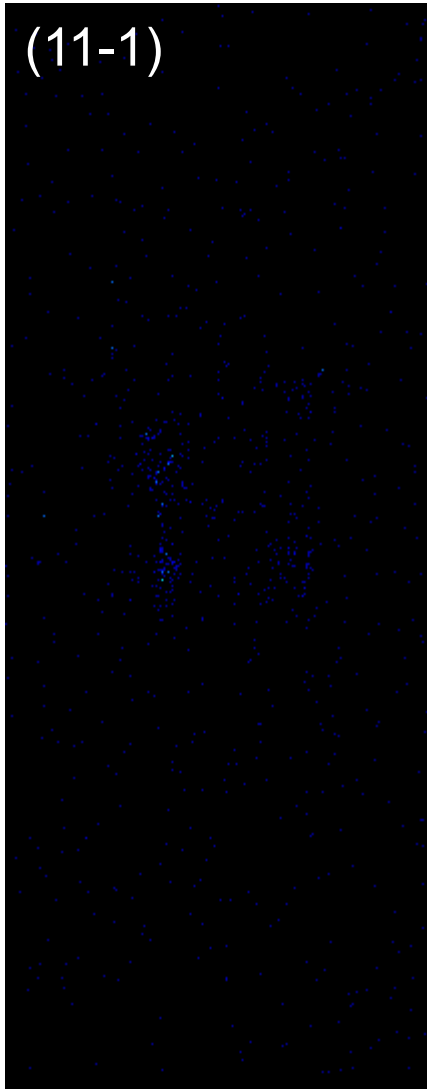
Patterned Gold Nanocystal Samples

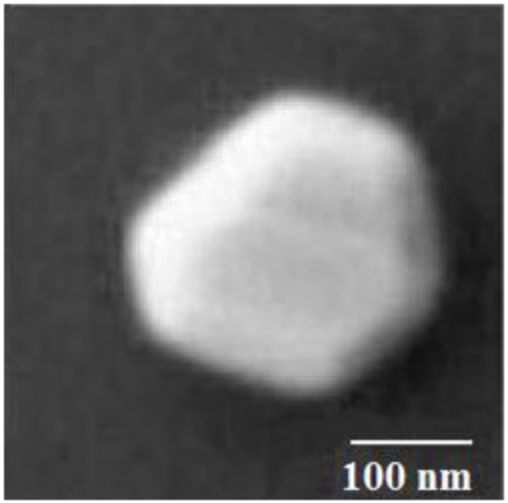


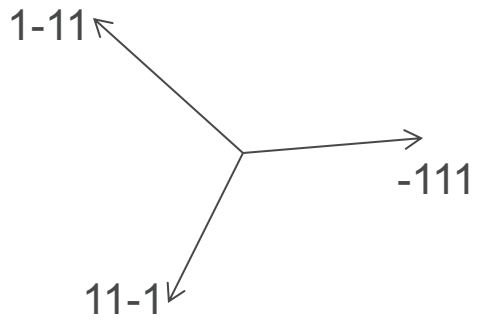




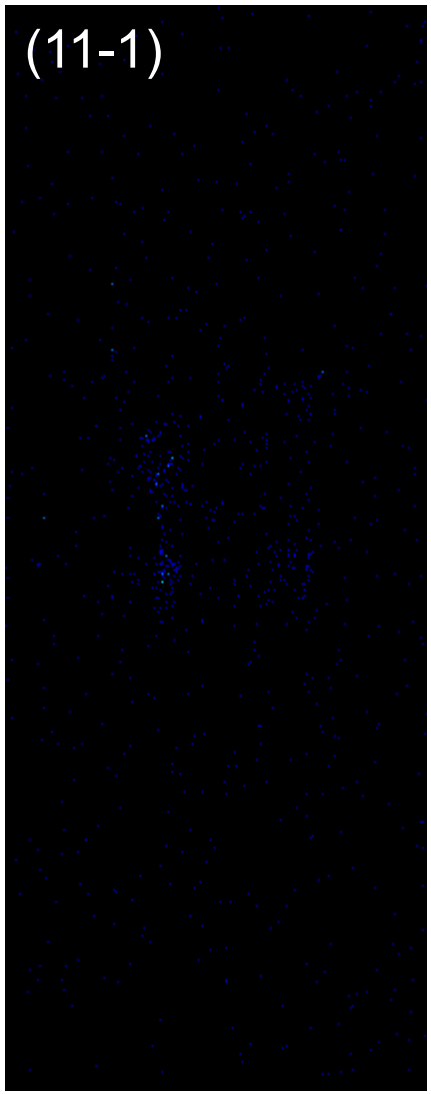
Multiple reflection reconstructions

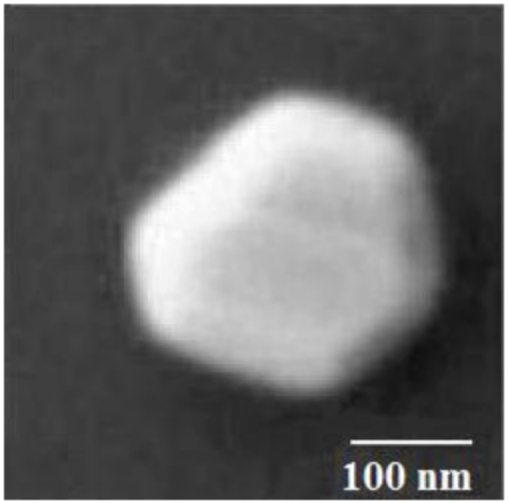


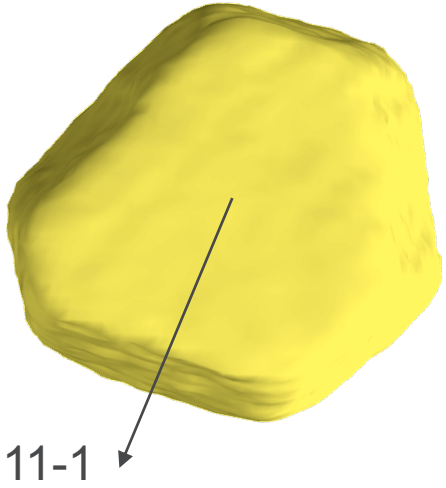


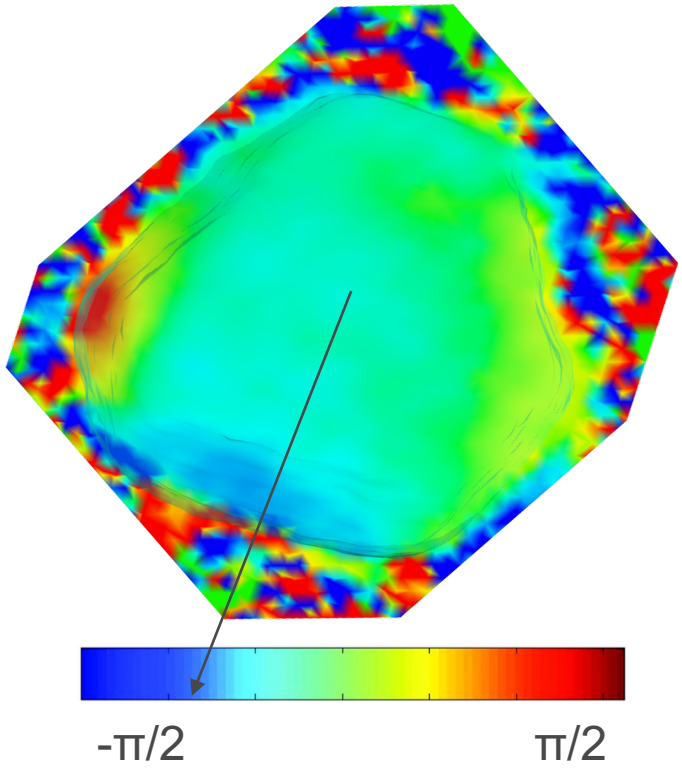


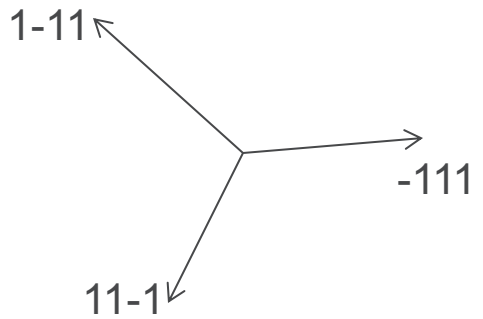
Multiple reflection reconstructions



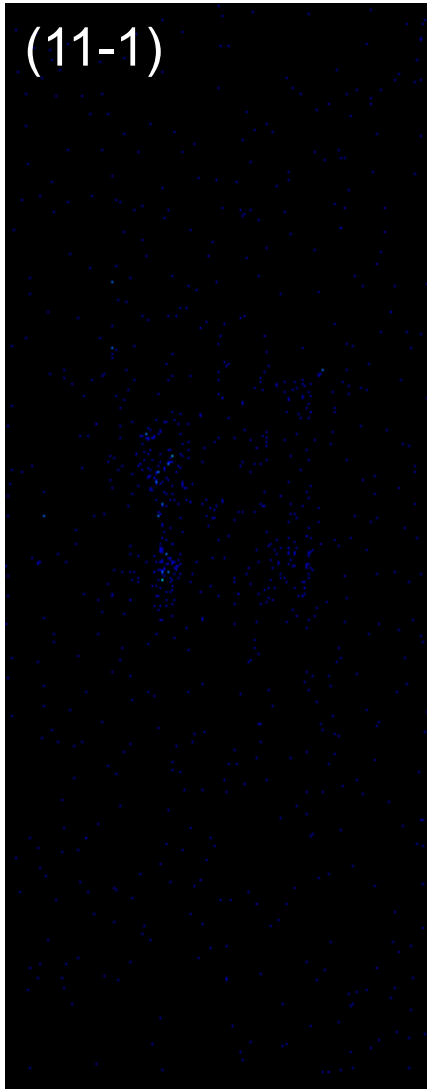


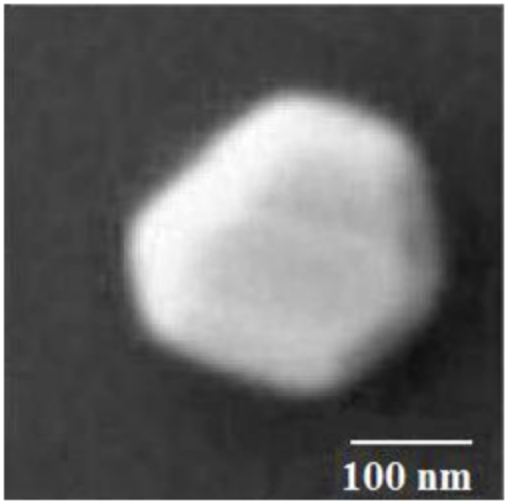


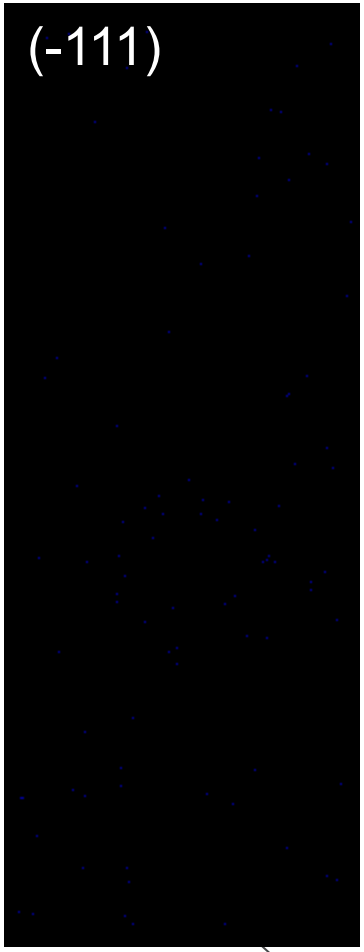


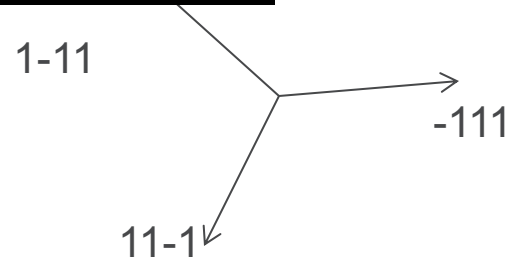


