X-Ray Imagining and Phase Reconstruction with HHG Sources

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X-Ray Imagining and Phase & Tomographic Reconstructions with Southampton x-ray sources

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University of Southampton

X-Ray Sources @ Southampton

- Ultrafast Laser HHG soft x-ray source
- MuVis large sample Tomographic imaging suite
- National Crystallography Centre
  - Single crystal diffraction
  - Powder diffraction
  - Protein Crystallography
- X-Ray absorption, EXAFS {Diamond Light Source}

The Phase Problem

- Difficulty with x-ray optics means we can't image in the way we might in the optical region
- This means we record
  - Shadow images
  - Diffraction patterns
  - Intensity but not phase

What do we do?

- Ignore the phase
  - Radiography
  - Tomography
- Work out the phase
  - Crystallography – work out phase of each Bragg spot using experimental and computational methods
  - Imaging – use over sampling and constraints to calculate the phase and so computationally re-image the x-rays

DON’T WORRY ABOUT THE PHASE

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X-Ray

Rhodri and Lucy Owen explore consumer concerns from across Wales and Rachel Treadaway-Williams investigates the issues worrying Welsh consumers.

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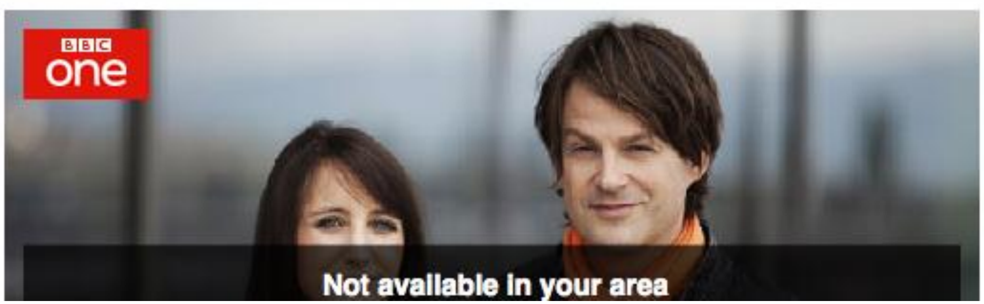

Coming up

MONDAY, 19:30 on BBC One (Wales only)
Series 11
Episode 13
Consumer series with Lucy and Rhodri Owen and reporter Rachel Treadaway-Williams.

X-Ray guides

If you have a consumer problem, let the X-Ray team help you with our handy guides.

Reports

Mr Windows

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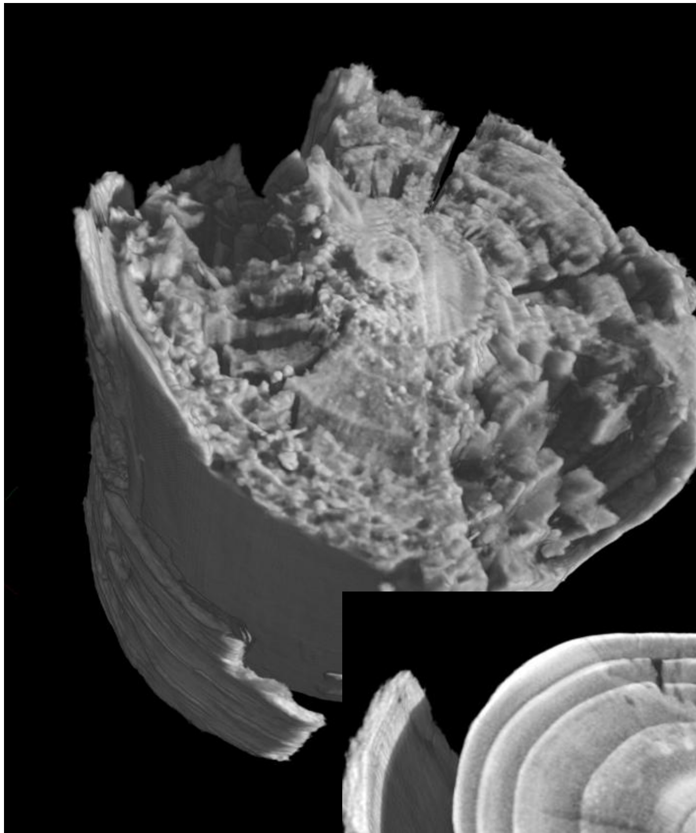
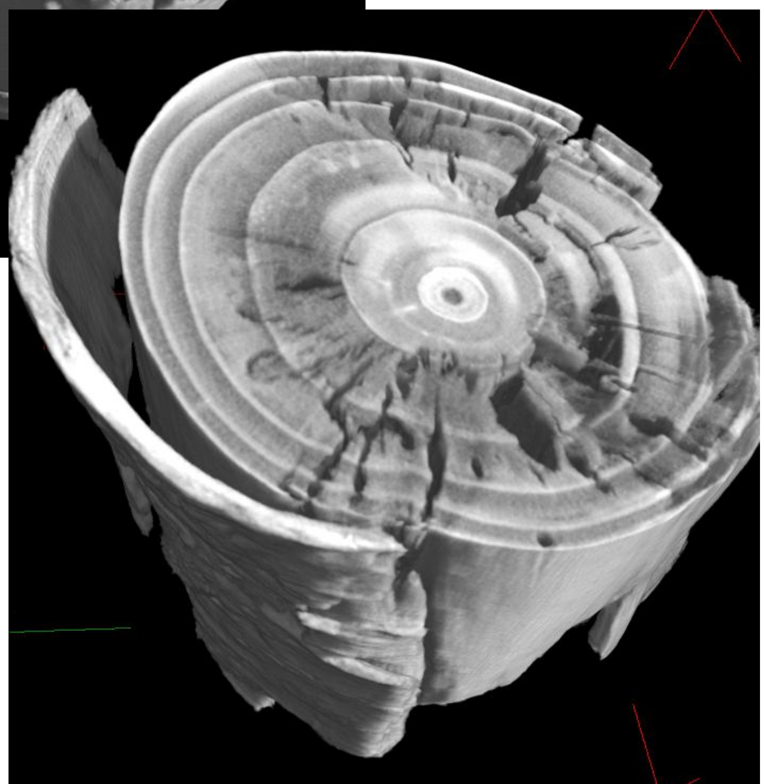
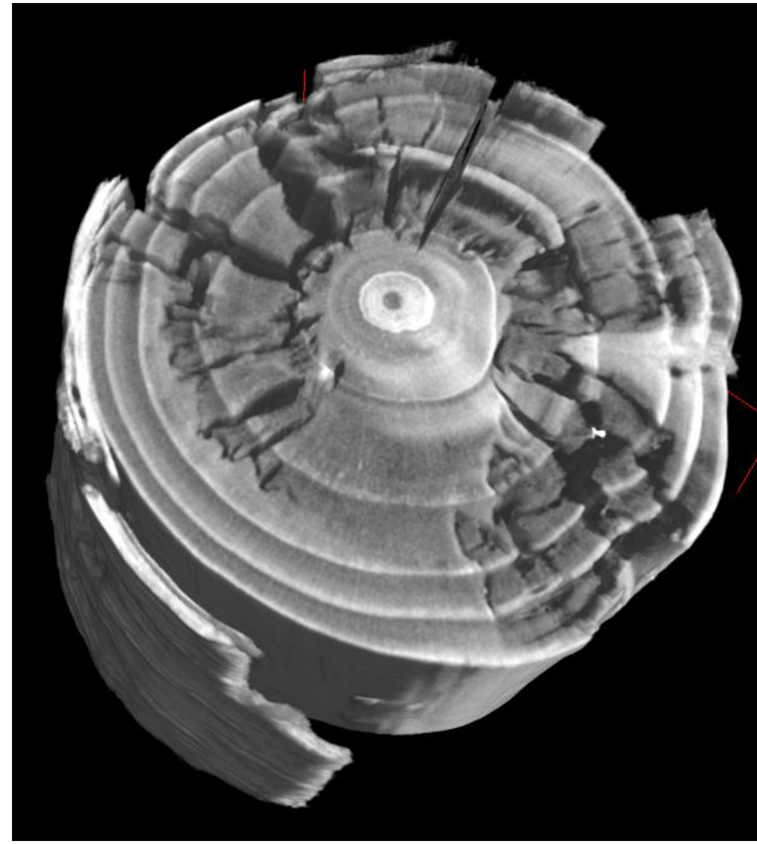
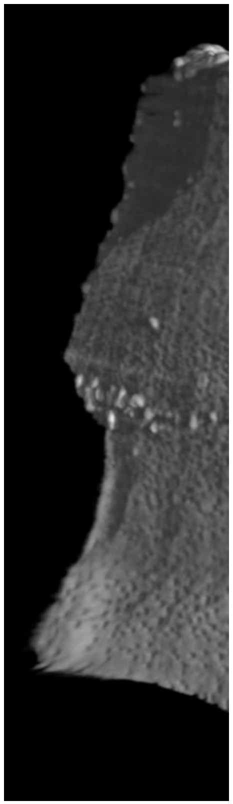
Tree branch

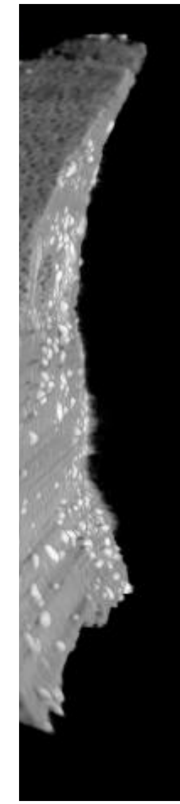






Archaeological charcoal samples





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X-ray microtomographic imaging of charcoal

