Imaging Challenges at Diamond

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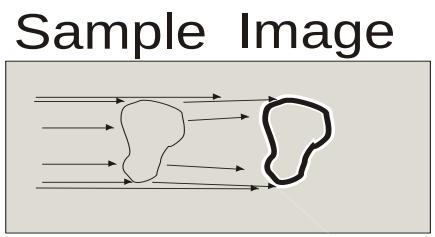
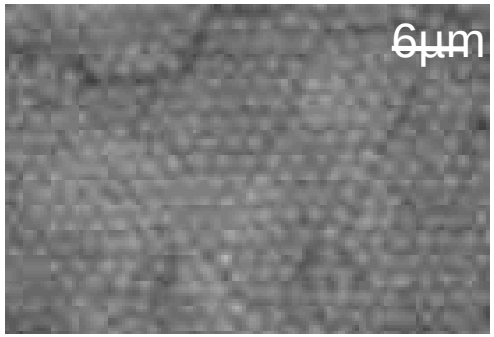
Bath 28th Jan – 1st Feb 2019

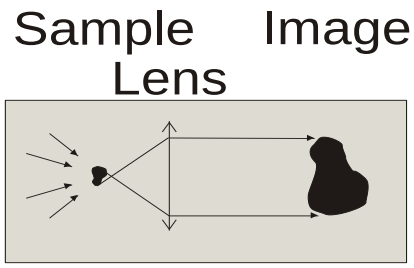
Imaging - Methods

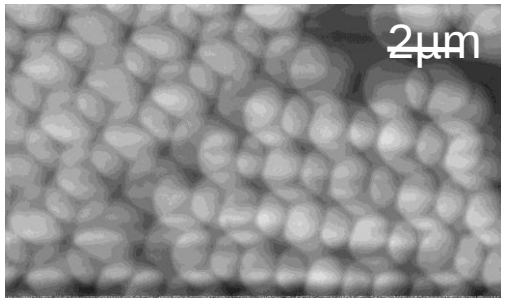
In-line phase contrast
-**µm** resolution
-easy to use
-large field of view



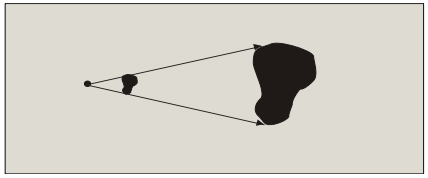


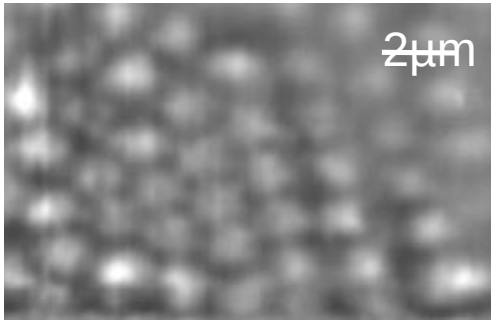
Full-field microscope
- **50nm** resolution
- imaging of phase objects
- combined methods





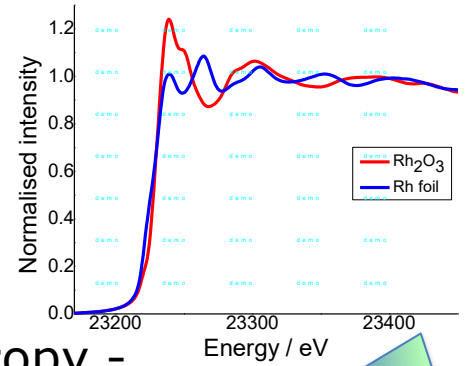
Cone-beam imaging
-**sub-µm** resolution
-dose efficient
-sub-100nm source