Multiple reflection reconstructions

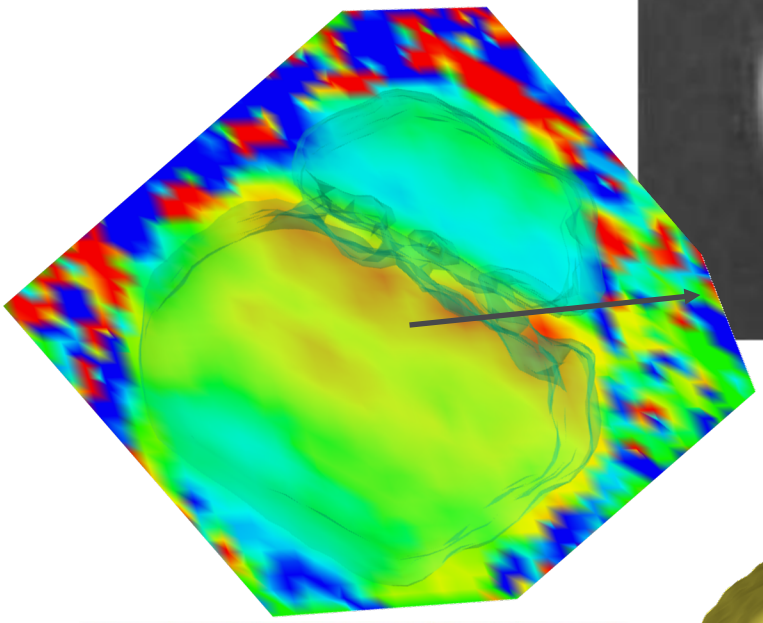


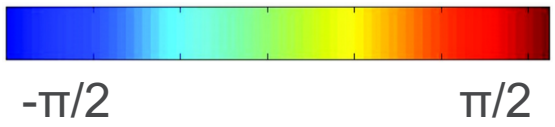


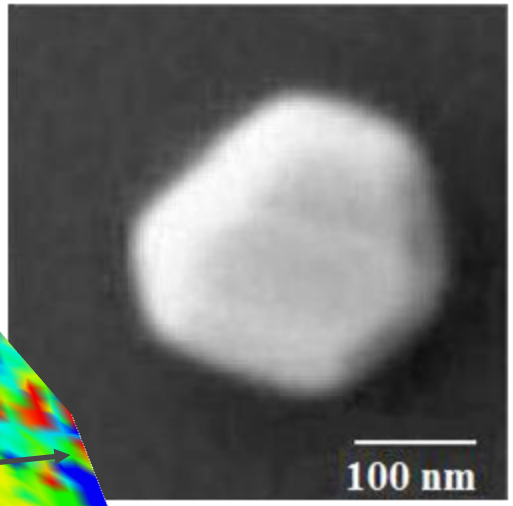


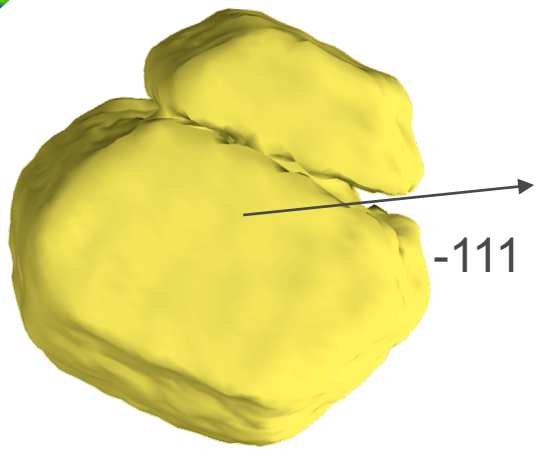


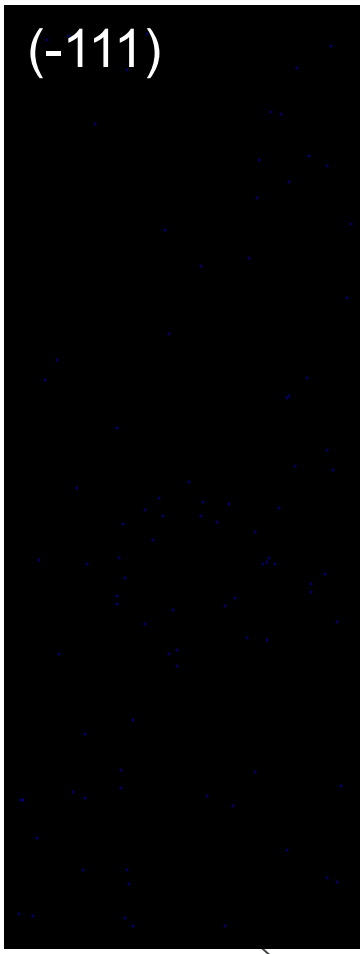
Multiple reflection reconstructions

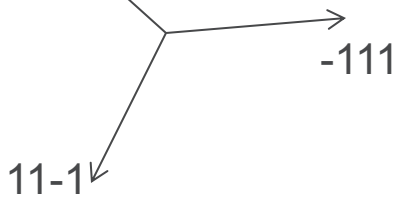




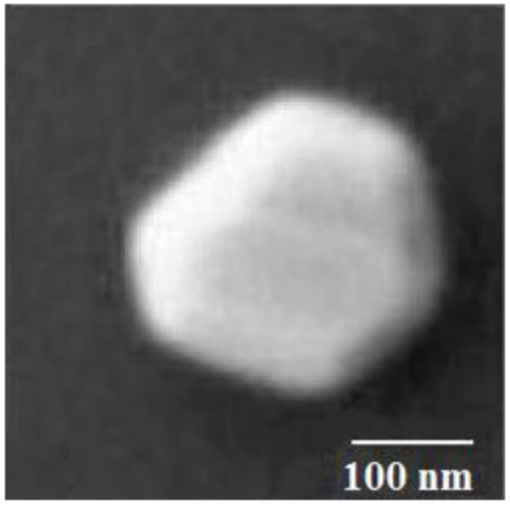




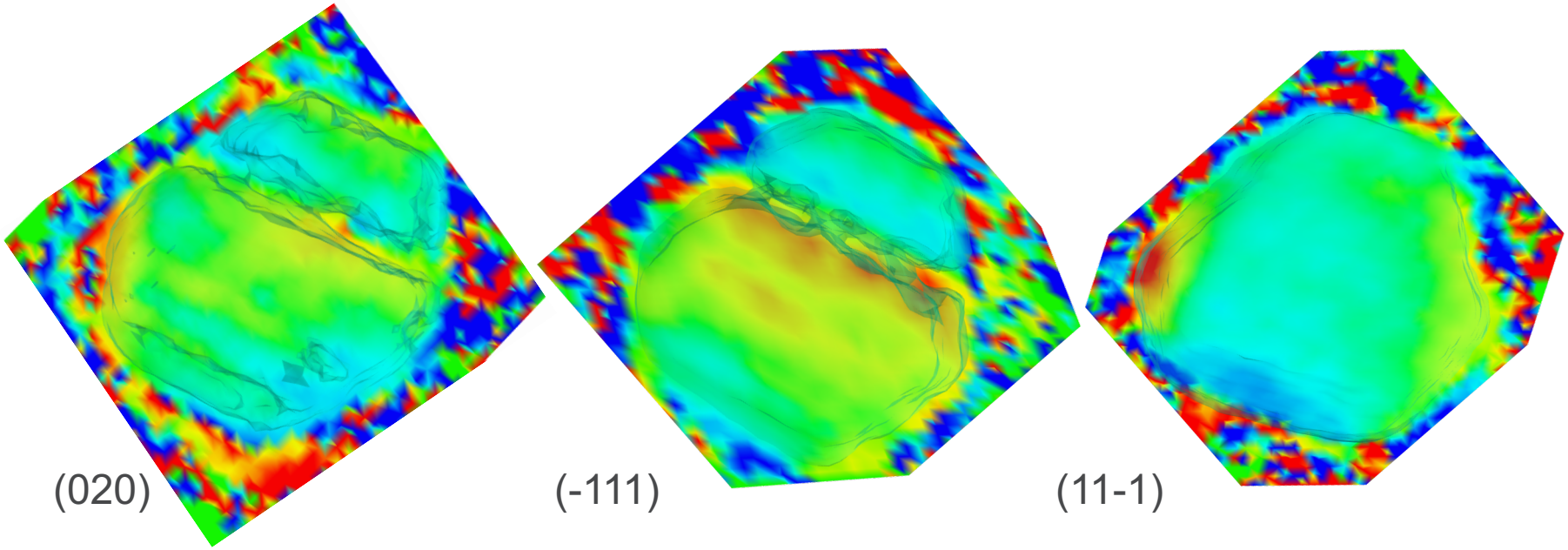




Multiple reflection reconstructions

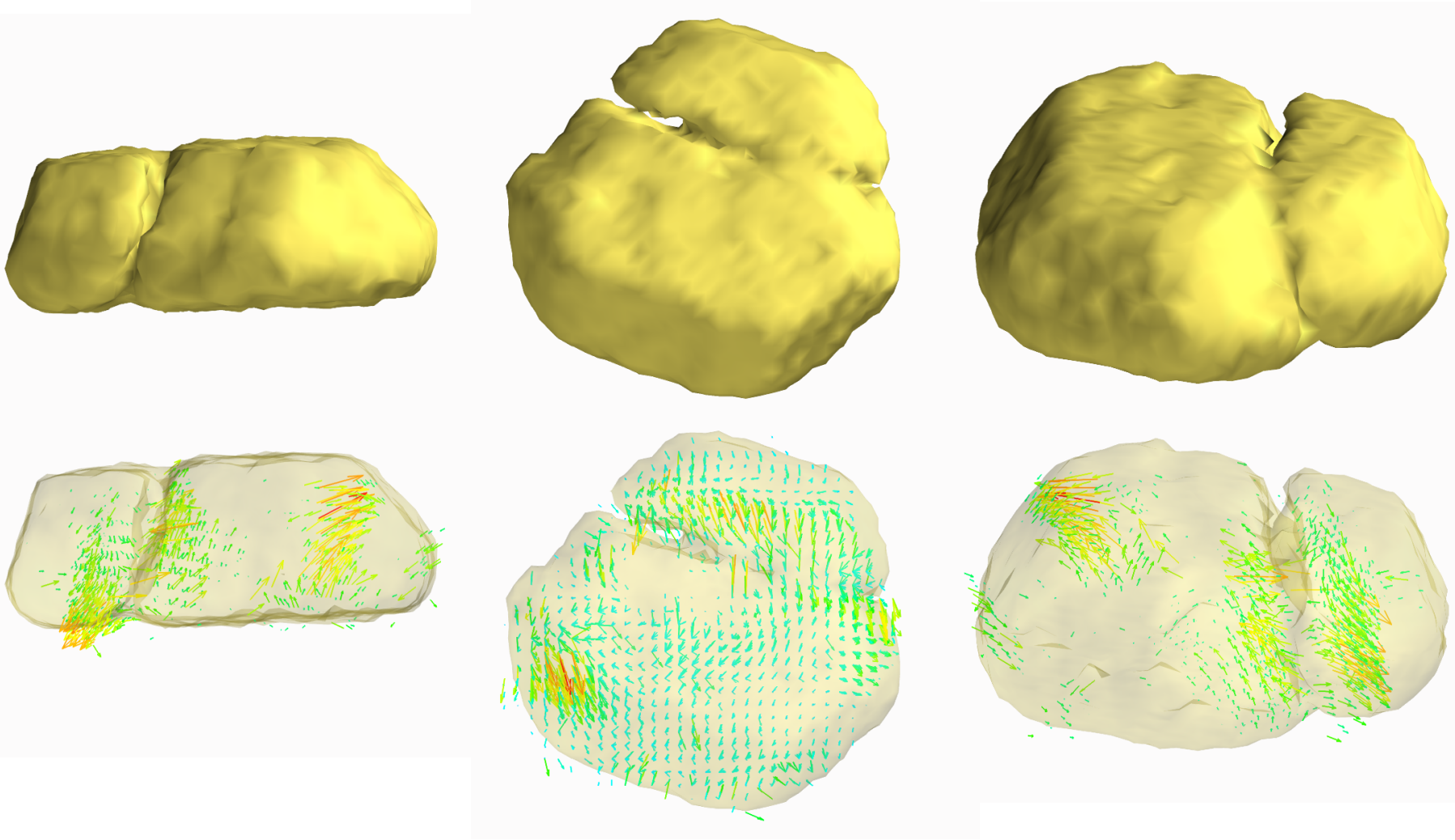


$$\varphi = q.u(r)$$



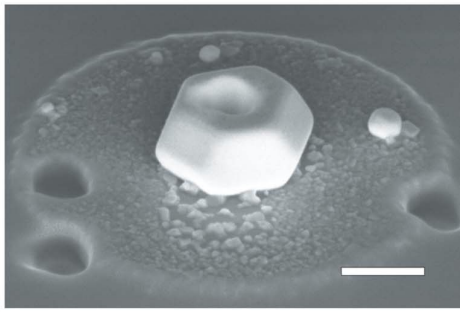
(020) (-111) (11-1)