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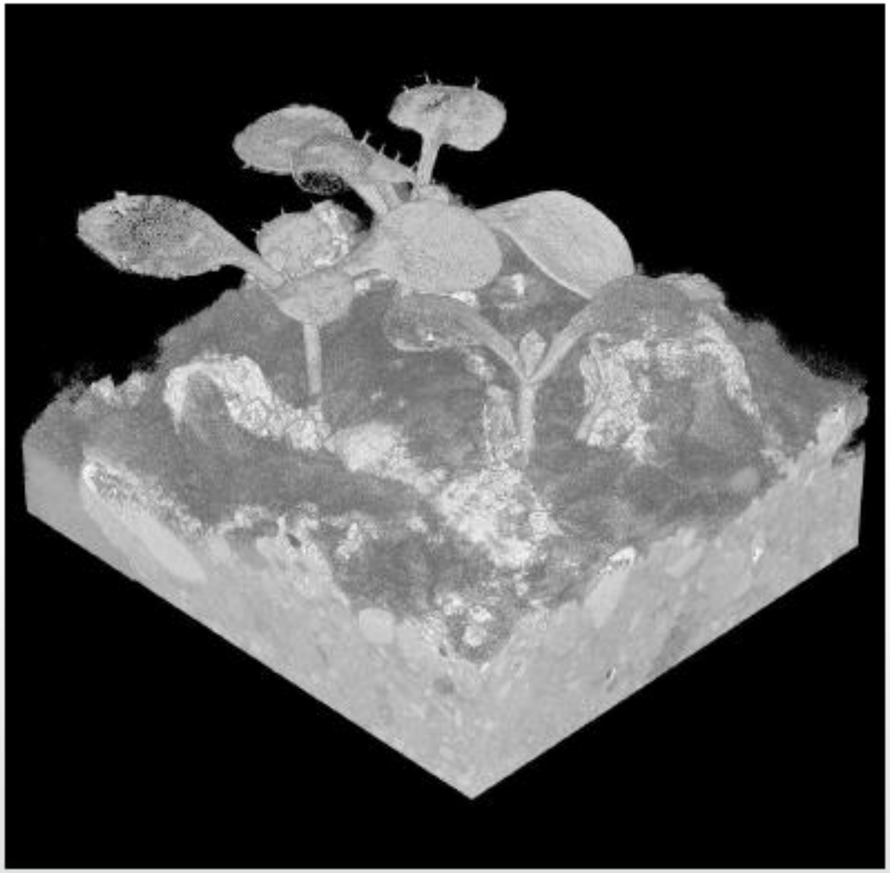
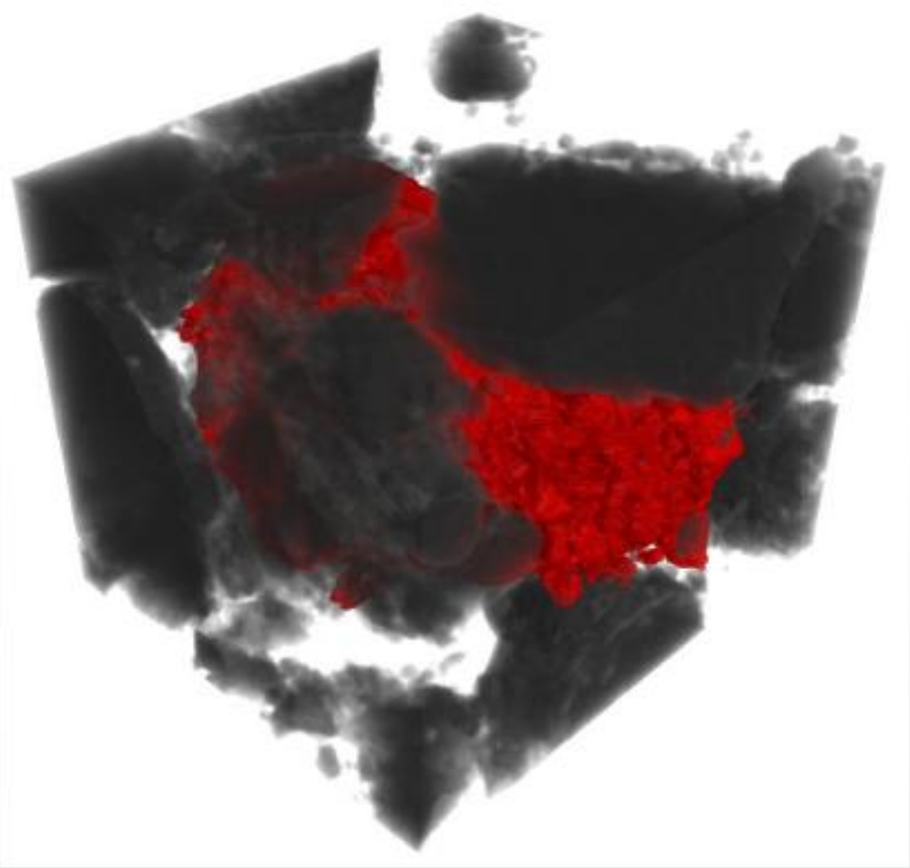
A B S T R A C T

We assess the potential of X-ray microtomography as a tool for the non-destructive, three-dimensional examination of the internal structure of charcoal. Microtomographic analysis of a series of charcoals produced by the experimental pyrolysis of pine wood at temperatures from 300 and 600 °C in nitrogen only and in nitrogen mixed with 2% oxygen indicates that, despite substantial shrinkage, observed porosity, pore size and pore connectivity are all increased by pyrolysis and also by chemical oxidation. Analysis of a number of altered and unaltered archaeological and geological charcoals has demonstrated the capacity of the technique to identify and map the distribution of authigenic mineral contamination within charcoal fragments. The results are of significance to radiocarbon dating in that they provide insights into the mechanisms by which charcoal can be contaminated by extraneous carbon in the natural environment.

Bressan

In-Vivo CT of Soil and Plant Roots



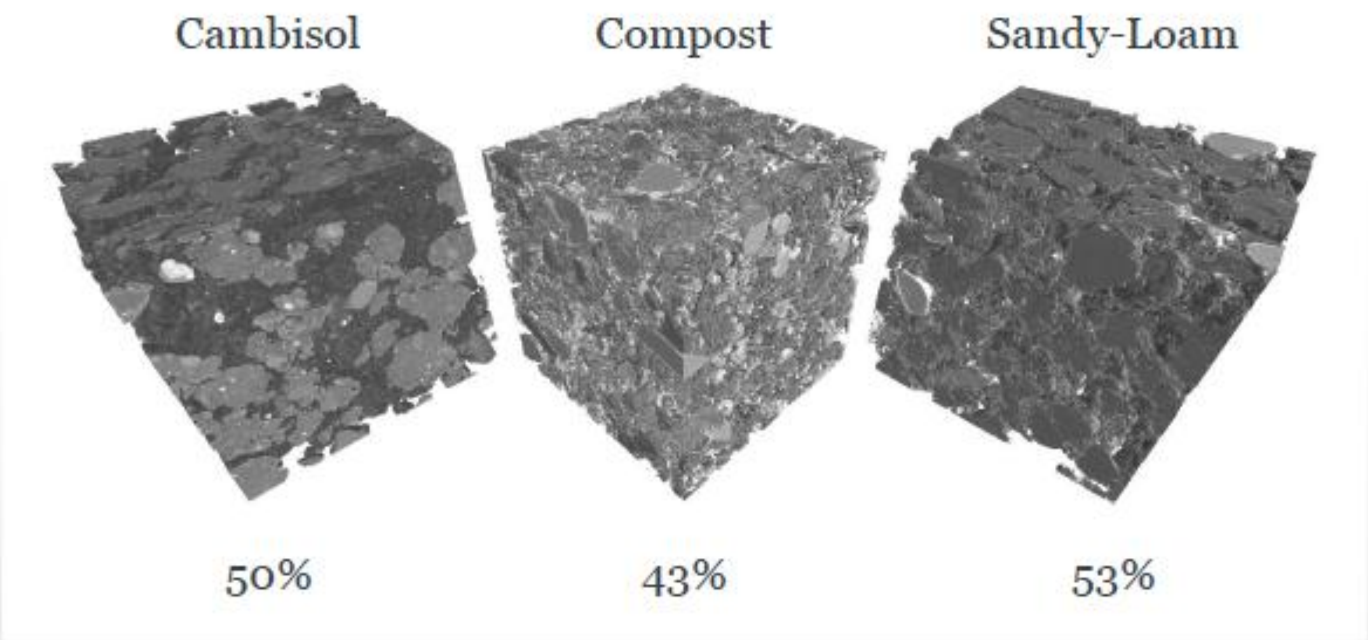


Samuel Keyes – School of Engineering Sciences

Non-Destructive Investigation

- CT imaging holds significant promise in allowing non-destructive studies over a sustained period.
- First use of a high-energy industrial CT scanner for root imaging was in the late 1990's (160-200µm - Heeraman 1997), with partial success since.
- This is a young field with few concrete results.

Image Processing



- Aggregate and macro-pore space were differentiated using a region growth algorithm within the VGStudioMAX environment.

CALCULATE THE PHASE

ncs | UK National Crystallography Service

Welcome

The Service is an amalgamation of resources at two centres; laboratory-based facilities in the Chemical Crystallography Laboratory at the School of Chemistry, University of Southampton, together with provision of a synchrotron-based facility on station I19 at the Diamond Light Source.

Latest News

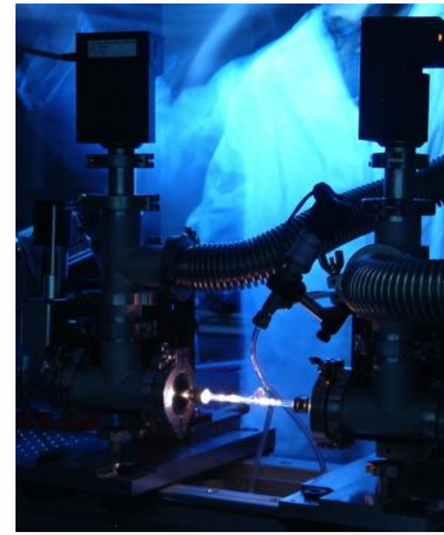
09/05/2011: The new allocation period has started, the service has had a record number of requests. If you applied but have not received an email about your allocation please contact us on (read more)

24/11/2010: We are pleased to announce that we have selected Rigaku to be our equipment supplier. The equipment will include the FRE+ SuperBright (Mo) boasting both the new VariMax VHF (Very High Flux) and a... (read more)