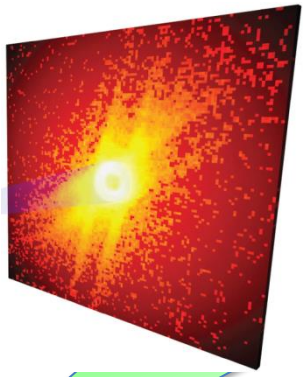


Full-field imaging with different spatial resolution

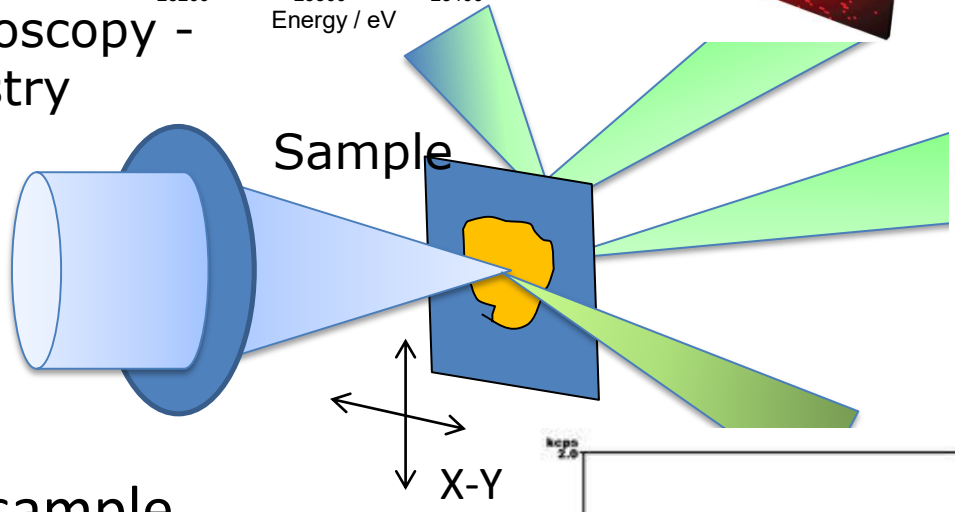
Scanning Probe X-ray Microscopy

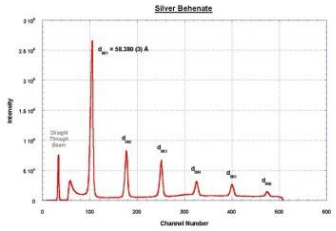


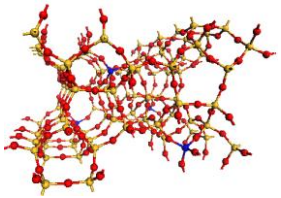
Spectroscopy - chemistry



Ptychography/Imaging – Spatial Organisation

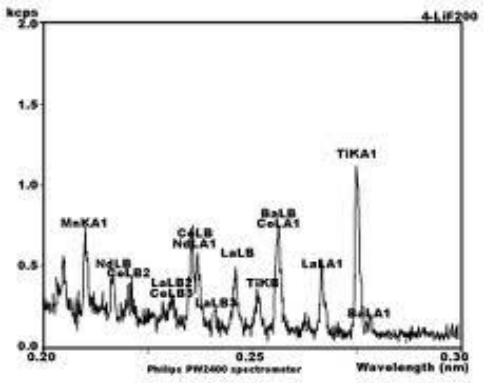






Diffraction - structure

Scan the sample,
Scan the energy,
Rotate the sample



XRF - Elemental composition

Sampling

Science focus:

- Battery and electrochemistry challenge

Imaging Experiments at Diamond:

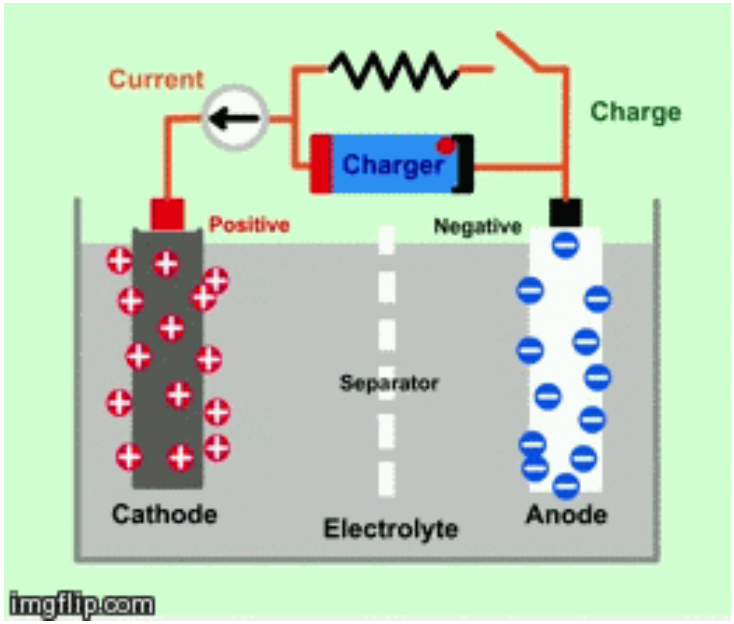
- Challenges of scanning experiment
- Extending to 3D and 4D tomography experiments