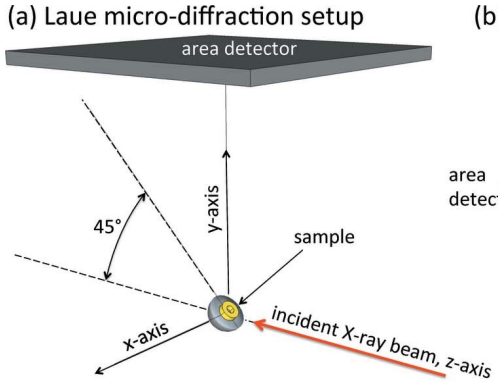
Vector Displacement Field of Gold lattice

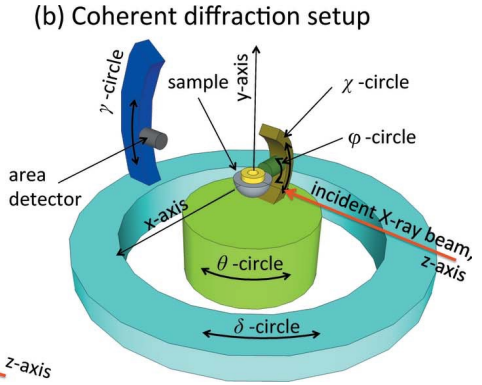


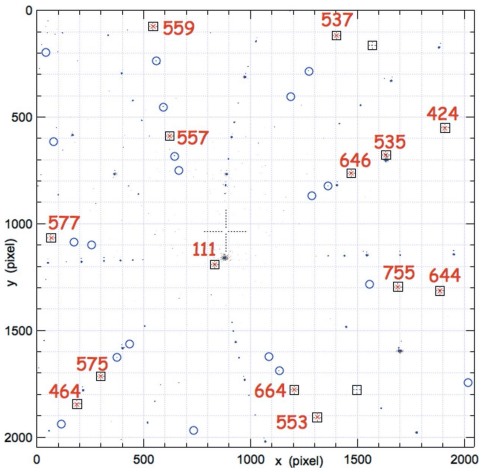
Produced by combining images from (11-1) (020) (-111)

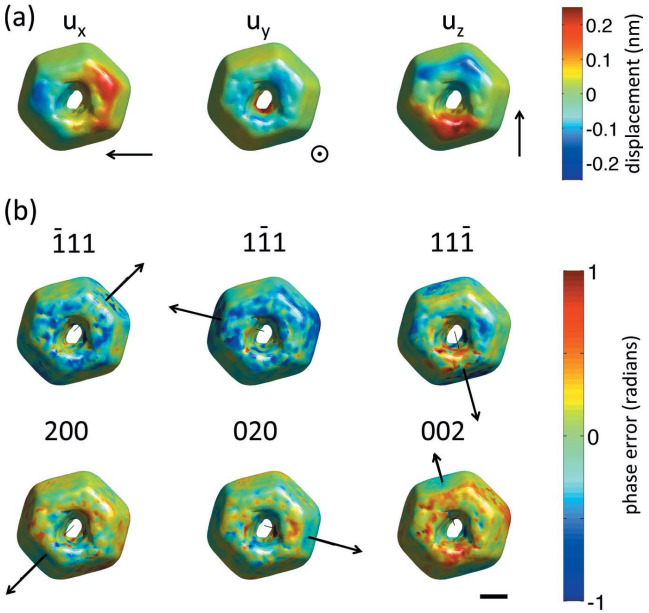
LAUE @ 34-ID-E
BCDI @ 34-ID-C

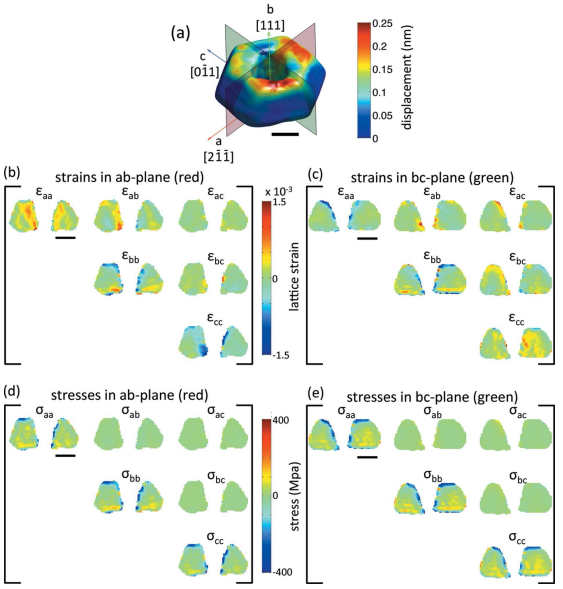










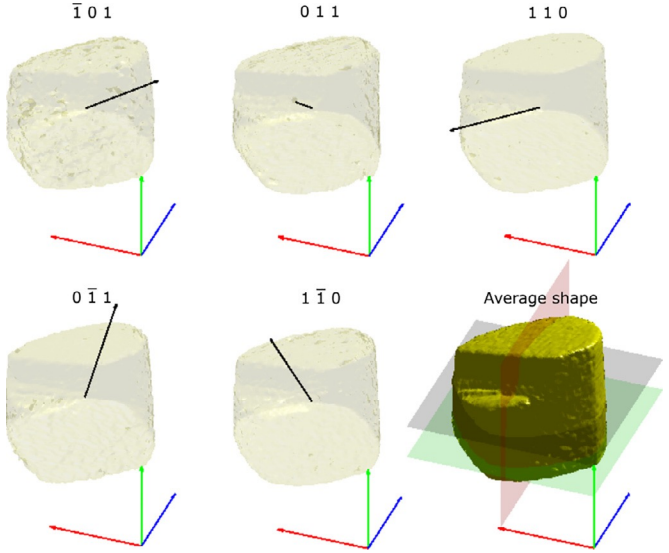


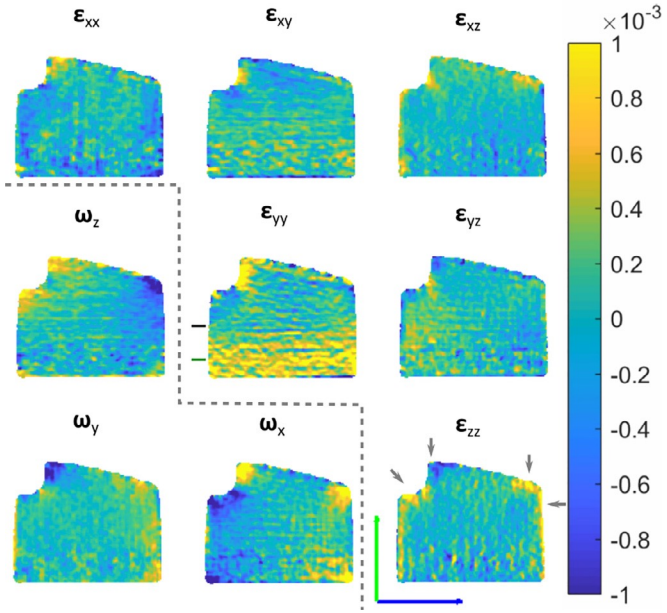
reciprocal lattice vectors:
a* b* c*
-6.773459 +0.130656 -9.377983
+1.778382 +11.375979 -1.125984
+9.208746 -2.100812 -6.680493

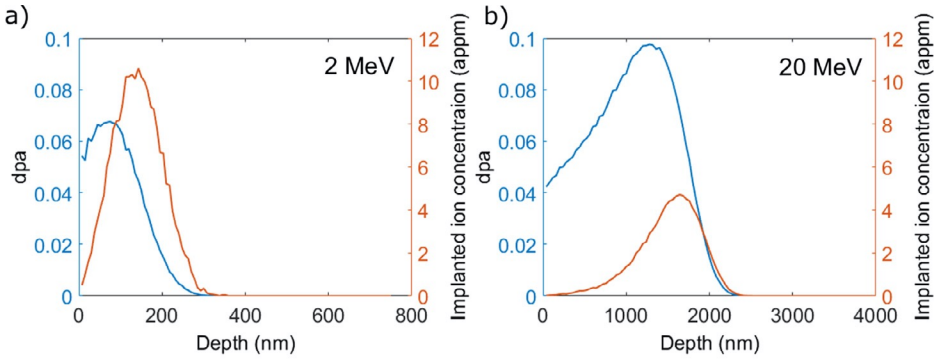
Hofmann, F., Phillips, N. W., Harder, R. J., Liu, W., Clark, J. N., Robinson, I. K., Abbey, B.IUCr (2017). *J. Synchrotron Rad.* **24**, 1048–1055.

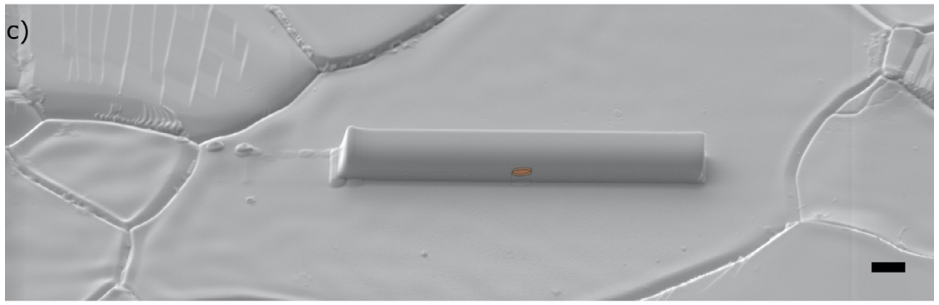
Nanoscale lattice strains in self-ion implanted tungsten

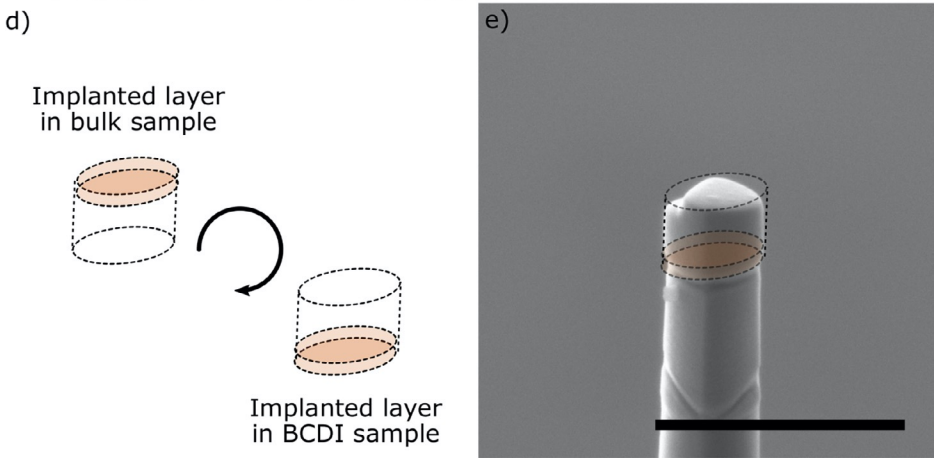
Phillips, N. W., et al. (2020). *Acta Materialia*. **195**, 219–228.