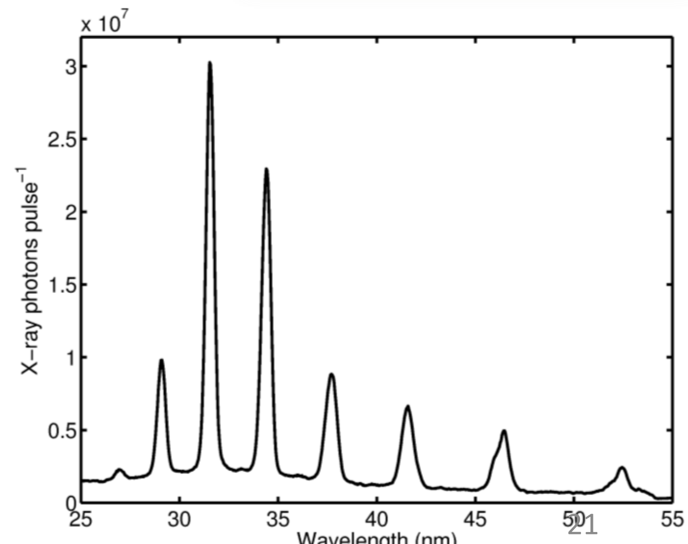
TAKE ADVANTAGE OF COHERENT SOURCES

Southampton HHG source

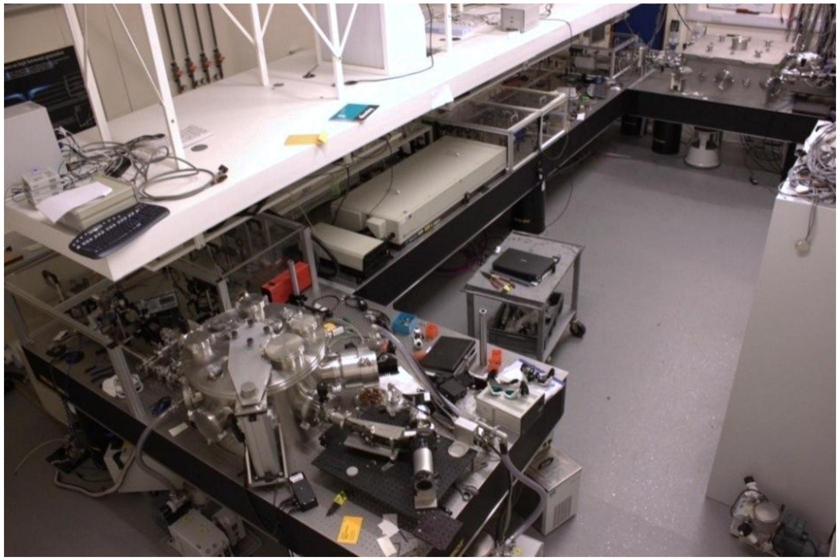
- Pump laser: Ti:sapphire – 800 nm
  - 38 fs pulses, 3 mJ pulse energy, 1 kHz rep rate
  - mid 10^{14} W/cm^2 when loosely focused
- Geometrical phasematching via capillary waveguide or Guoy shift in gas cell (both sources used experimentally)

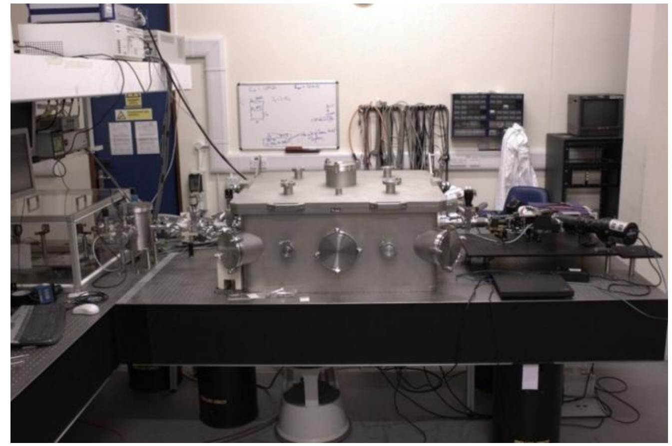


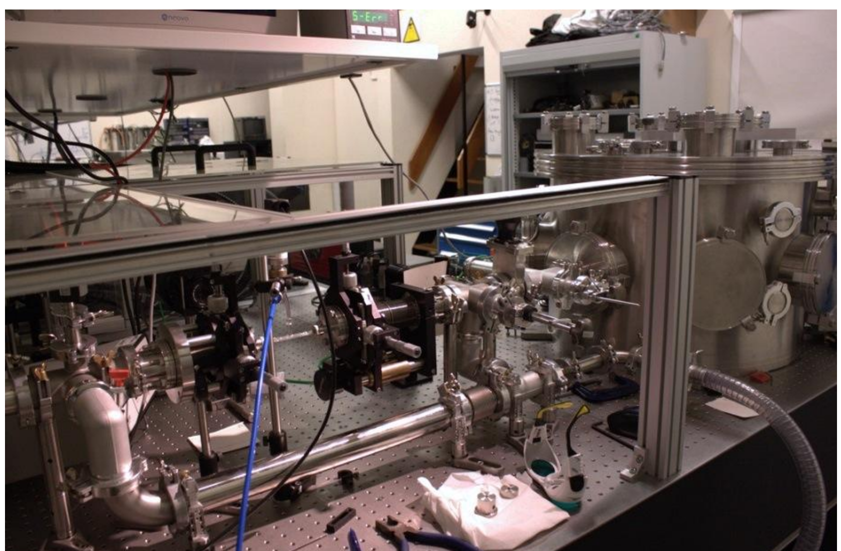
- Southampton XUV source output parameters:
  - Wavelength 18-40 nm
  - Efficiency ~10^{-5} – 1W input, 10 μW out
  - High spatial coherence
  - 10^{12} photons per second in a 1 mrad beam, 1% bandwidth
  - M^2 ~ 2 before focusing
  - Pulse envelope length ~10 fs

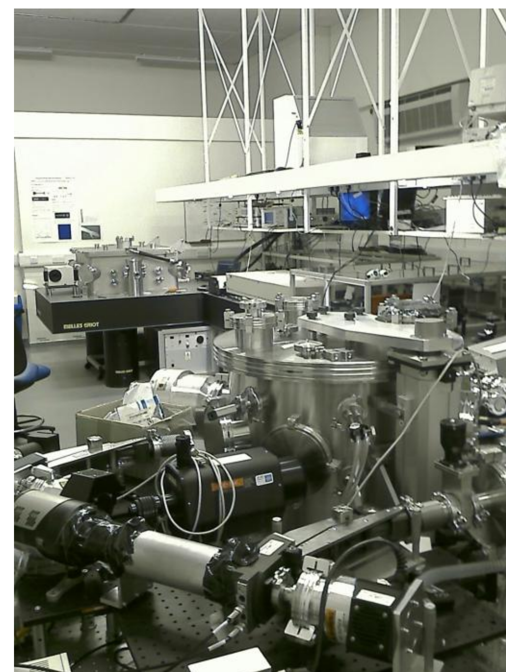


The HHG lab at Southampton









Unique source properties

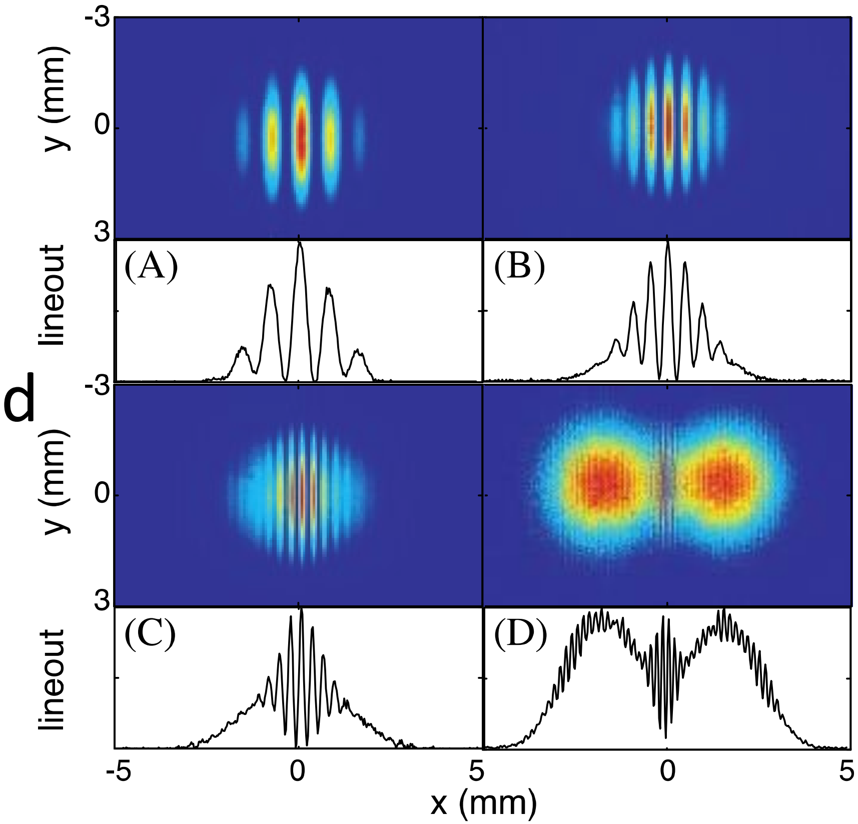
- Lab-based source of 30 eV-1 keV
- Spatially coherent
  - Imaging – major research topic
  - Lithography – inspection of components
- Short (attosecond) pulses – dynamics of electrons in atoms/molecules
- Highly synchronised – low jitter, pump/probe experiments possible

But:

- Flux is low (1 nJ/pulse, 1 μW average)
- Lasers are expensive & complex (at the moment)

Spatial coherence

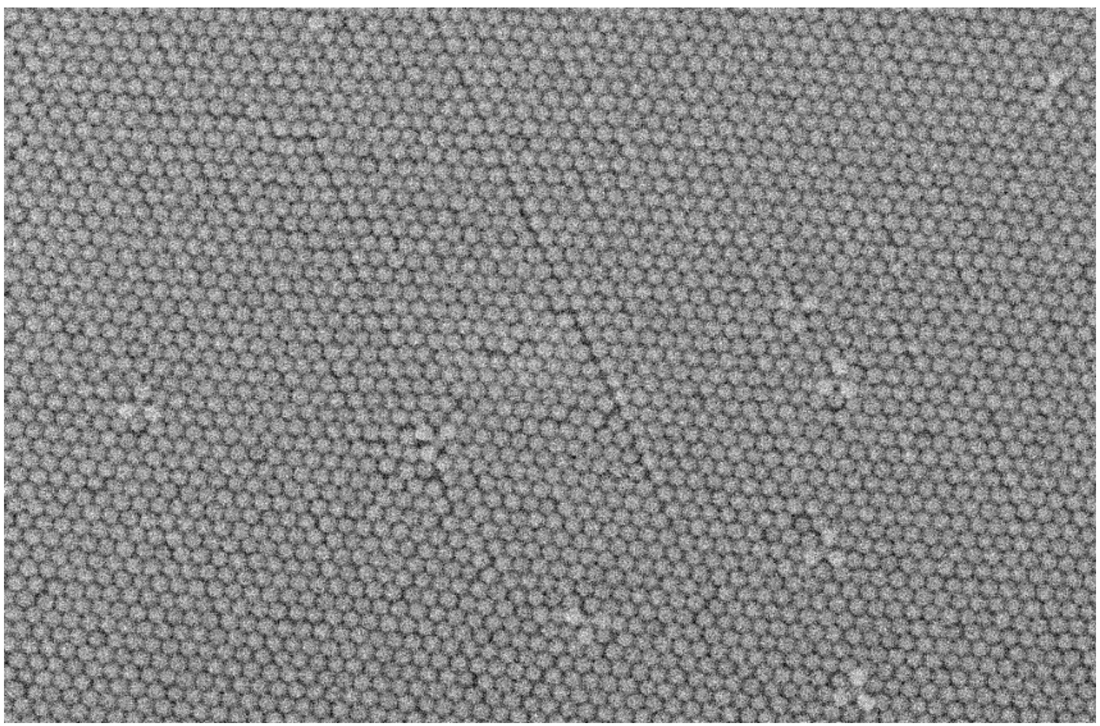
- nonlinear frequency conversion preserves spatial coherence of source
- HHG beans demonstrated to be highly spatially coherent via two-beam interference
- Important for many experiments – imaging, focusing



Bartels, R. a et al. Generation of spatially coherent light at extreme ultraviolet wavelengths. Science (New York, N.Y.) 297, 376-8(2002).