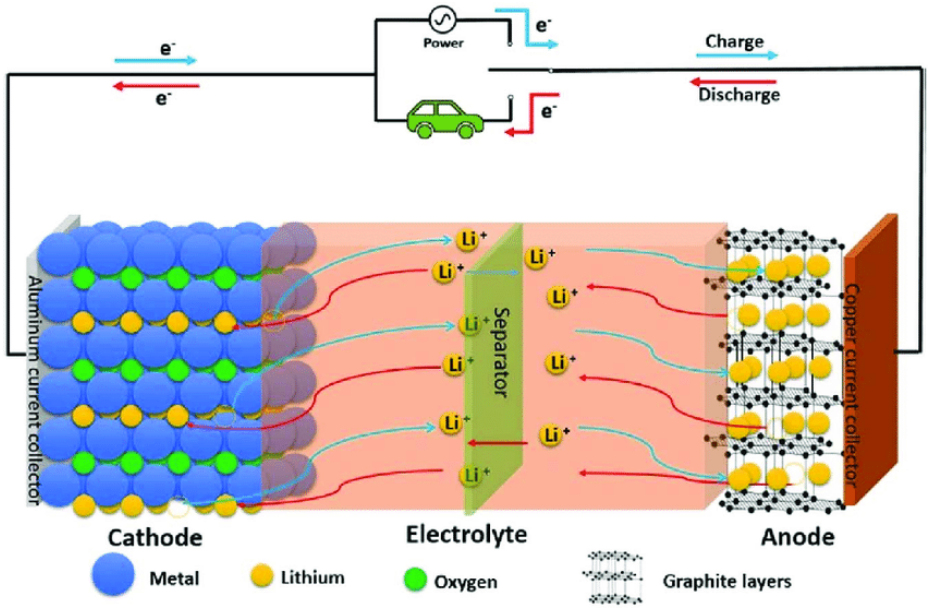
- Challenge - how can make experiments faster ?

Faraday Challenge

- Government's programme to develop cost-effective, high-performance, durable, safe, low-weight and recyclable batteries.
- The ambition of the programme is to make the UK the go-to place for the research, development, manufacture and production of novel battery technologies for both the automotive and the wider relevant sectors.
- £246 million in funding
- £42 million allocated to first 4 projects (2017)
- Call for next 4 projects just issued
- Ability to characterize battery materials is a key aspect

How does a battery work ?



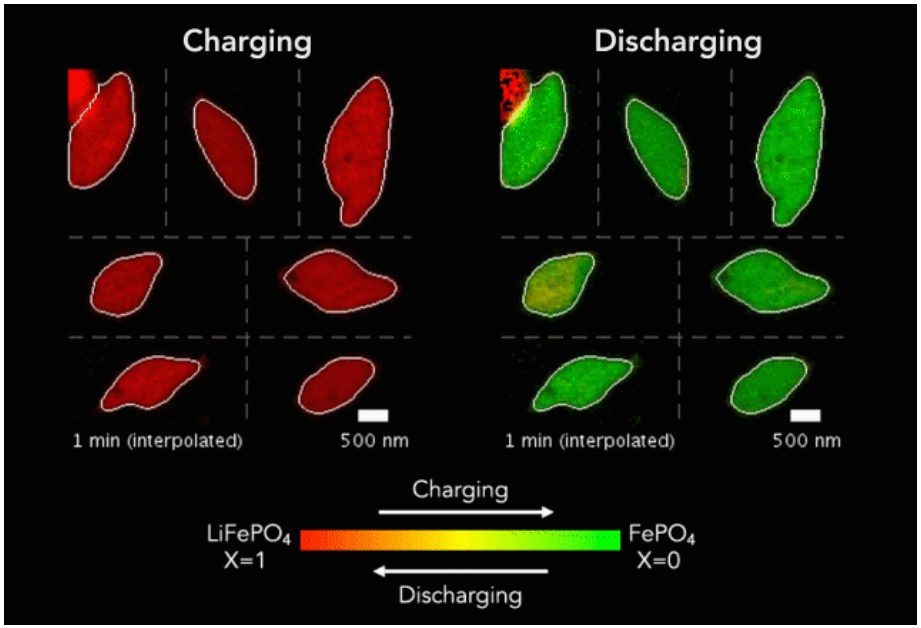


Why does it fail ?