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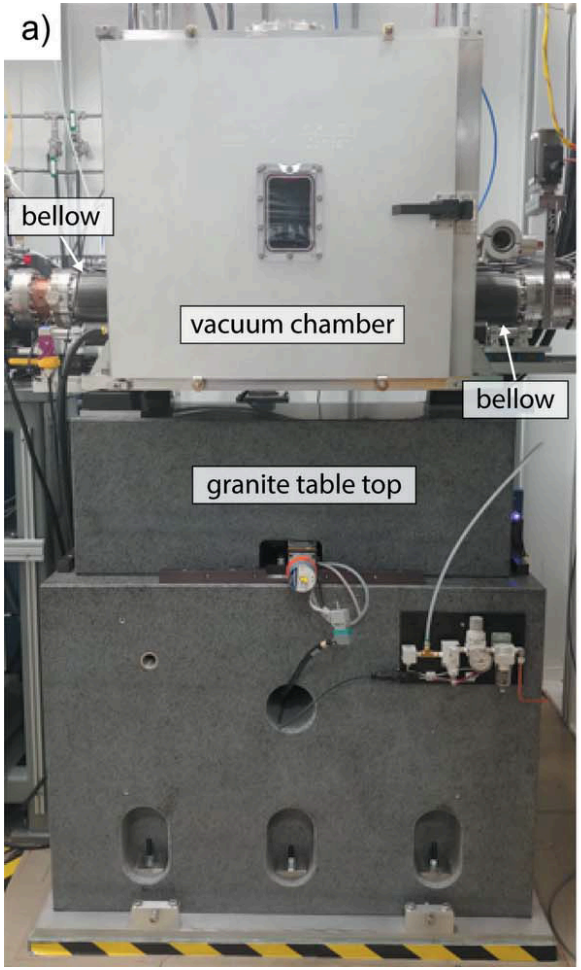
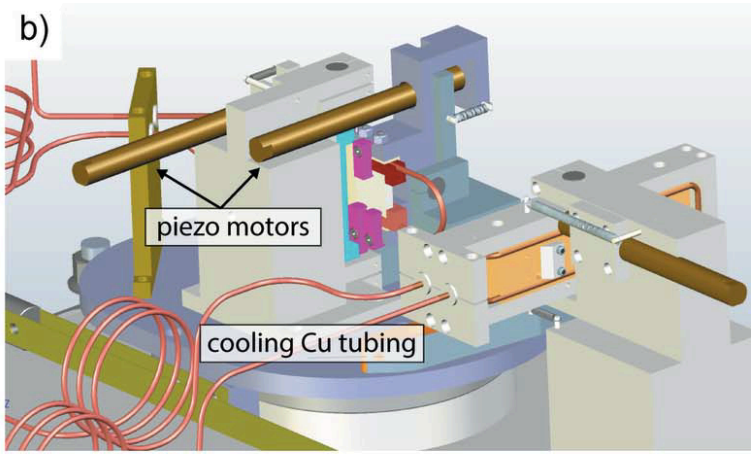
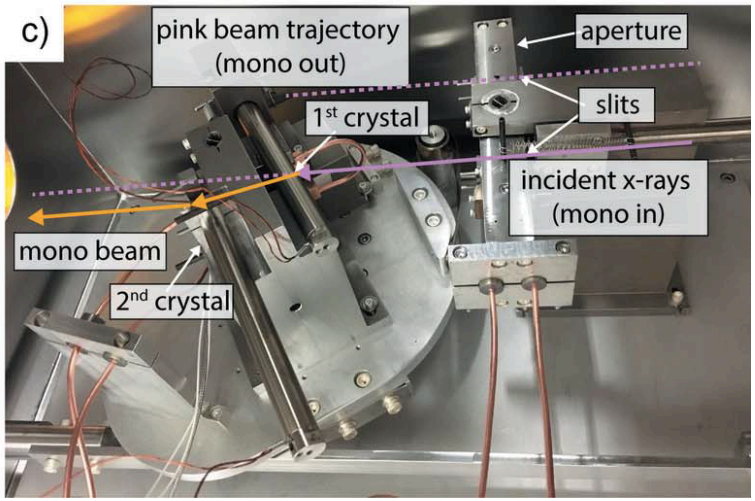
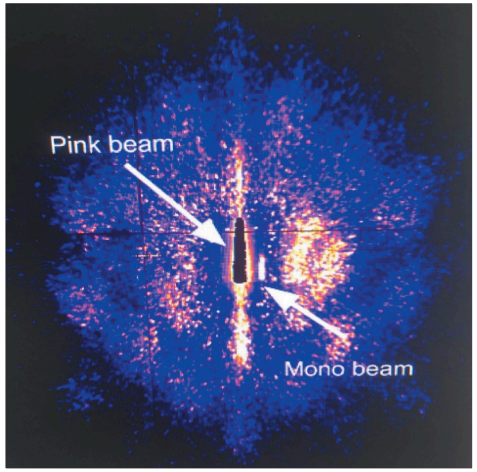
Commissioning of a movable double-bounce monochromator at 34-ID-C



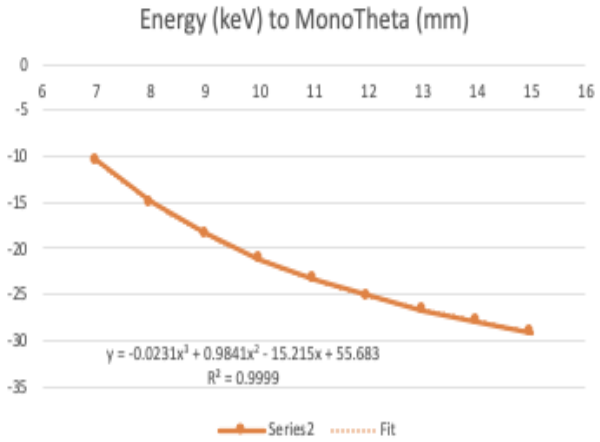


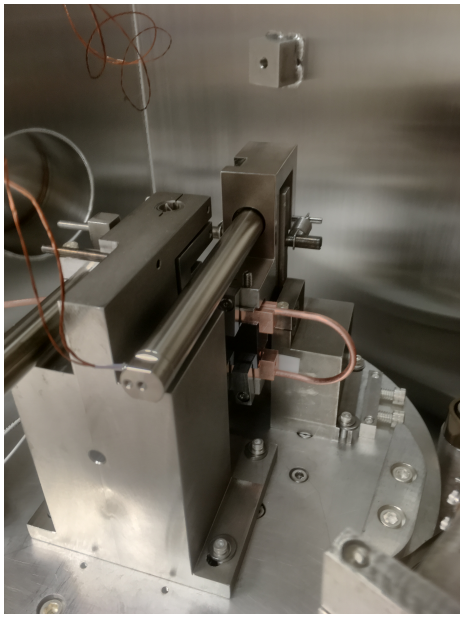




Energy Calibration

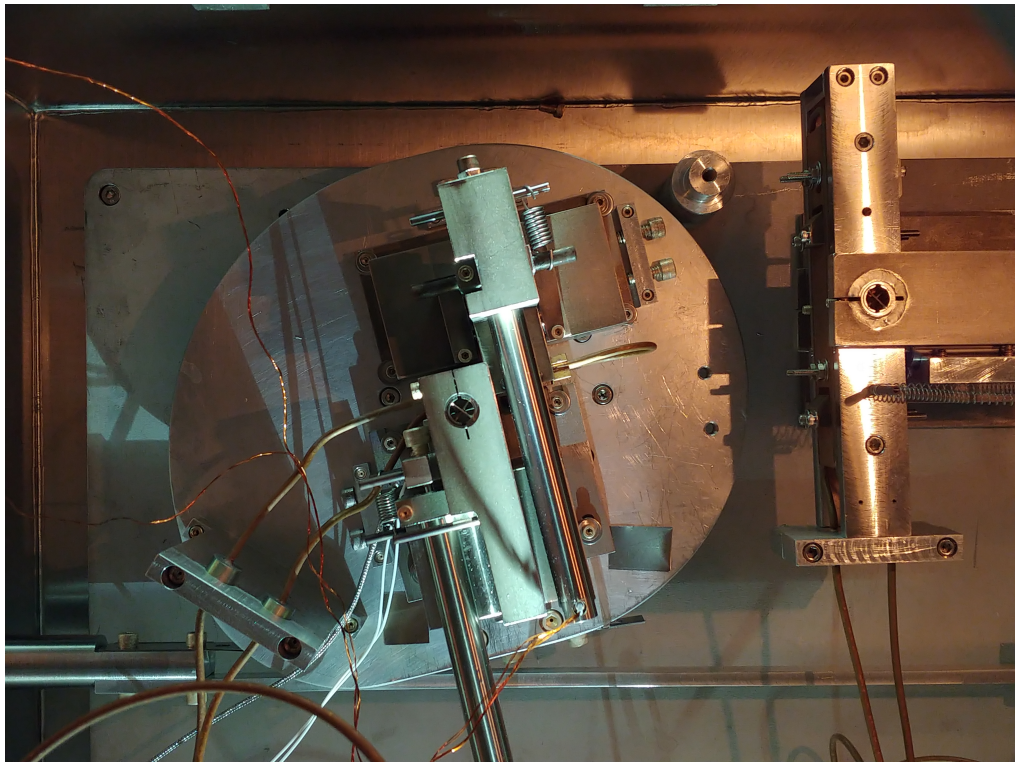
Energy (keV)	mcr (degree)	MonoTable (mm)
7	16.407	-10.4625
8	14.309	-15.0225
9	12.691	-18.4238
10	11.403	-21.1275
11	10.355	-23.315
12	9.4835	-25.1388
13	8.748	-26.665
14	8.1188	-27.965
15	7.5743	-29.1275