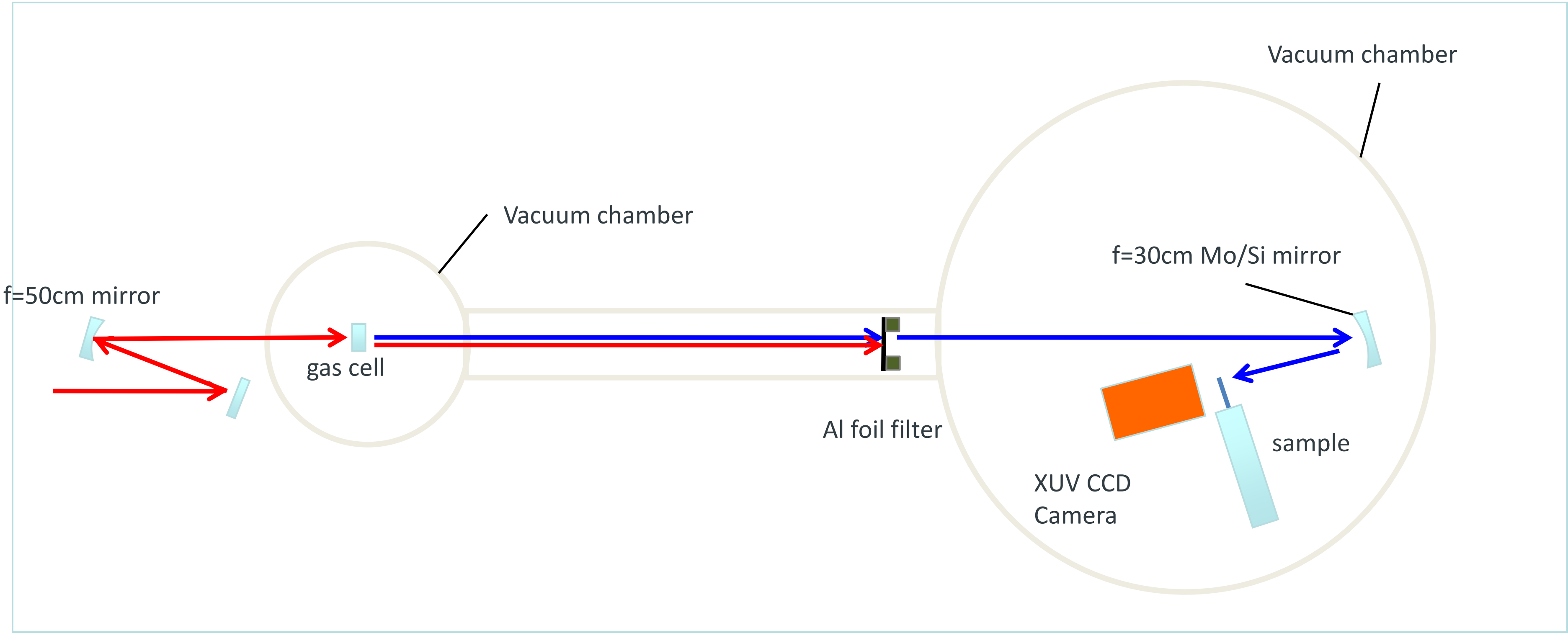
Samples: self-organised PMMA sphere arrays

- Samples produced by C.F. Chau, ORC, Southampton.



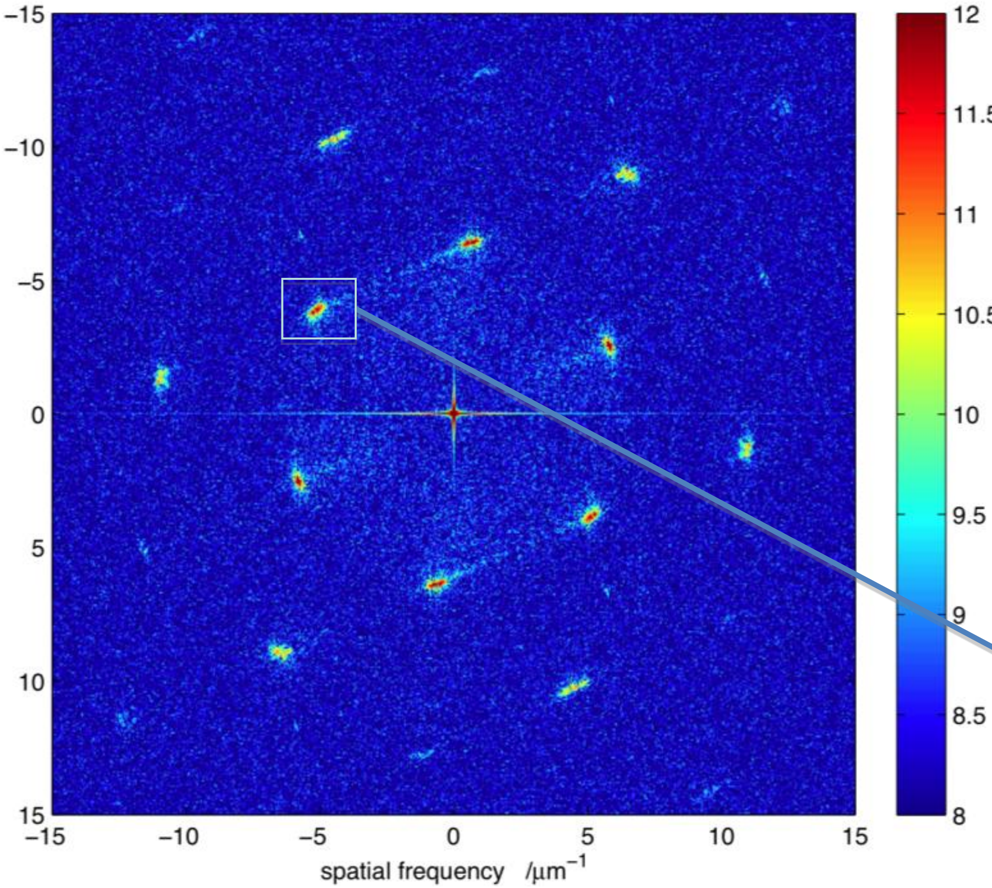
- Diameter 196nm, size variation < 5%
- Single layer of spheres on 50 nm SiN membrane
- Ordering good, but not perfect.
- Uses: photonic/plasmonic crystal templates

Experimental setup

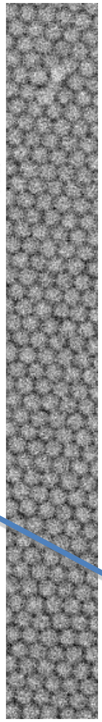


XUV source: HHG in Ar-filled capillary or cell, peaked at ~29 nm
XUV mirror: Spherical Mo/Si multilayer (IOF Jena)
Detector: ANDOR XUV CCD, 17mm from sample

Samples: self-organised PMMA sphere arrays



F. Chau, ORC,



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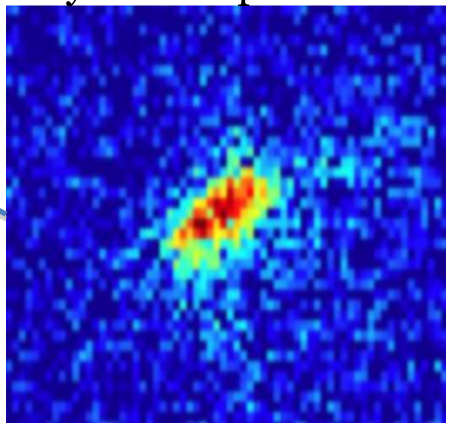
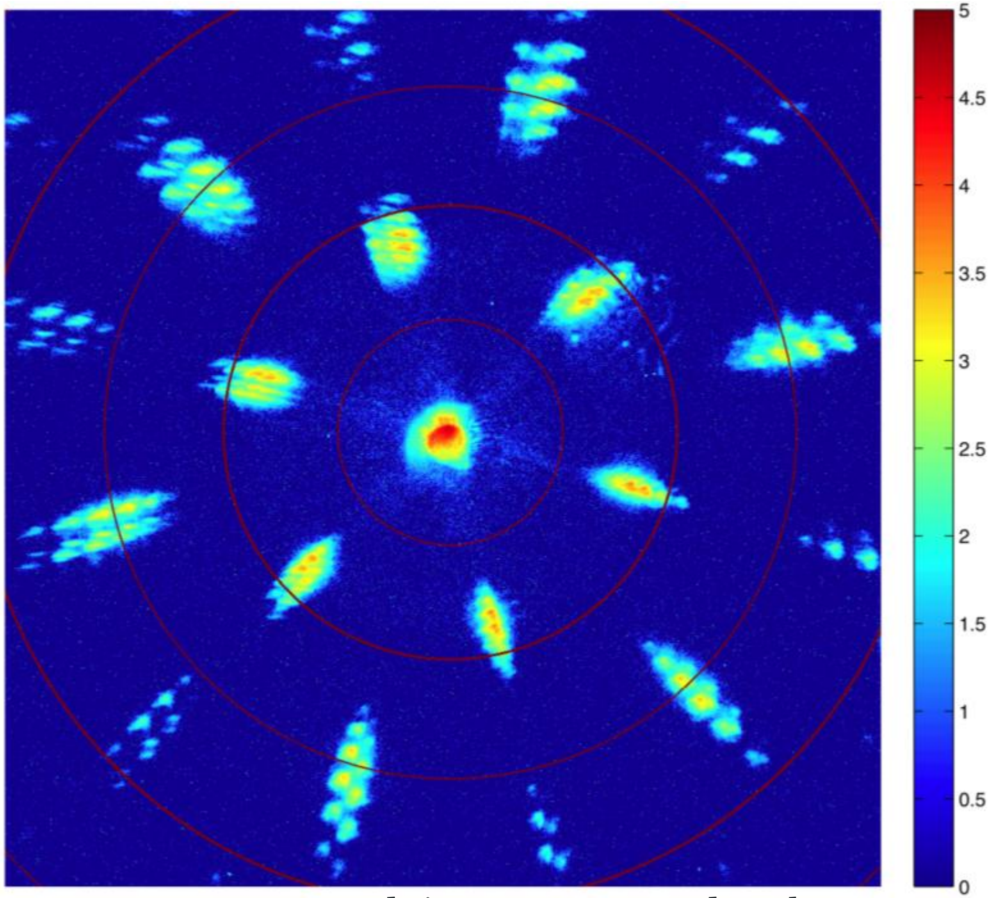