- It’s a chemical reaction – lots of reasons..
  - The internal structure of the particles
  - Defects
  - Contaminants
  - The charge/discharge cycle time

We need to be able to follow/measure changes to understand the underlying mechanisms

X-ray measurements..



"Origin and hysteresis of lithium compositional spatiodynamics within battery primary particles", J. Lim,Y. Li, D. H. Alsem, H. So, S. C. Lee, P. Bai, D.A. Cogswell, X. Liu, N. Jin, Y. Yu, N. J. Salmon, D. A. Shapiro, M. Z. Bazant, T.Tyliszczak, W. C. Chueh, *Science* 05 Aug 2016

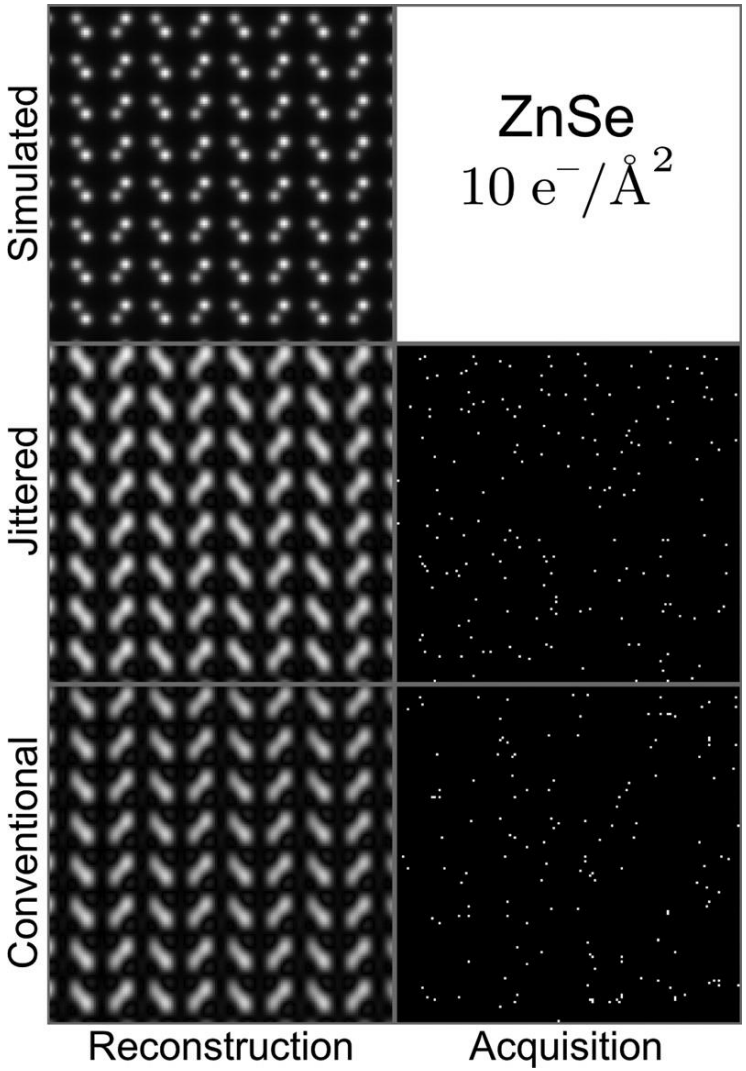
Only in the last 2-3 years that people have started to successful image systems in operation

The Challenge

- How to perform experiments fast enough to watch evolving systems in operation

- Example –
  - Experiment in March 19 which takes 20 hours to acquire a single 3D data set from a battery sample
  - A 5 day expt will allow you to look at 1 sample and 5 conditions
  - A moderate 3-4 fold increase in speed would make a substantial impact