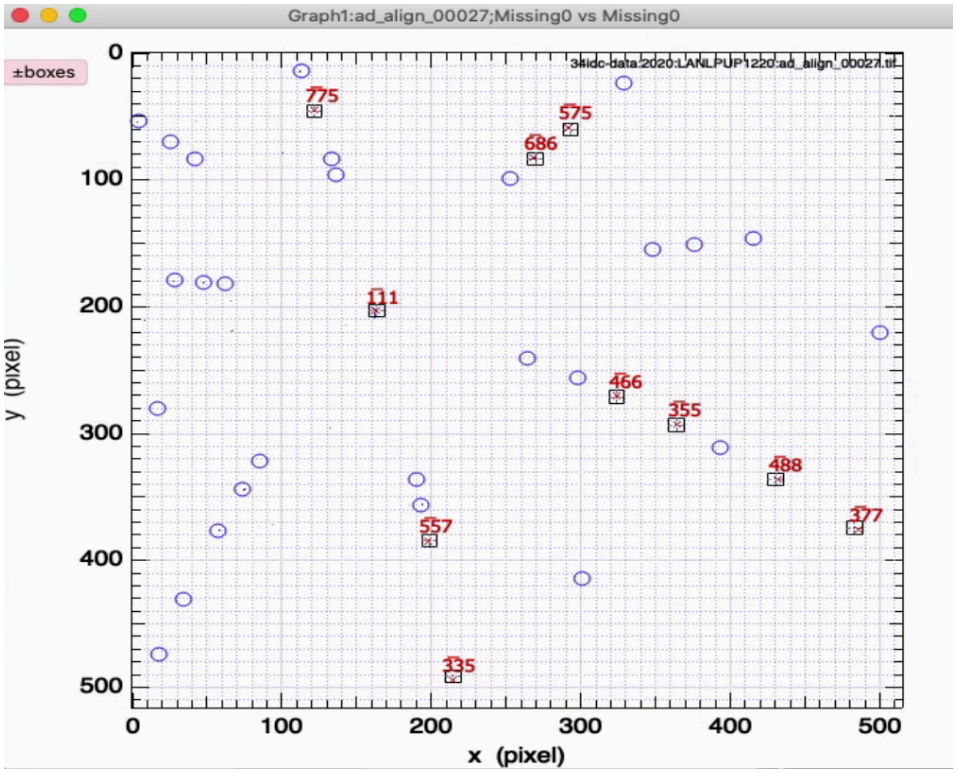
y=-0.0231x^3+0.9841x^2-15.215x+55.683

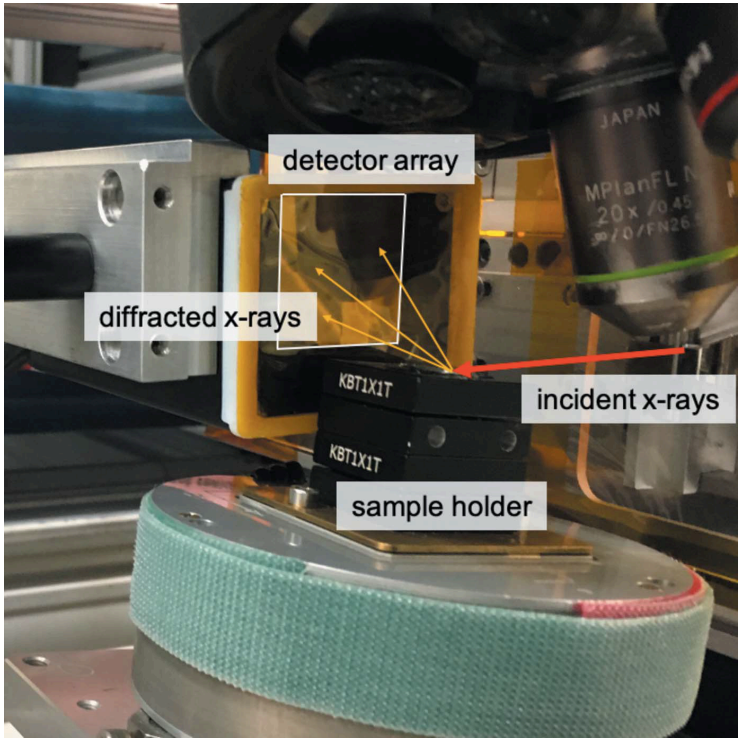
Energy (keV)	MonoTheta Cal.	MonoTheta Meas.	ΔMonoTheta
7	-10.5244	-10.4625	0.0619
8	-14.8818	-15.0225	-0.1407
9	-18.3798	-18.4238	-0.044
10	-21.157	-21.1275	0.0295
11	-23.352	-23.315	0.037
12	-25.1034	-25.1388	-0.0354
13	-26.5498	-26.665	-0.1152
14	-27.8298	-27.965	-0.1352
15	-29.082	-29.1275	-0.0455



Laue Detector Calibration with Energy using LaueGo

Manually optimized R and P and saved in *geoN_2020-12-10_10-23-48.xml*





OptimizeAll(CalibrationList_000270,$"",$"")
Nenergies = 5
Detector 0
started at **R={1.2092,1.2092,1.2092}**, **P={16.95,0,-27.16}**mm, |R| = 120°
sample started at axis = {3.00763, -0.0430946, -1.66093}, |sample angle| = 196.871°
ended at **R={1.21684,1.20658,1.21452}**, **P={17.329,0.501,-27.648}**mm, |R| = 120.343°
final sample rotation is {2.98265,-0.0436981,-2.71797}, |sample angle| = 231.219°,
Δrho = {-0.025, -0.0006, -1.1}, |Δrho|=61°
error started at 0.44055, reduced to 0.00272677, after 64 iterations
(final - initial) --> ΔR={0.0076,-0.0026,0.0053}, ΔP={0.379,0.501,-0.488}

(hkl) after E-cal	(hkl) before E-cal	Px	Py	E-measured [keV]
(1-11)	(111)	163	204	2.588 (12.94/5)
(3-53)	(335)	213	490	13.1
(5-53)	(535)	363	293	12.32
(3-55)	(355)	59	376	13.66
(7-55)	(755)	293	62	13.635

Laue orientation 0.7 degree error!

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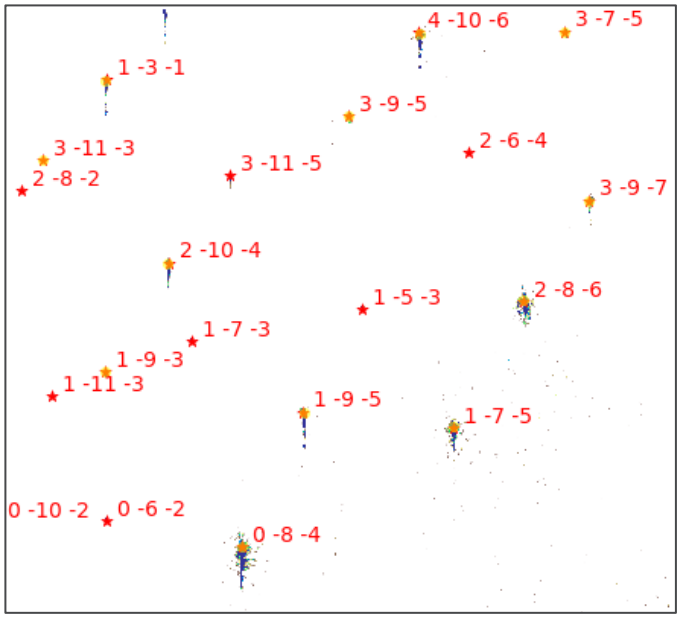
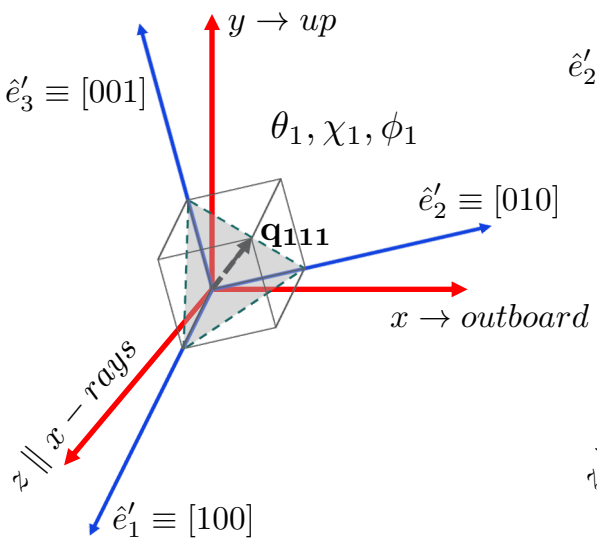
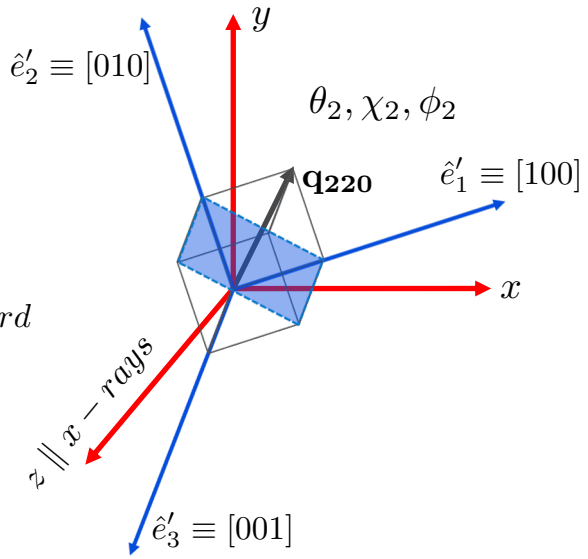
Laue Detector Calibration with Two Bragg Peaks

1. Determine sample orientation by locating two Bragg peaks on diffractometer.
2. Forward model Laue pattern with known sample orientation.
3. Nelder-Mead multiple parameter optimization for Laue detector
4. Reduced sample orientation error to 0.04 deg. with Laue







Zhang et *al.* (in preparation)

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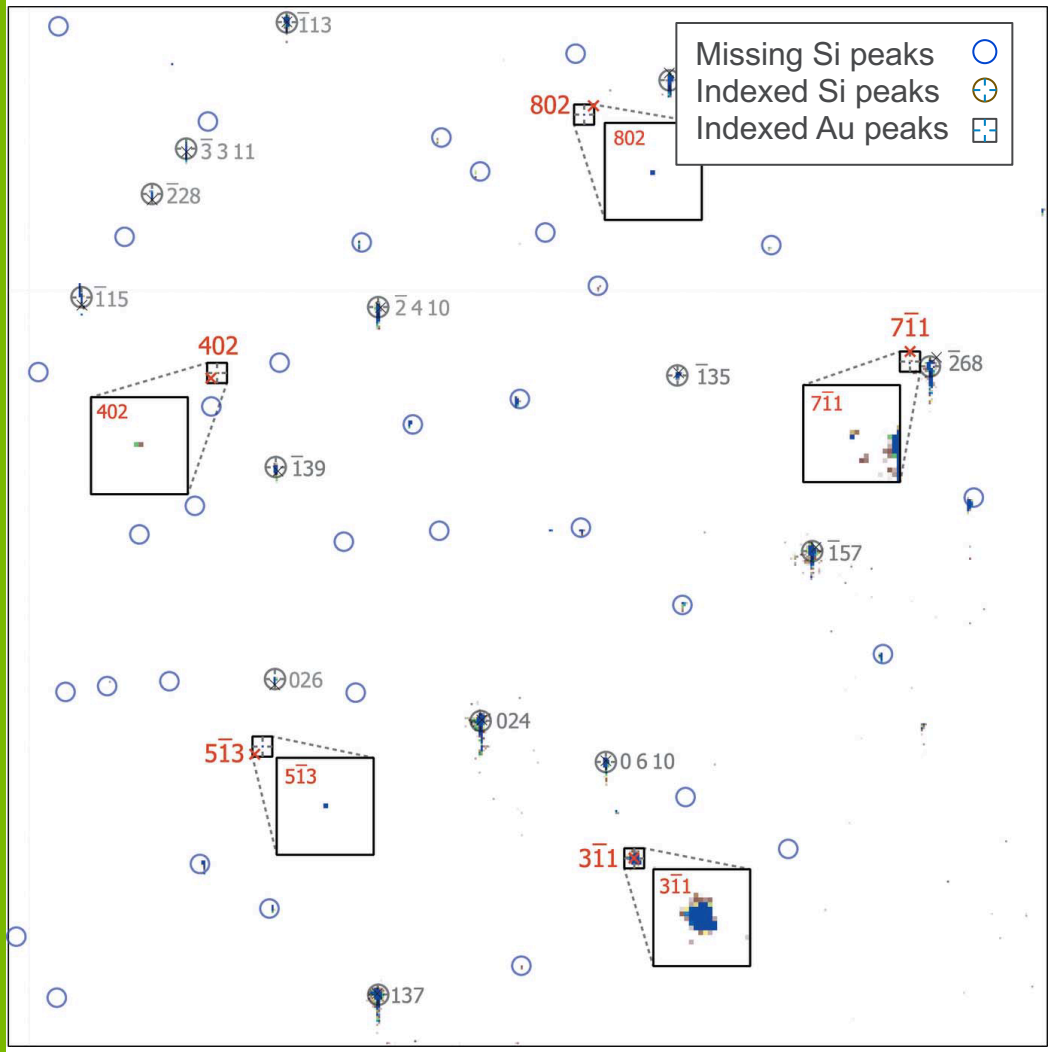
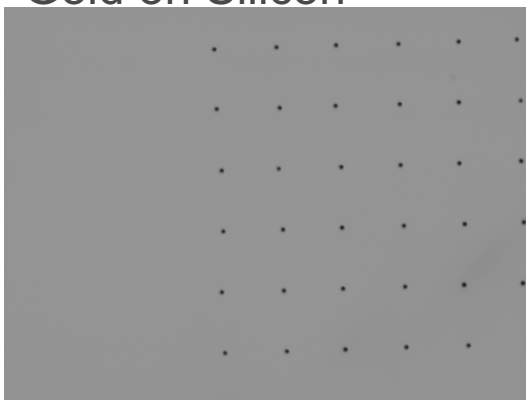
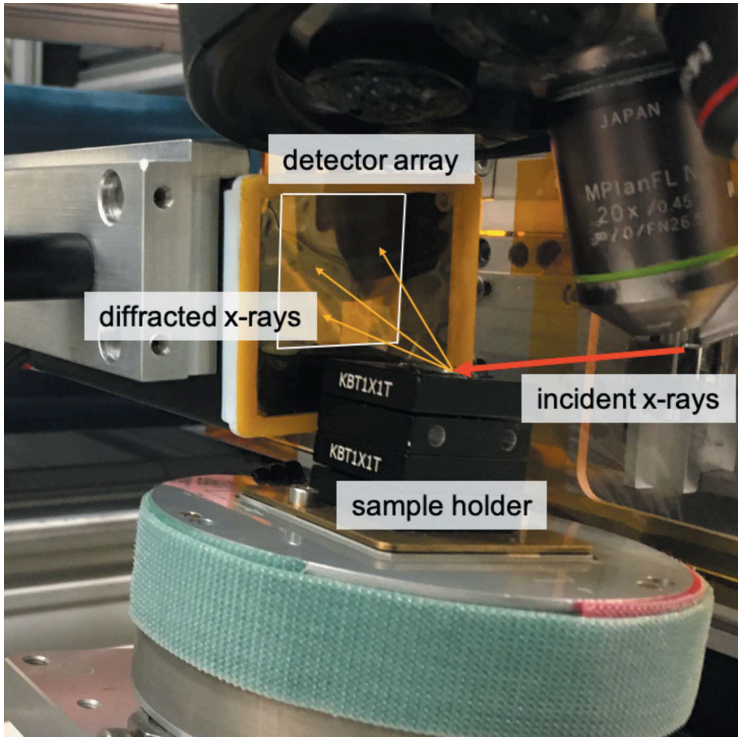
Laue diffraction @34-ID-C