Scattering from ordered sphere regions

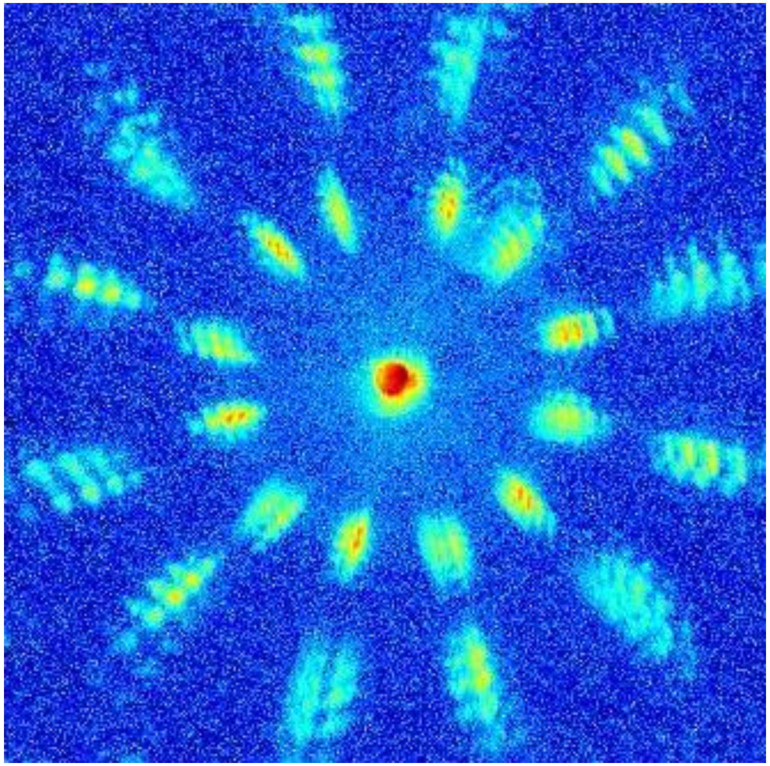


Red rings are 100 mrad angle contours
Intensity scale is logarithmic.

XUV transmission diffraction from 196nm sphere array, ~10μm XUV focal spot on sample

•Radially: multiple wavelengths give multiple spots
•Tangentially: structural information
•Other distortions arising from XUV phase front distortion

Scattering from multiple grains



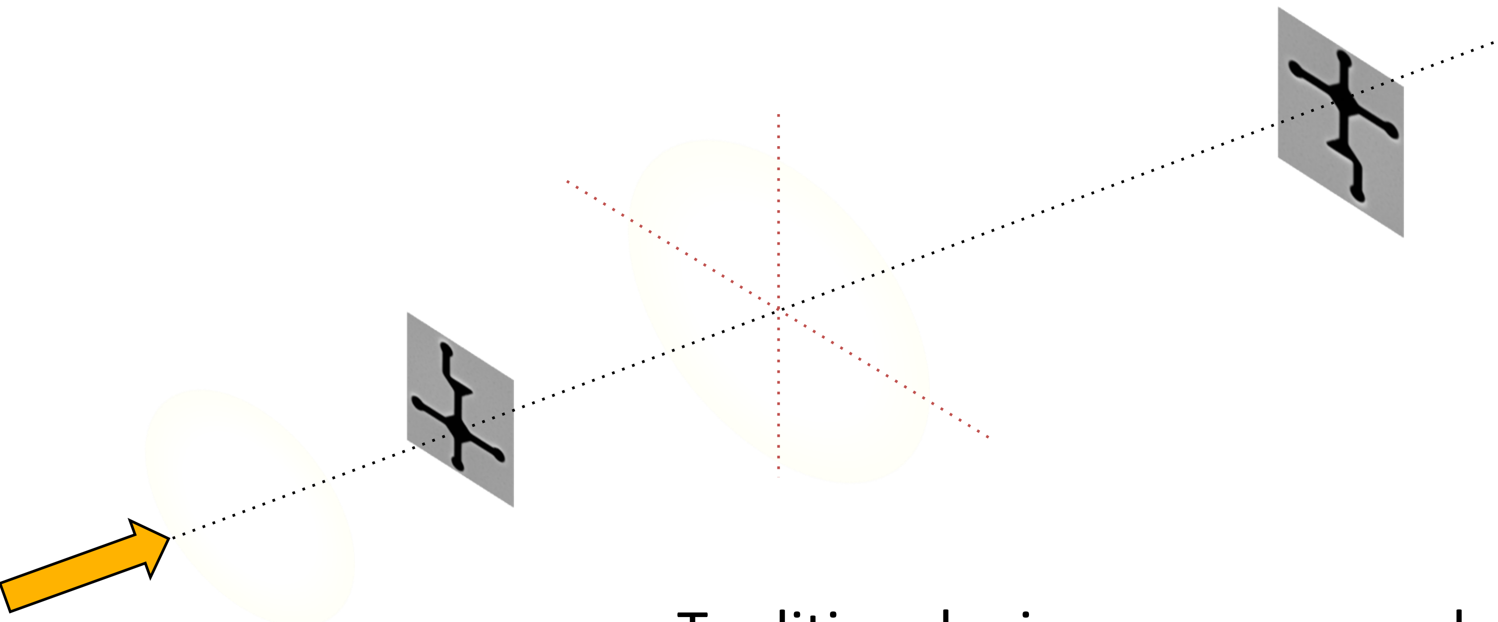
Scattering patterns are very sensitive to grain boundaries

This image shows the ~20μm XUV spot positioned over two grains, with ~30°between the lattice orientations

XUV beam positions with single crystal diffraction patterns are common across samples

Lensless imaging: Phase retrieval

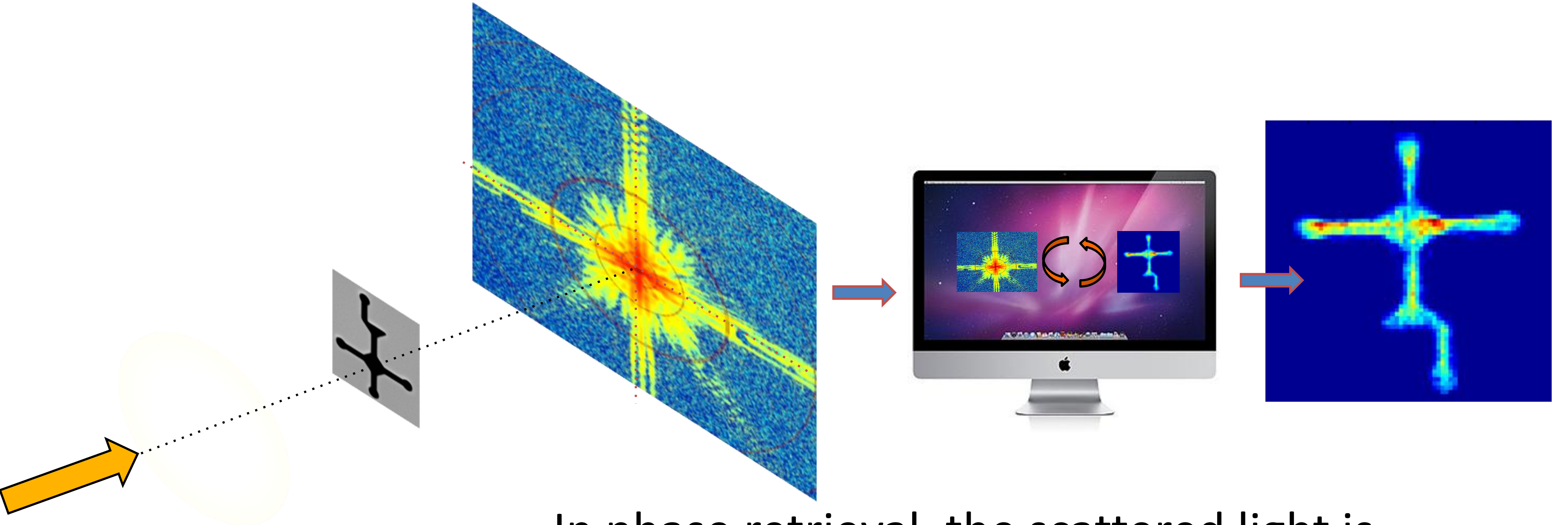
Collection of scattered radiation from an object



Traditional microscopy uses a lens to re-phase the different Fourier components to create an image – only intensity information is retained.