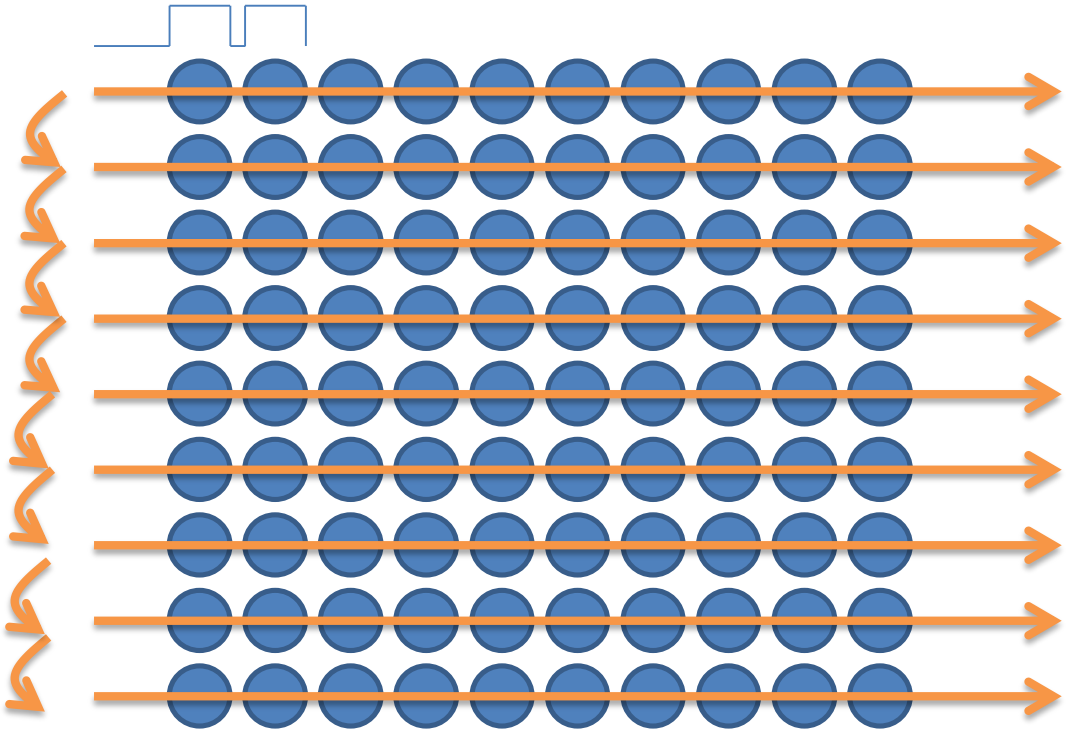
Examples in electron microscopy..



- Appl. Phys. Lett. **112**, 043104 (2018);
- Maths: you can throw away data !
- Reality: How?

X-ray scanning spectroscopy experiment :

Current experiment….