Pateras et *al. J. Synchrotron Rad.* **27** 1430-1437 (2020)

Gold on Silicon





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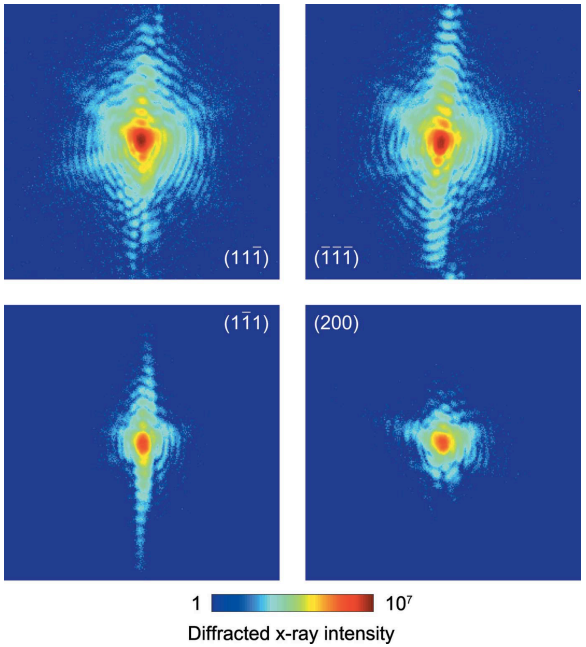
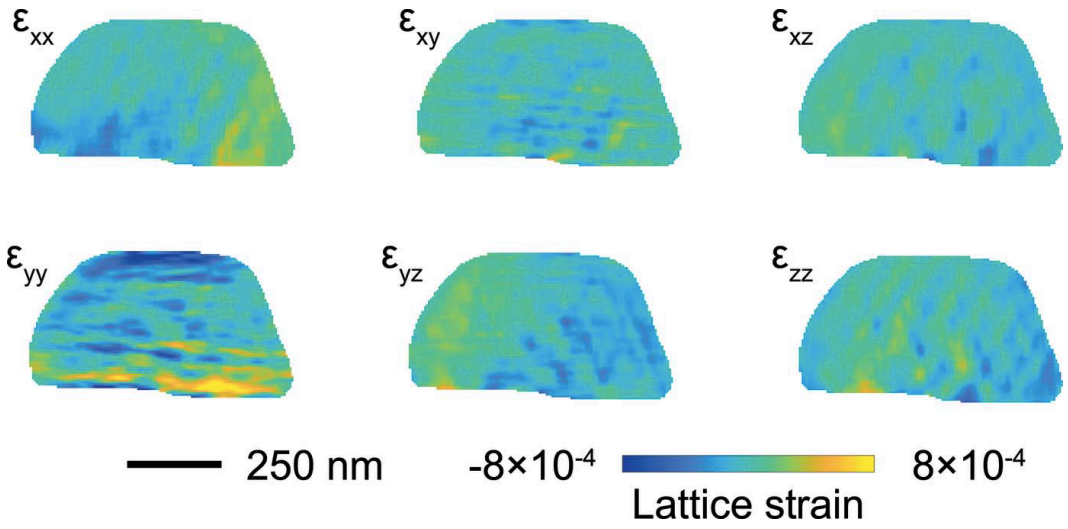
Multi-reflection BCDI

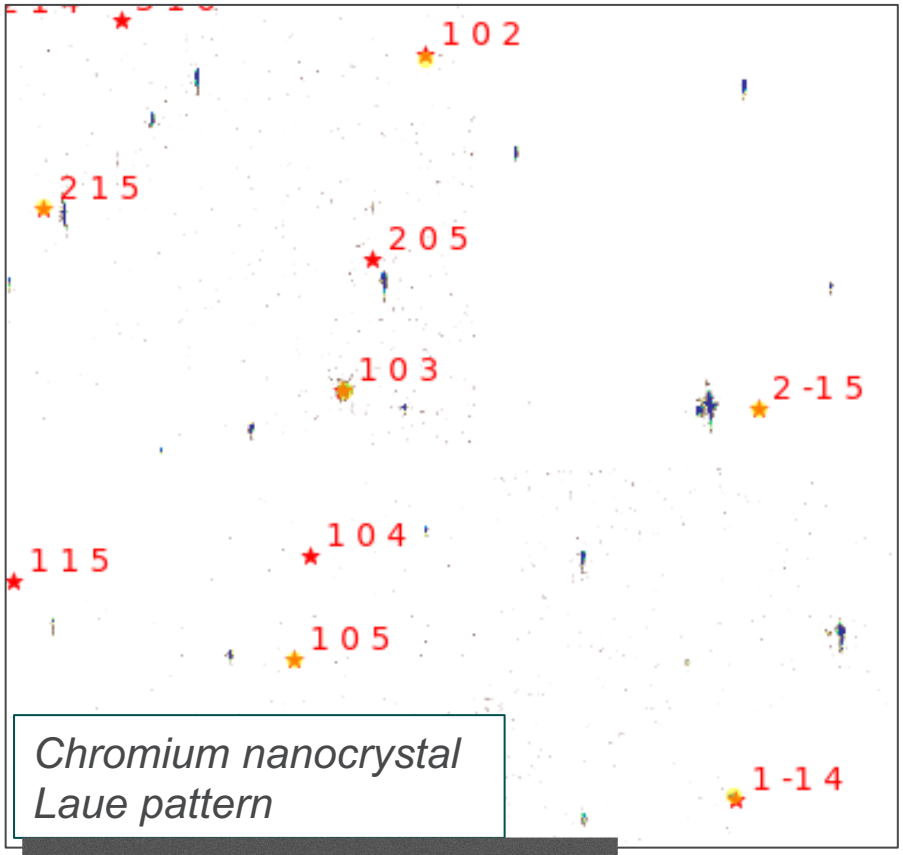
- Independently reconstructing the phases from each Bragg peak Hoffman et al. *J. Synchrotron Rad.* **24**, 1048-1055 (2017)
- Volumetric full strain tensor





Pateras et *al. J. Synchrotron Rad.* **27** 1430-1437 (2020)

Maturing into a user tool Courtesy Dr Ericmoore Jossou and Dr. Simerjeet Gill (BNL)

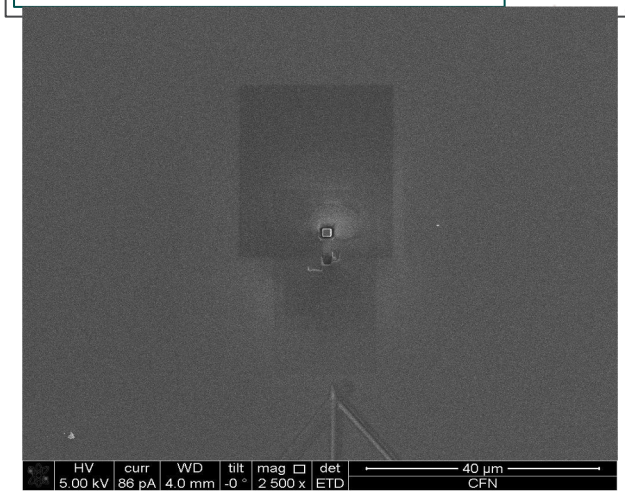


Chromium nanocrystal Laue pattern

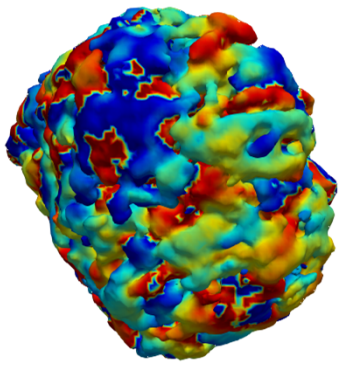
1. Algorithms for automatic blob finding
2. Procedures for peak sorting

   (Sample vs. substrate peaks)

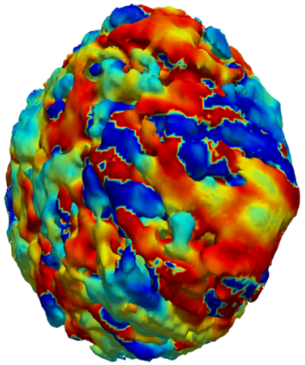
(hkl)	Error (deg.)
(1 0 2)	0.063
(1 0 3)	0.040
(1 0 5)	0.031
(1 -1 4)	0.079
(2 1 5)	0.030
(2 -1 5)	0.003