Lensless imaging: Phase retrieval

Collection of scattered radiation from an object



In phase retrieval, the scattered light is collected and phases lost.
Phase info is re-established via iterative algorithm

Coherent Diffractive Imaging (CDI)

- Developed for X-ray crystallography – the “phase problem”
- Iterative application of constraints in object and Fourier space:

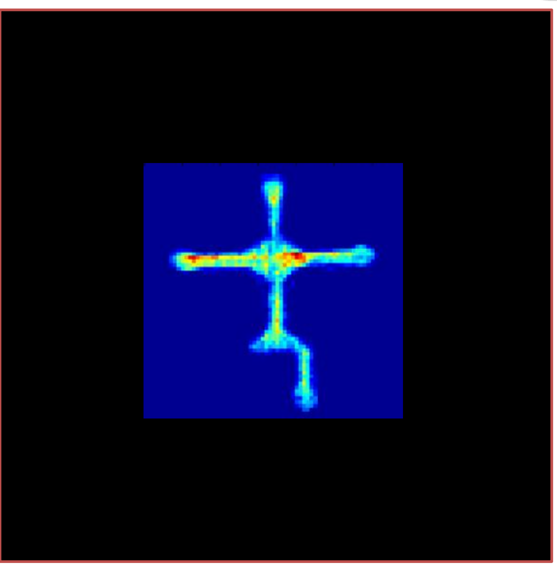
Guess object from autocorrelation & random phase

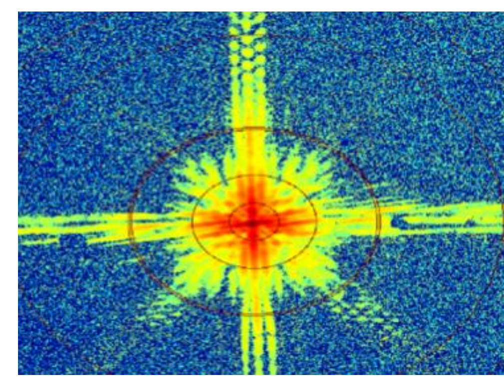
Take FFT

replace intensity with known pattern (Fourier space constraint)

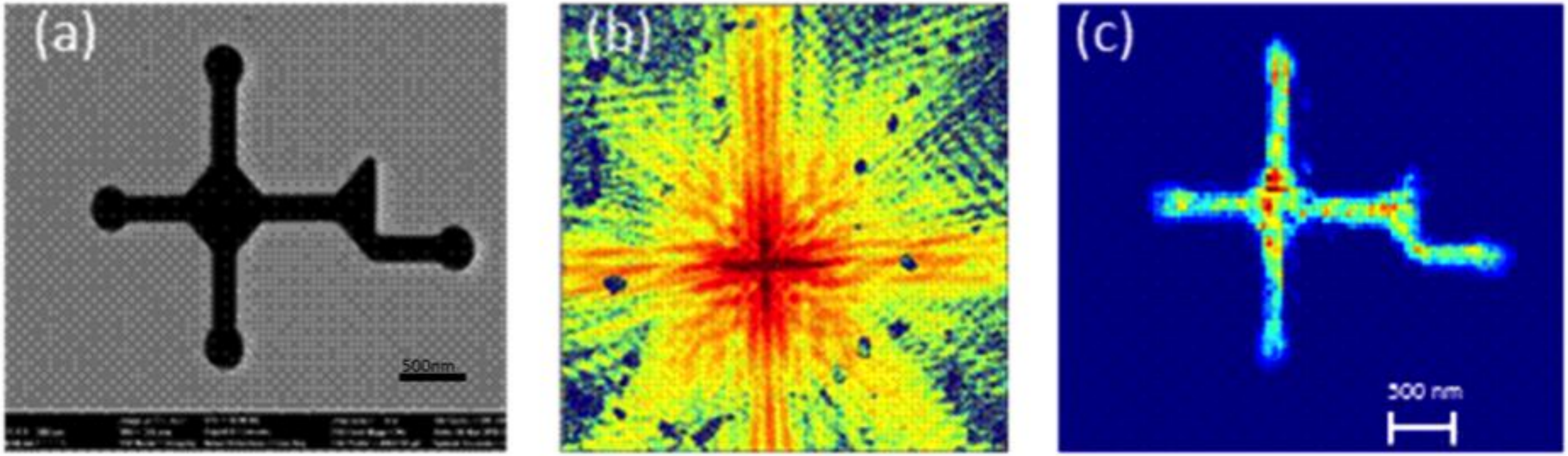
Reverse FFT

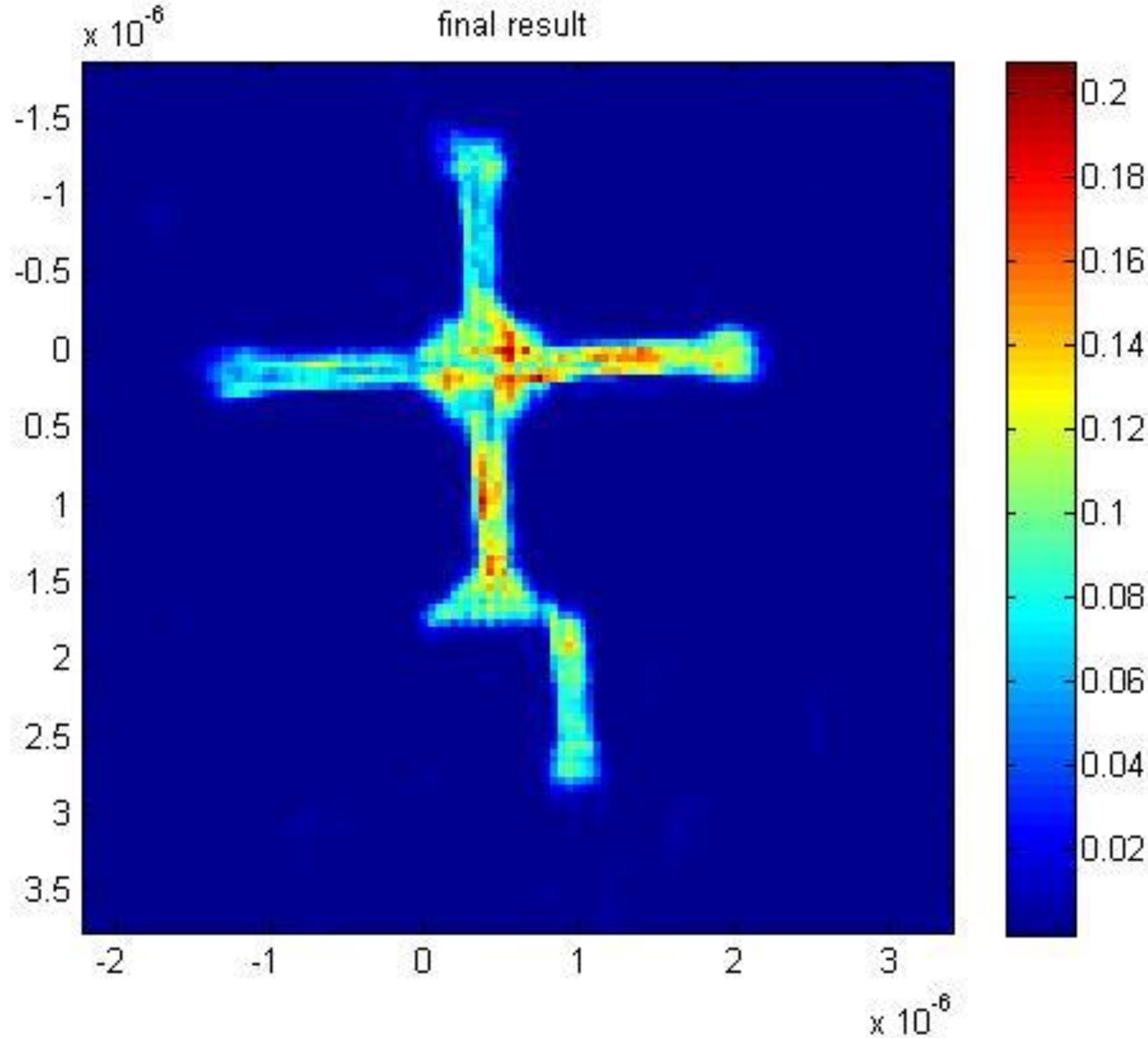
is the outer region black? – make it black (real space constraint)





Test results





Phase information is useful

UltraFast Xray Group

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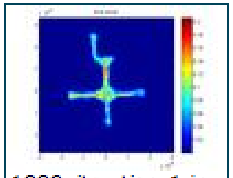
Reconstructing the new data

3rd August 2011 @ 22:52

Just a quick post to show some pictures from the reconstructions of the latest data and update everyone on progress.

Looking at data taken on 2nd August - the old 2um sample taken with a single wavelength. The diffraction patterns look v clean, with good detail right out to the edge - haven't worked out the resolution yet.

Tried to get Ben's CDI routine working, but it broke badly, and I couldn't fix it immediately, so i went back to my old routines (from 2 years ago! can't believe it's been that long) and got 'hio6.m' working. This is running on Boxer under Windows, but not using anything clever.



1000_iterations1.jpg

This one is a very plain HIO reconstruction, 1000 iterations after binning the data to 512x512. Shrinkwrapping every 100 (I think - notes at work right now). You can see that it reconstructs beautifully, and this happens every time - no need for multiple phases to get it to work.

(Once I'd remembered to background-subtract properly, of course - before that it all failed dismally)

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Ultrahigh 22 nm resolution coherent diffractive imaging using a desktop 13 nm high harmonic source

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Abstract: New diffractive imaging techniques using coherent x-ray beams have made possible nanometer-scale resolution imaging by replacing the optics in a microscope with an iterative phase retrieval algorithm. However, to date very high resolution imaging (< 40nm) was limited to large-scale synchrotron facilities. Here, we present a significant advance in image resolution and capabilities for desktop soft x-ray microscopes that will enable widespread applications in nanoscience and nanotechnology. Using 13nm high harmonic beams, we demonstrate a record 22nm spatial resolution for any tabletop x-ray microscope. Finally, we show that unique information about the sample can be obtained by extracting 3-D information at very high numerical apertures.

OCIS codes: (340.7460) X-ray microscopy; (100.5070) Phase retrieval; (340.7480) X-rays, soft x-rays, extreme ultraviolet (EUV); (190.2620) Harmonic generation and mixing.

electron lasers and high harmonic sources, and a wide range of successful image retrievals.