Raster scan a sample at constant velocity

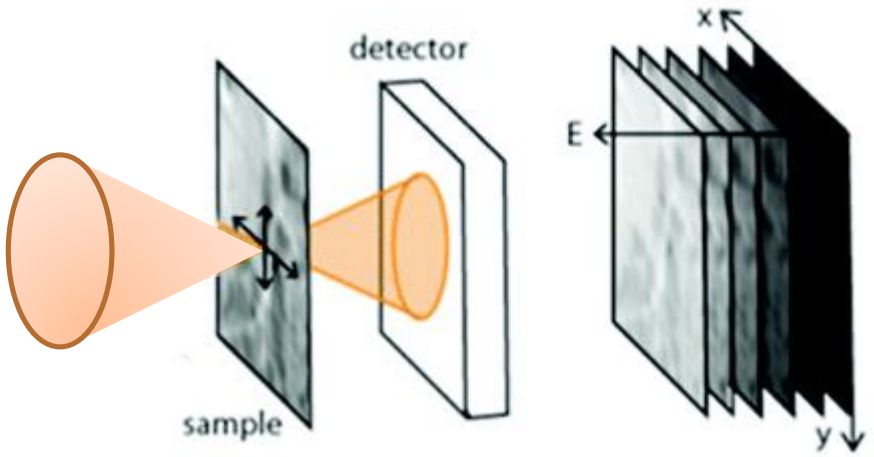
Collect signal at fixed time intervals

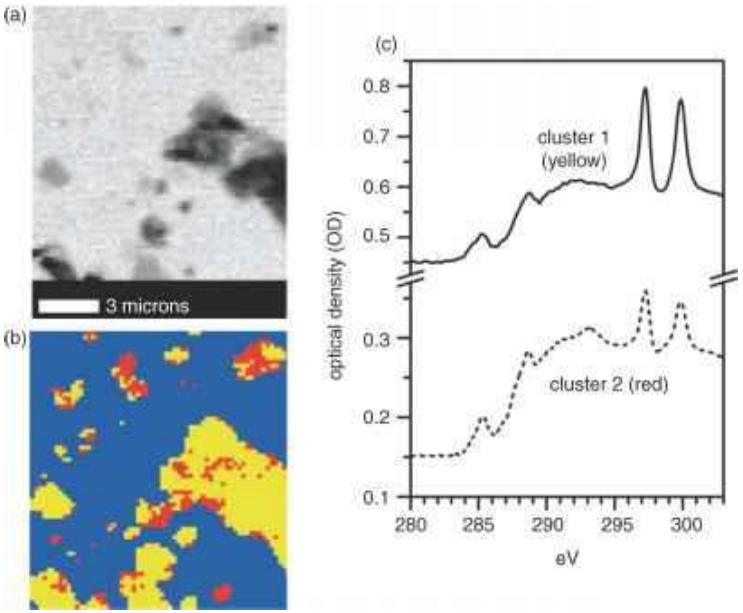
Rotate sample or change energy to get 3D or spectroscopic information

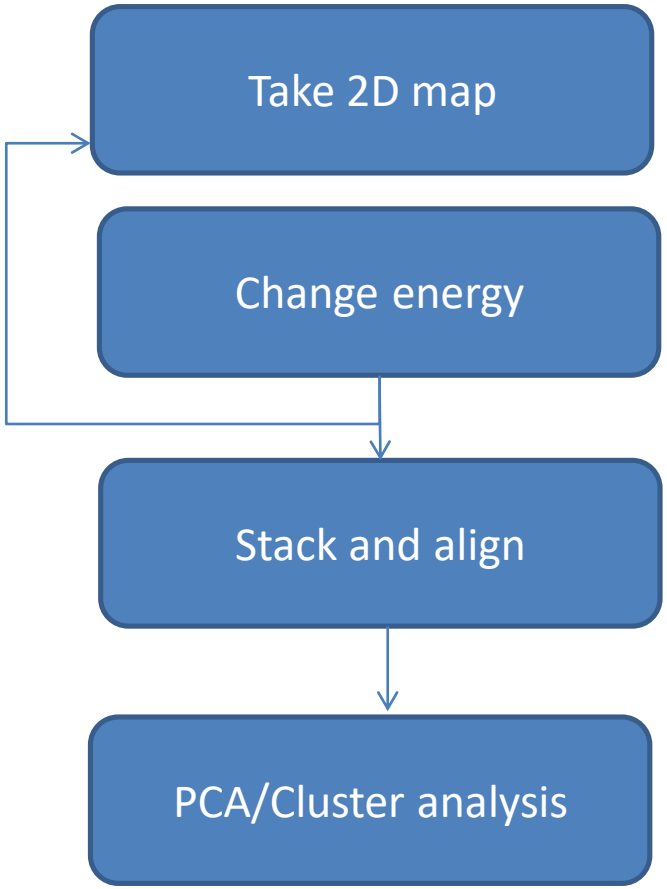
Can turn/on/off beam

Sample can follow a continuous path – spiral, raster, snake – dictated by motorized motion