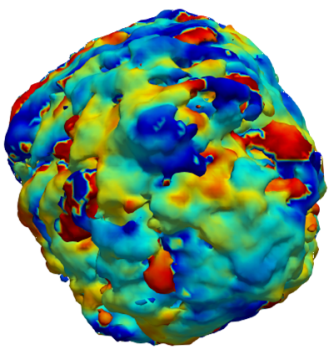
0-1-1



1-10



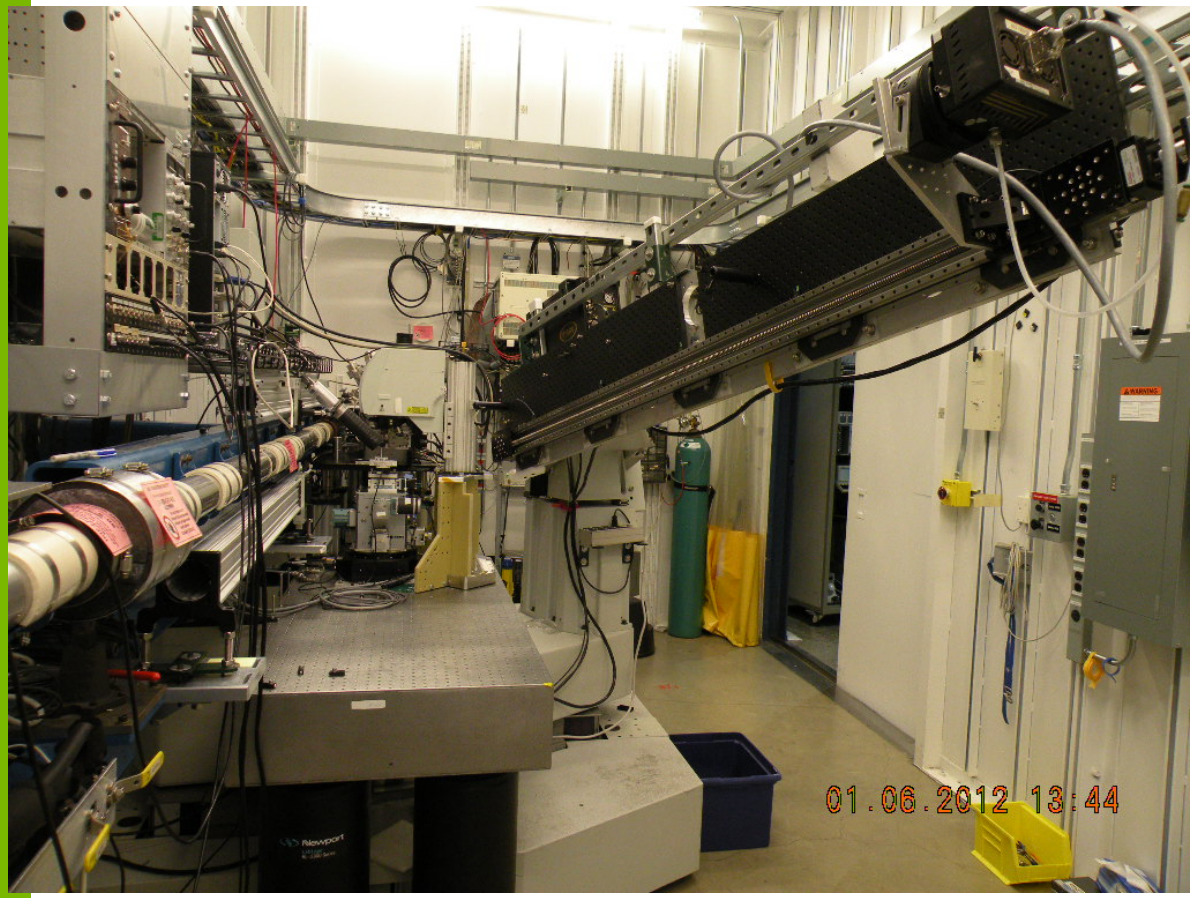
101

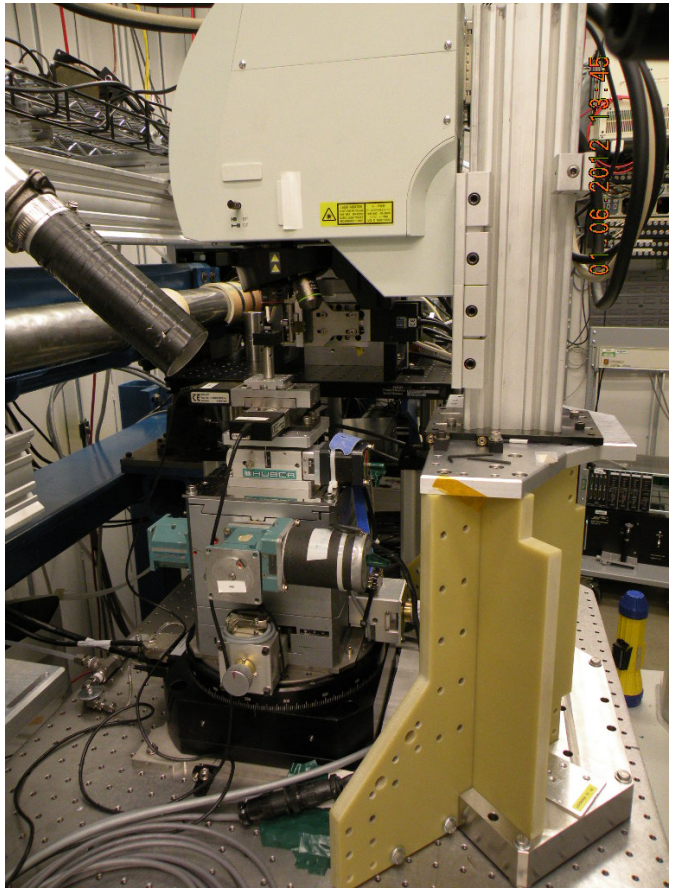


APPLY FOR BEAMTIME @ 34-ID-C!

EMAIL: RHARDER@ANL.GOV OR WCHA@ANL.GOV

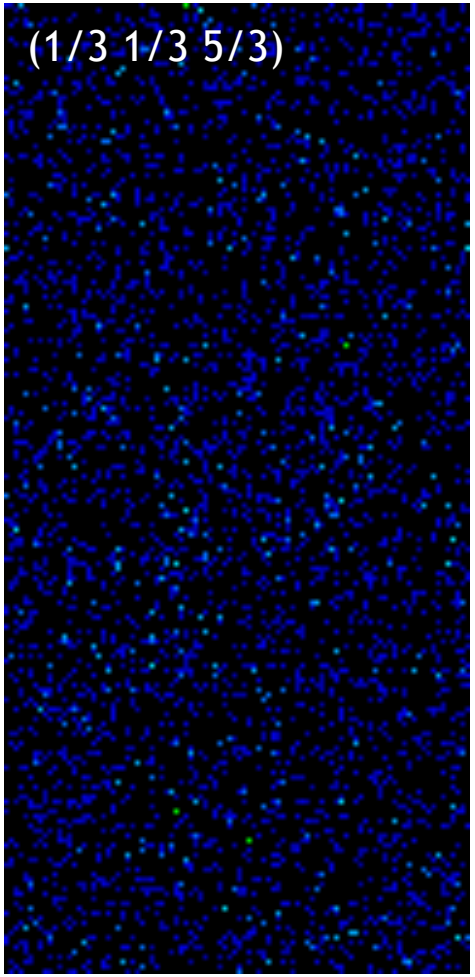
https://wiki-ext.aps.anl.gov/s34idc

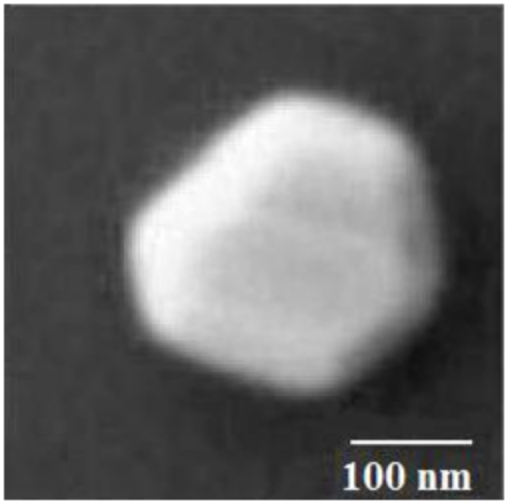




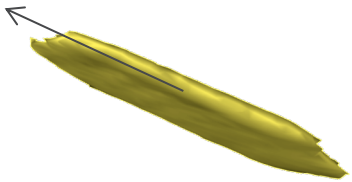
https://www.aps.anl.gov/

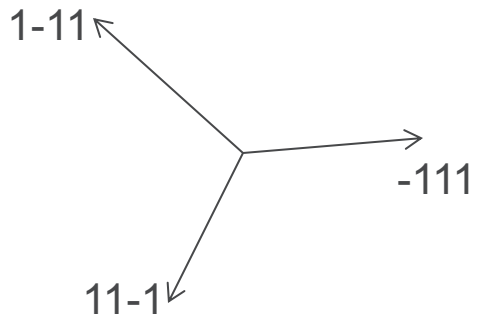
Multiple reflection reconstructions



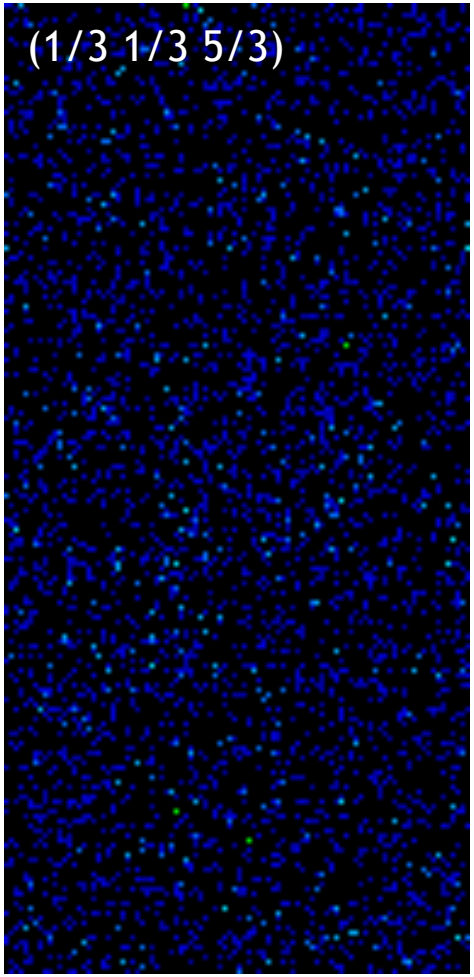


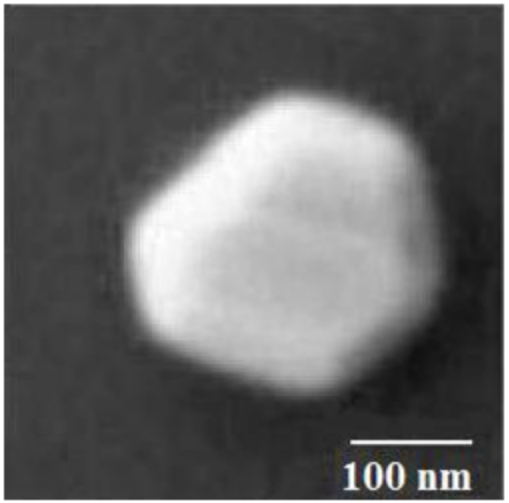
(1/3 1/3 5/3)

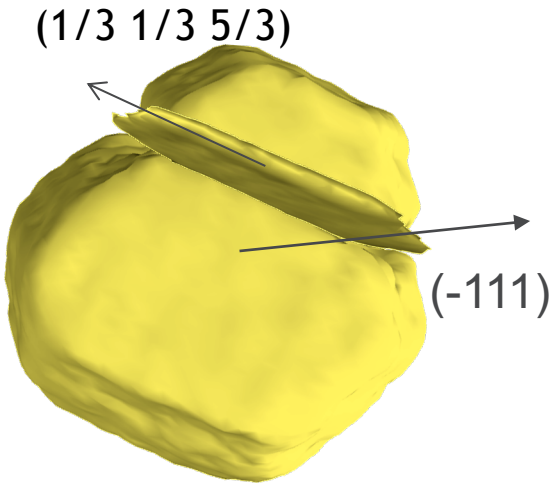


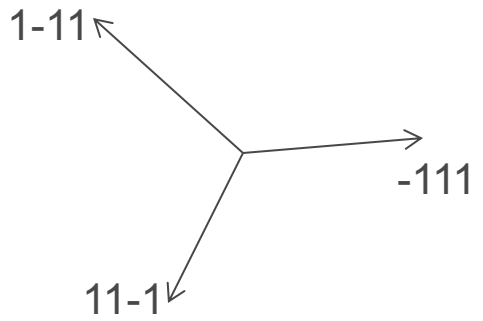


Multiple reflection reconstructions





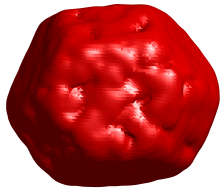




3D LATTICE DISTORTIONS AND DEFECT STRUCTURES IN ION-IMPLANTED NANO-CRYSTALS.

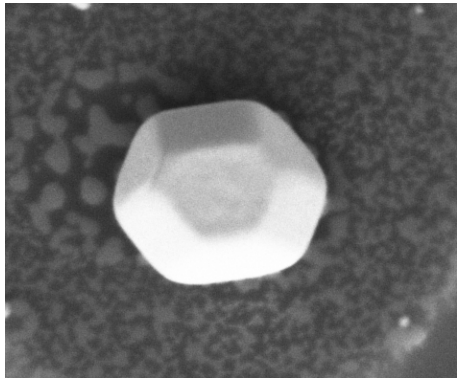
HOFMANN, F. *ET AL. SCI REP* 7, 45993 (2017).