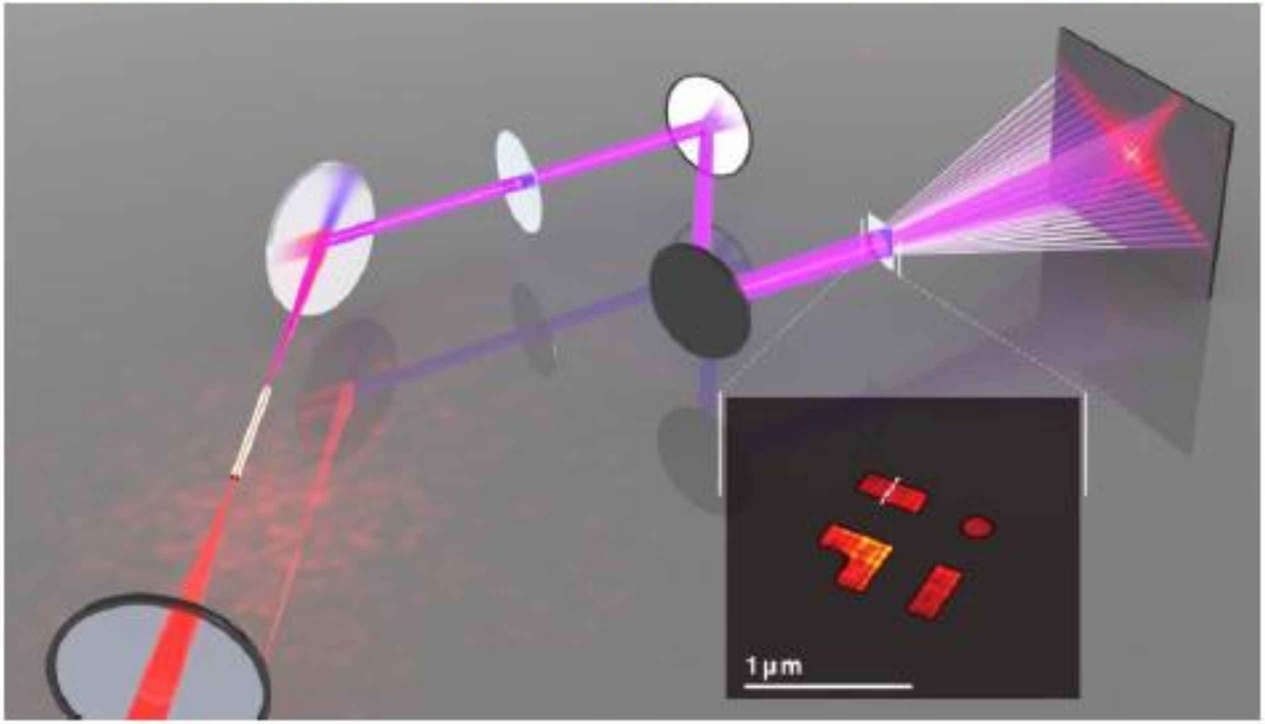


Fig. 1. Experimental configuration for tabletop soft x-ray coherent diffractive imaging. A femtosecond laser is focused into a gas-filled waveguide. Bright, coherent 13 nm high harmonic beams are produced and focused into the sample. The resultant diffraction pattern is captured on a CCD camera and the image is retrieved using an iterative phase retrieval algorithm.

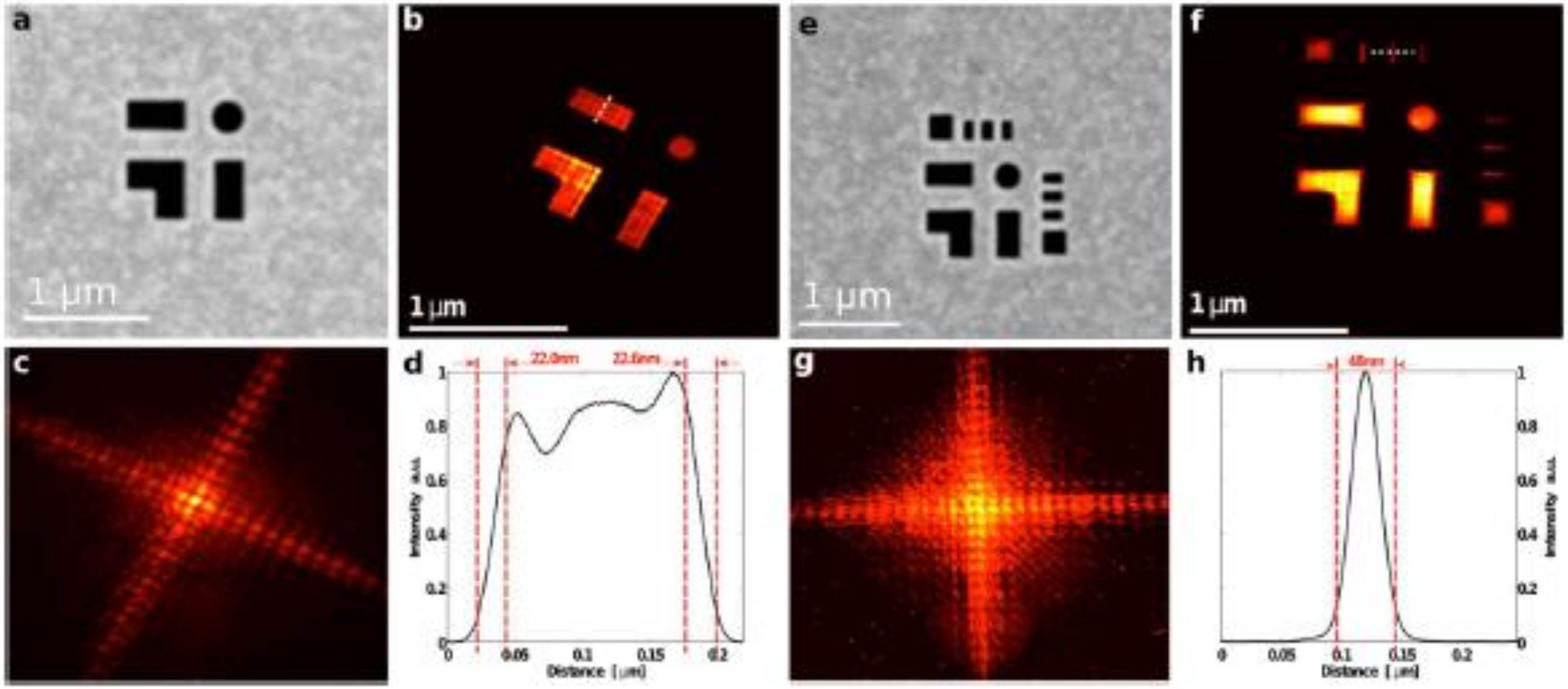
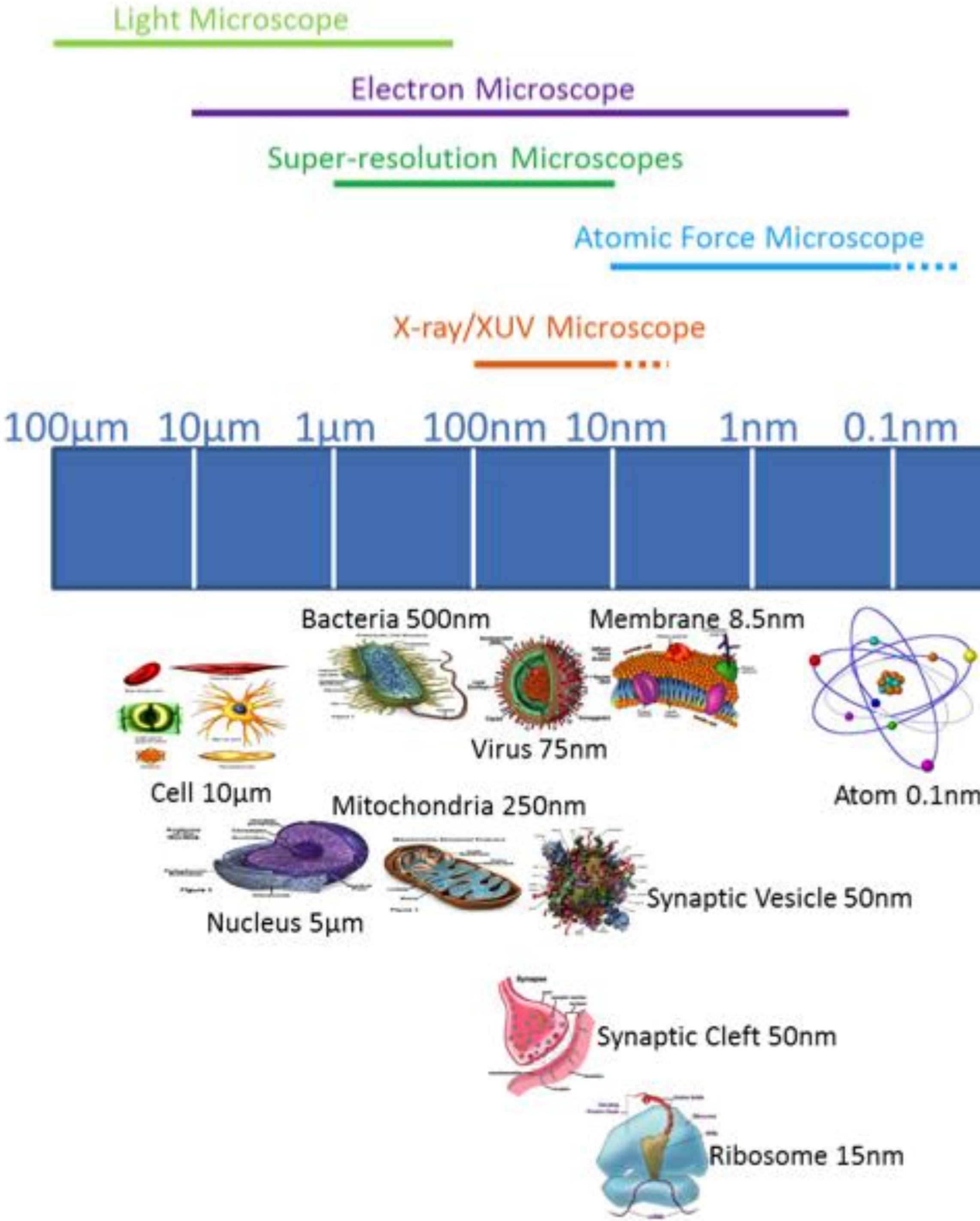


Fig. 2. Sub-25nm resolution confirmed using a knife-edge test. (a), (e) Scanning electron microscope images of samples J409 and J407 respectively. (b), (f) Object intensities reconstructed using the HIO algorithm. (c), (g) Scatter patterns for objects J409 and J407 respectively. (d) Lineout showing an edge with a ~22nm 10% to 90% dimension. (h) Object J407 displays minimum feature sizes of ~50nm (e^{-2} diameter) providing a rough estimate of resolution. The lineouts in (d) and (h) were taken along profiles marked in (b) and (f) by white dashed lines.