Take maps at a range of energies

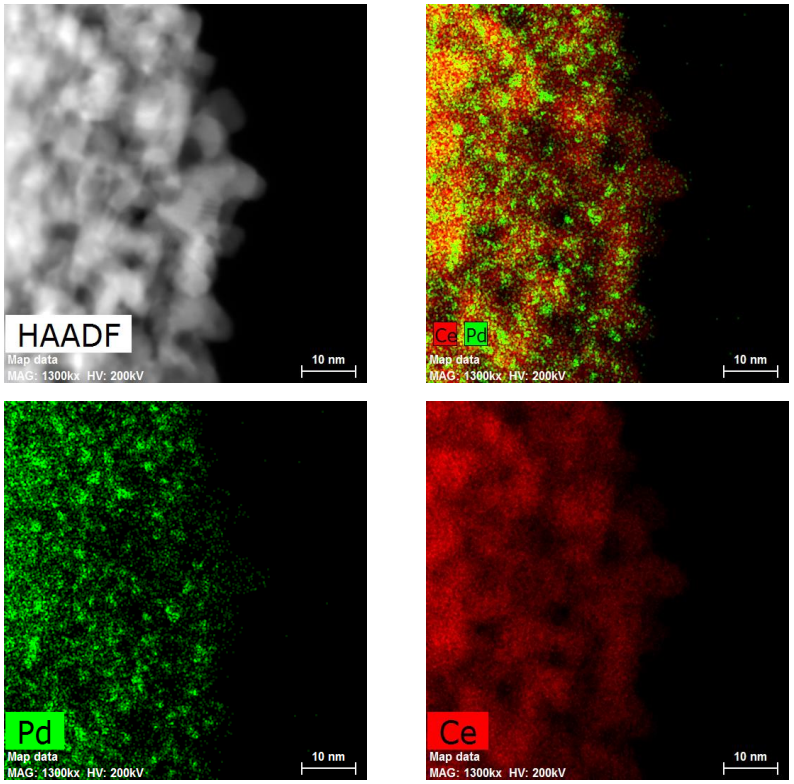


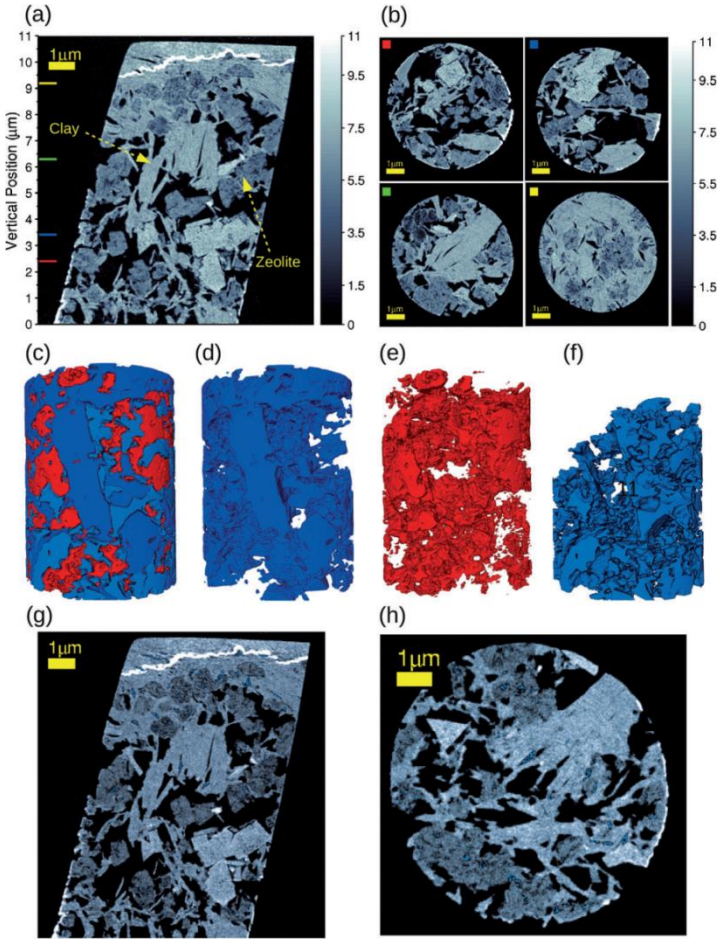




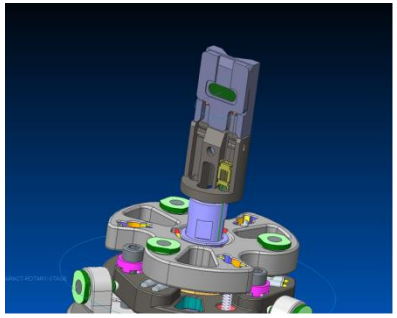
Multimodal methods – 3D/4D

EM elemental mapping of Pd/CeO_2





Bulk EXAFS / X-ray micro and nanostructure imaging in 2D and 3D/ EM atomic resolution



3D Pore Structure of catalysts from electron-density by ptychography at cSAXS (da Silva et. al ChemCatChem 2015, 7, 413 – 416) 3D rendering of the pores in light blue, the zeolite type Y in blue, and the metakaolin clay in red.

Sampling

For any given experiment:

- Can we produce an optimized sampling scheme to reduce the experiment time?
  - With the data provided – could we have measured faster ?
  - Optimal Trajectory from scan to scan to optimize collection?
  - How to apply compressed sampling to hyper-spectral data ?
  - Optimal scan sequence ? (currently sequential energies…)

For a set of measurements

- If I measure one full data set can I reduce the time for the subsequent sets – e.g. when charging and discharging the battery ?

- If something drastic happens during battery cycling would the scheme capture this ?

Data Live Example
Sampling data set 1