BCDI reconstructed morphology

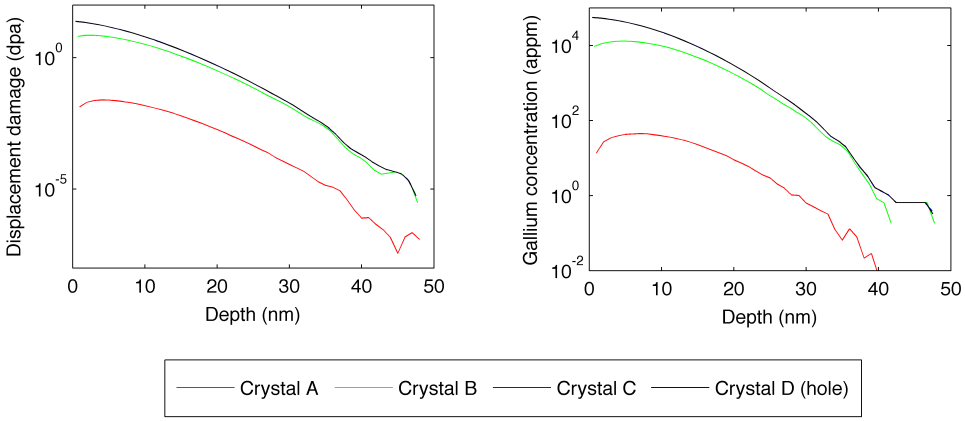


1 µm

Scanning electron micrograph



SRIM-predicted damage and Ga concentration



FIB milling conditions:

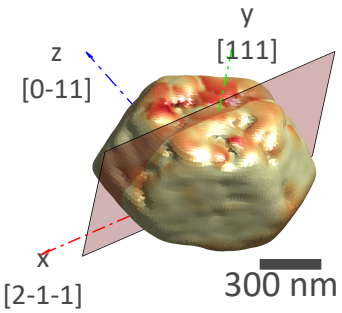
- 30 keV Ga^+
- 50 pA
- 1.5 x 10^8 ions/ μm^2

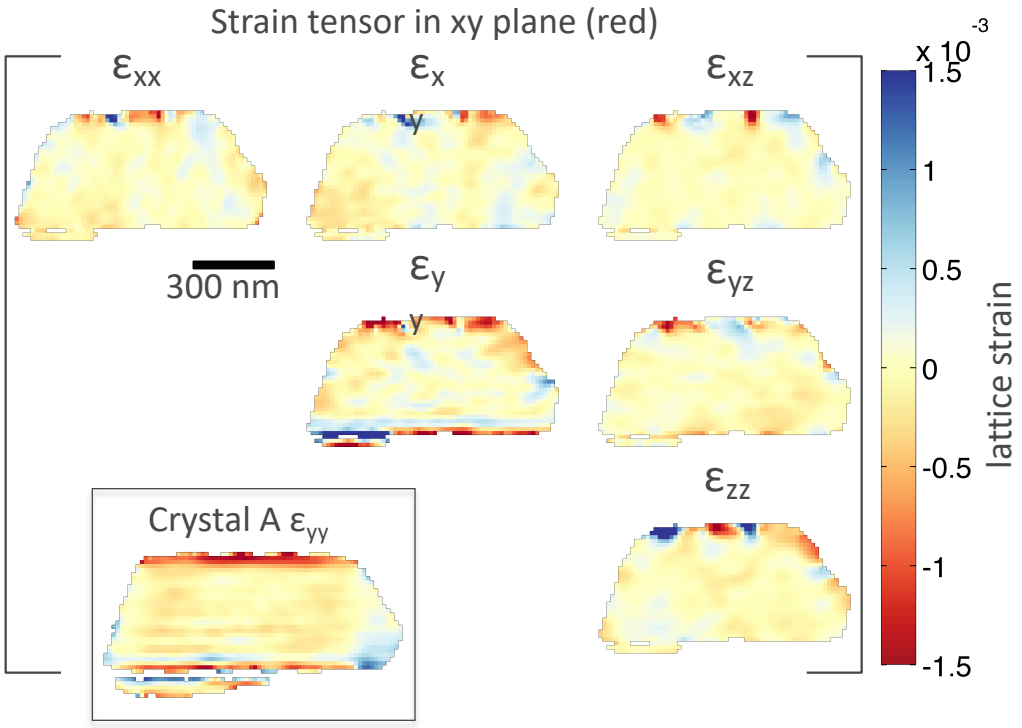
This causes (SRIM calculation):

- ~20 nm thick damaged layer
- max. ~24 dpa
- max. ~0.054 at. fr. Ga
- ~40 nm Au removed by sputtering

3D LATTICE DISTORTIONS AND DEFECT STRUCTURES IN ION-IMPLANTED NANO-CRYSTALS.

HOFMANN, F. *ET AL. SCI REP* 7, 45993 (2017).





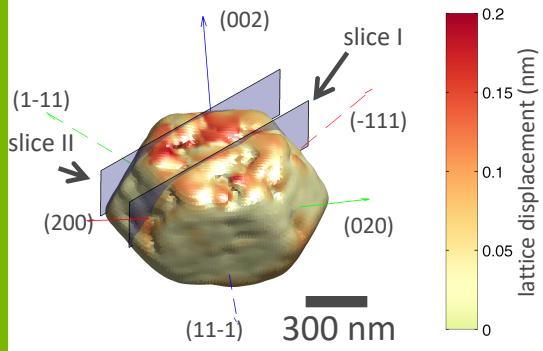
- Non-uniform ε_{yy} strain in implanted layer.
- Large positive and negative strains also in all other strain components.

Very different from ion imaged crystal

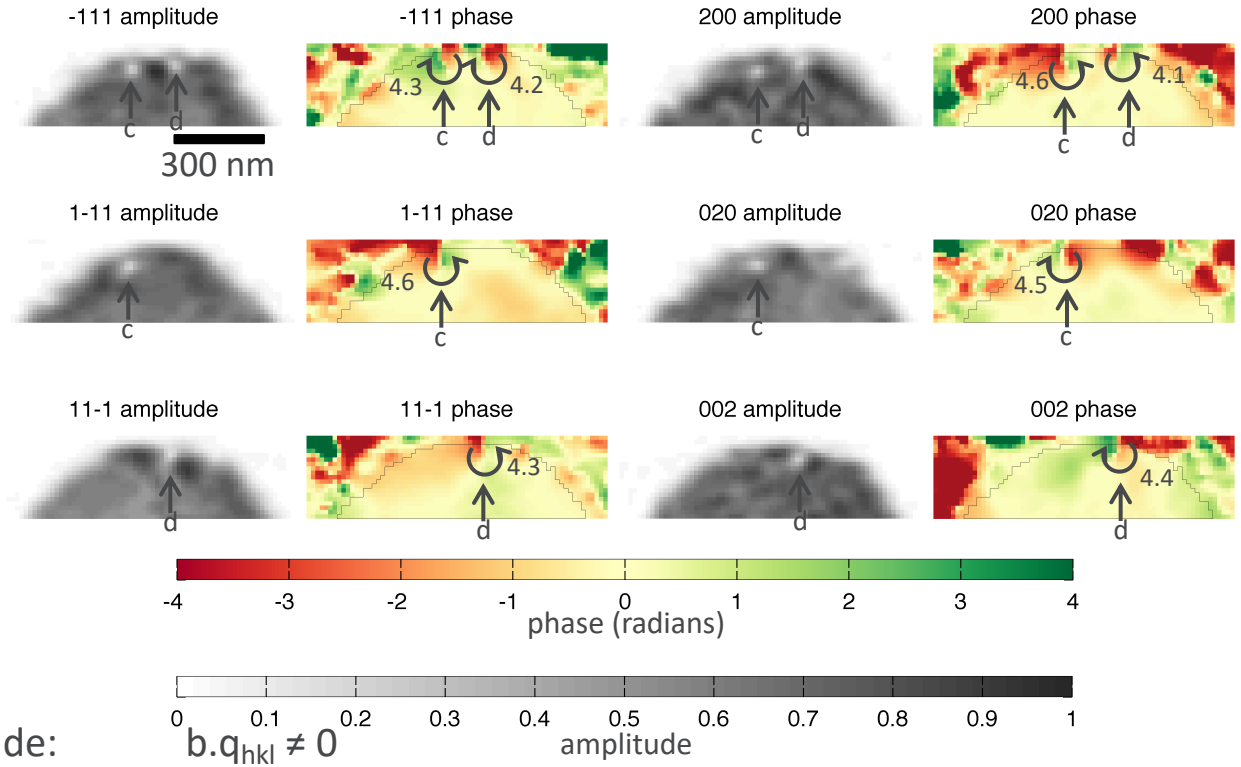
3D LATTICE DISTORTIONS AND DEFECT STRUCTURES IN ION-IMPLANTED NANO-CRYSTALS.

HOFMANN, F. *ET AL. SCI REP* 7, 45993 (2017).

Reconstructed crystal C



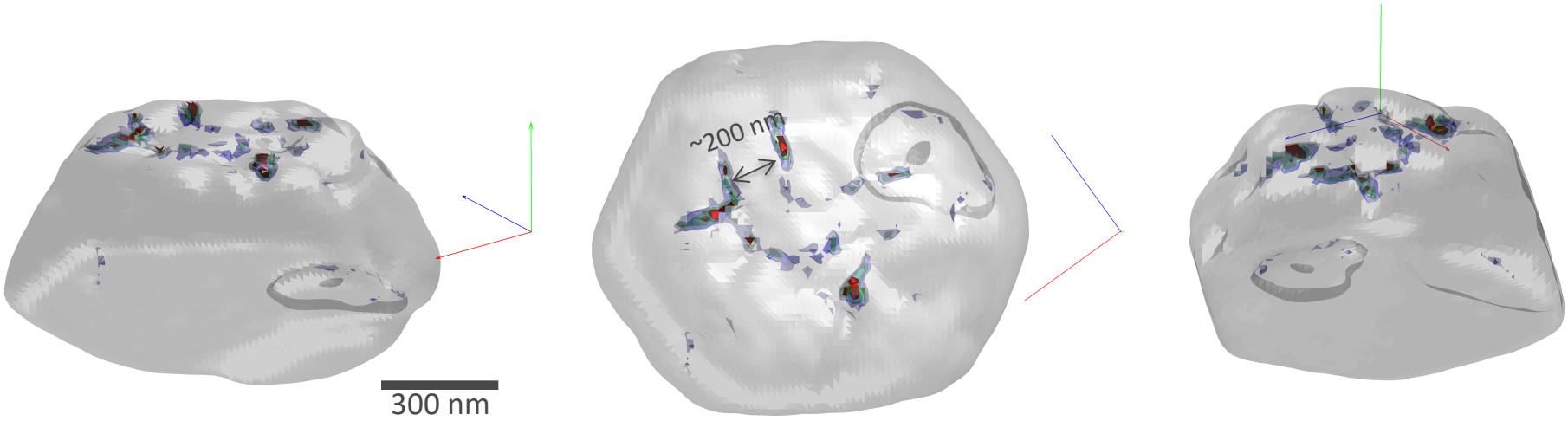
Crystal reflection amplitudes and phases on slice II



- Dislocation visibility in amplitude: $b.q_{hkl} \neq 0$
- Phase jump in Burgers circuit: $\Delta\psi_{hkl} = b.q_{hkl}$
- Defects are stair-rod dislocations with b = a/3<110>
  - -> Formed by interaction of 2 Shockley partials e.g. a/6[21-1]+a/6[-21-1] -> a/3[01-1]
  - -> Sessile hence retained?

3D LATTICE DISTORTIONS AND DEFECT STRUCTURES IN ION-IMPLANTED NANO-CRYSTALS.

HOFMANN, F. *ET AL. SCI REP* 7, 45993 (2017).



- Visualisation of 3D dislocation structure by plotting von Mises stress (300 Mpa (blue), 400 Mpa (green), 500 Mpa (red))
- Length scale of dislocation spacing ~200 nm
- A simple solution to biharmonic equation, $\Delta^2\mathbf{u}(\mathbf{r}) = 0$, admissible only at free surfaces, is: $\mathrm{u}_y(\mathbf{r}) = A\sin(kx)\exp(-ky)$, $\mathrm{u}_x(\mathbf{r}) = \mathrm{u}_z(\mathbf{r}) = 0$
- $k = 0.03\ \mathrm{nm}^{-1}$ gives a good fit to the depth-dependent strain due to FIB milling.
  -> The associated lateral lengthscale is $L = 2\pi/k = 210\ \mathrm{nm}$

Simple model provides remarkably good estimate of dislocation self-organisation length scale!