Light Microscope
Electron Microscope
Super-resolution Microscopes
Atomic Force Microscope
X-ray/XUV Microscope
100µm
10µm
1µm
100nm
10nm
1nm
0.1nm
Bacteria 500nm
Membrane 8.5nm
Virus 75nm
Cell 10µm
Mitochondria 250nm
Atom 0.1nm
Synaptic Vesicle 50nm
Nucleus 5µm
Synaptic Cleft 50nm
Ribosome 15nm

Biological Sample Preparation

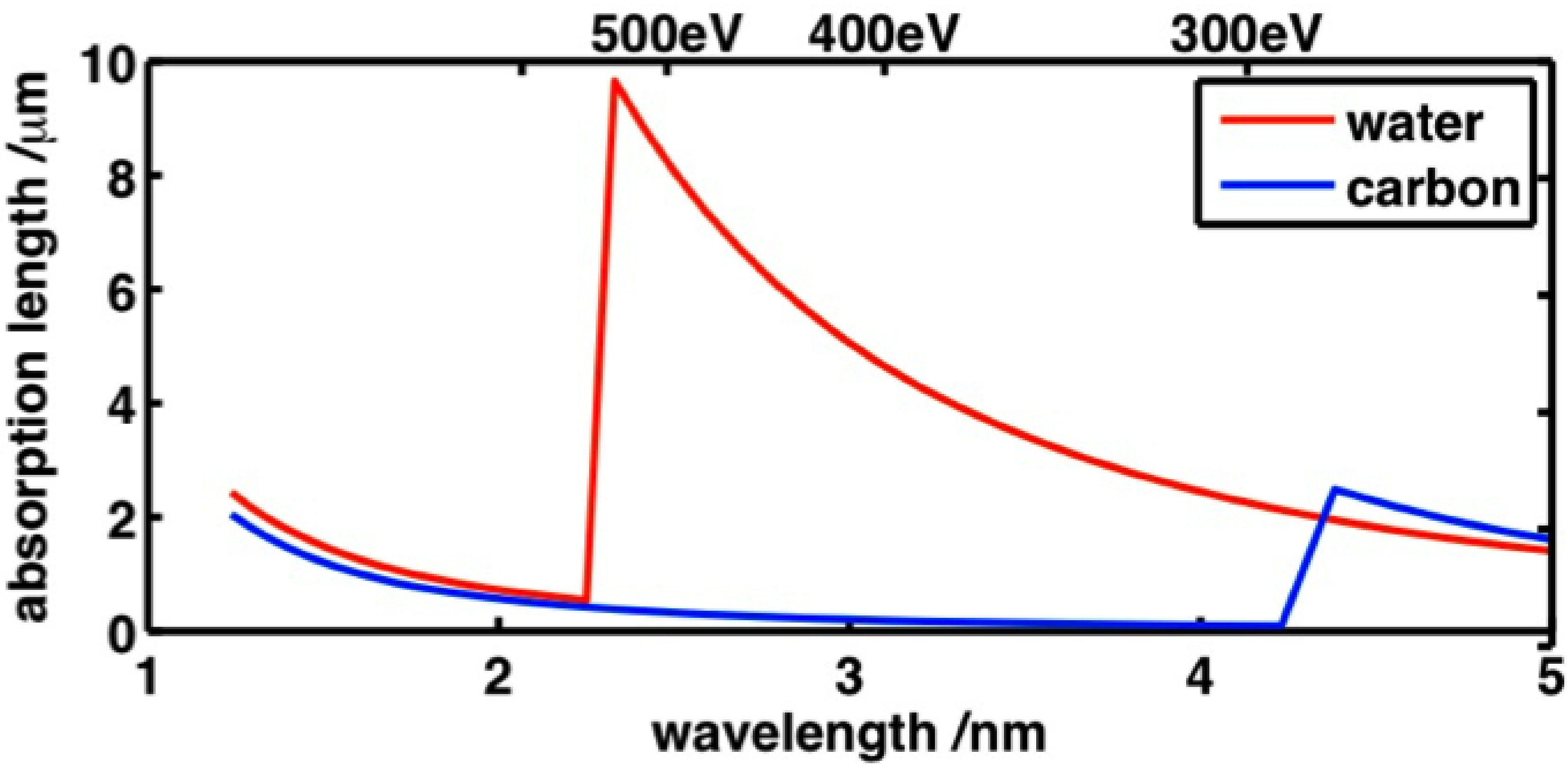


Fig 4: The water-window in the soft X-ray regime provides natural contrast for biological imaging

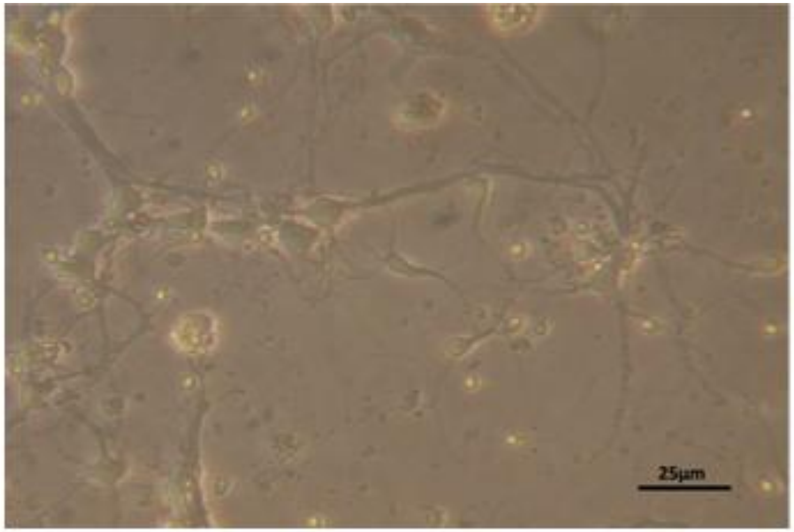


Fig 7: Light Microscope Image of Cultured Neurons(6DIV)

R L Card

Imaging neurons

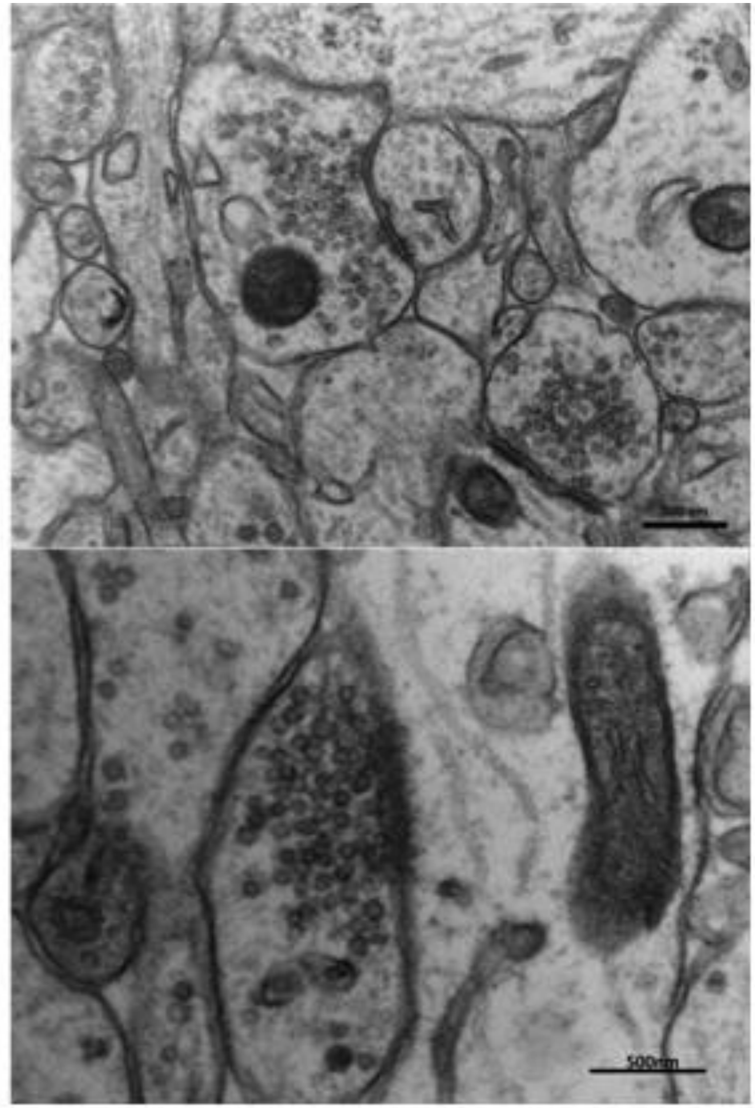


Fig 6: TEM images of synapses in hippocampal tissue from a CSP knockout mouse

R L Card and J L Bailey

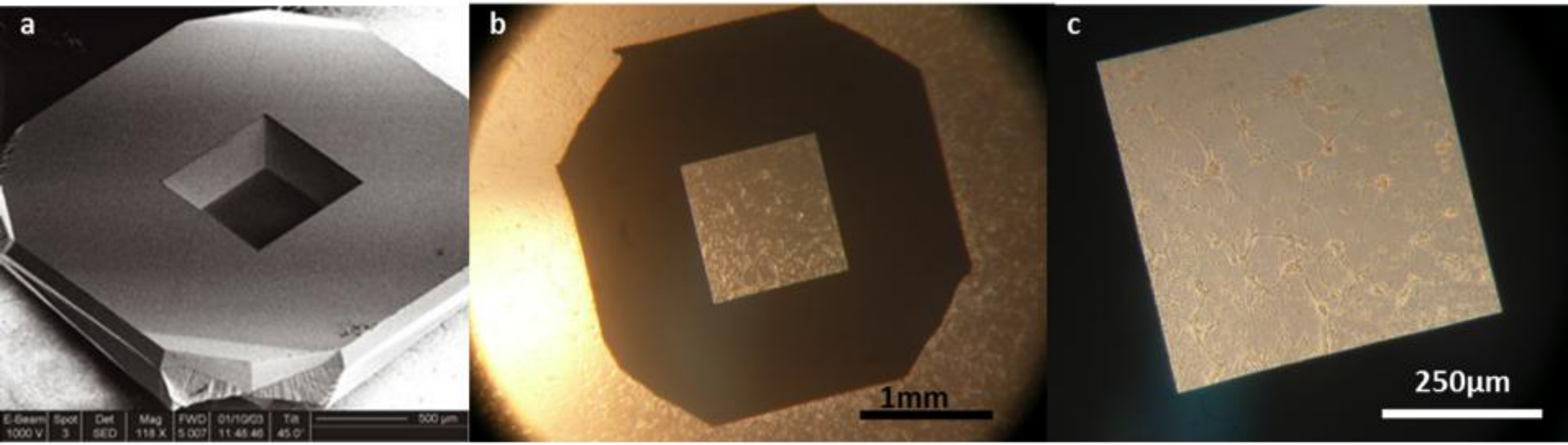


Fig 5: (a) A Si_3N_4 TEM substrate from Silson, Northampton
(b) A Si_3N_4 TEM substrate in a well of a standard 6 well cell culture dish
(c) Cultured cortical neurons (6DIV) adhered to the Si_3N_4 TEM substrate

Future

- Now developing the water window imaging at 4 nm
- Plan for 1 A coherent source

Southampton Ultrafast X-ray group