

# Microscopy & Microtechniques

## Pioneering Centre for Nanoscale Materials Research Launched on Harwell Campus

Professor A I Kirkland, Department of Materials, Parks Road, Oxford OX1 3PH  
Science Director, Electron Physical Science Imaging Centre, Diamond Lightsource Ltd., Diamond House, OX11 0DE

Industry and leading scientists gathered at Harwell Science Campus in September for the opening of a pioneering new centre for the study of nanoscale materials, set to boost the UK's world-leading science and technology infrastructure.

The electron Physical Sciences Imaging Centre (ePSIC), a collaboration between Diamond Light Source, the University of Oxford and global speciality chemicals company, Johnson Matthey, contains two state-of-the-art electron microscopes for the physical sciences, designed to provide scientists with atomic level images of a range of technologically important materials. Funding for the new centre based at Diamond included contributions from the Wellcome Trust, Jeol Ltd, the Biotechnology Biological Sciences Research Council and the Science and Technology Facilities Council.



Andrew Harrison (CEO at Diamond Light Source) and Angus Kirkland (Science Director at ePSIC)

The Harwell campus is home to Diamond, the UK's synchrotron, where currently 26 experimental stations (beamlines) are operational with funding in place to increase this number to 33 by 2020.

As part of Diamond's pioneering hard X-ray nanoprobe beamline (I14) and electron microscopy centre, Oxford University contributed a unique JEOL 300kV electron microscope dedicated to atomic scale imaging at world-leading resolution and Johnson Matthey installed a world-leading Jeol double-EDX and EELS capable microscope dedicated to chemical analysis with atomic scale resolution. Collaborations between Johnson Matthey, Oxford University and Diamond's I14 beamline will facilitate the interchange of samples between these systems and enable analyses at near-duty catalytic conditions to observe the influence of chemical and thermal challenges on material structure.

### World Class Facilities

Offering unrivalled facilities for research across the biological and physical sciences the hard X-ray nanoprobe will take structural analysis with detailed element mapping to the highest spatial X-ray resolution available anywhere in the world. The new microscopes will complement other advanced electron microscopes under the umbrella of the new centre, forming part of a National Facility for Cryo-Electron Microscopy (eBIC) for biological sciences.

Robert MacLeod, Chief Executive of Johnson Matthey said: "We are excited to be part

of this unique collaboration for UK science. R&D is at the heart of our future success and this new electron microscope will enable Johnson Matthey's scientists to actually 'see' and analyse individual atoms in real time within the structure of our materials. This unrivalled capability in physical sciences is crucial for us in driving innovation in new and existing products for our customers."

Professor Andrew Harrison, CEO at Diamond, explained: "Diamond is a world-leading centre for visualising physical and biological materials at the atomic and molecular level and it makes sense to complement our capabilities with electron microscopy. Information gained will give us microscopic properties and valuable insight into the electronic structure of materials, strength and much more. The centre will be opened to all and will operate like our beamlines, through both academic peer review and proprietary access. As a result, the Diamond synchrotron will become the first in the world to house such a complementary set of techniques."

Unveiling a plaque to commemorate the official opening; Sir John Meurig Thomas, formerly Director of the Royal Institution of Great Britain said; "This unique facility brings together academia, industry and government research laboratories in a fantastically advanced manner and marks an exceptionally exciting period for British, European and World science. It is highly appropriate that these new state of the art electron microscopes are located, not more than 17 miles away, from where in 1664 Robert Hook published his 'Micrographia', the first important work on microscopy (the study of minute objects by means of a microscope). He stated that it is the prerogative of all humans above all creatures to behold, consider, compare, alter, assess and improve on the works of nature. This is the recipe for going from pure to applied science. This facility will continue his work and give us many new and invaluable insights into the invisible world."

Angus Kirkland, Professor of Materials at the University of Oxford and Science Director at ePSIC, said: "This centre will provide a world class capability for materials imaging and the collaboration with Johnson Matthey will bring technologically important problems into focus to answer fundamental research questions." He described further how ePSIC's electron microscopy can help research scientists worldwide, summarising some current studies at ePSIC.



Sir John Meurig Thomas (formerly Director of the Royal Institution of Great Britain)



## Modern TEM Capability

"Electron Microscopy has evolved from laboratory based equipment housed in and operated by individual institutions to a model whereby National facilities equipped with state of the art instrumentation supported by staff scientists provide open access to a wide user base.

"Modern transmission electron microscopes (TEMs) provide complementary capability to that provided by synchrotrons and free electron lasers. The current generation of microscopes provide source brightness's intermediate between those of synchrotrons and free electron lasers but with probes that are two orders of magnitude smaller (100pm or less) and with comparable energy resolution (10meV or less). TEMs also offer in-situ capability in which samples can be studied under liquid and gas atmospheres approaching those used in many commercial processes. Electron tomography provides 3D information that is complementary to that acquired using X-rays but at a spatial resolution that can be several orders of magnitude higher. For many materials the beam damage is 1000 X less for 100keV electrons than for 1.5Å wavelength X-Rays which is critical for studies of biological materials.

"State of the art instruments such as those at ePSIC are expensive so it is important that they are accessible in a centralised facility and thereby provide tangible benefits and access to the wider science community. ePSIC will enable academic and industrial scientists from around the world to access the state of the art instrumentation following peer review. This will ensure that ePSIC attracts the best science and supports the most promising research projects. The instruments in the centre will provide key structural information that can be used to develop enhanced 'smart' materials for use in consumer technology, next-generation transportation and engineering and new catalysts for green technology and automotive applications. This will enable scientists working in the centre a means to visualise structures at atomic-resolution to address some of the great technology and engineering challenges of our time.

## ePSIC Research

"Initially research at ePSIC will concentrate on studies of a number of key materials challenges.

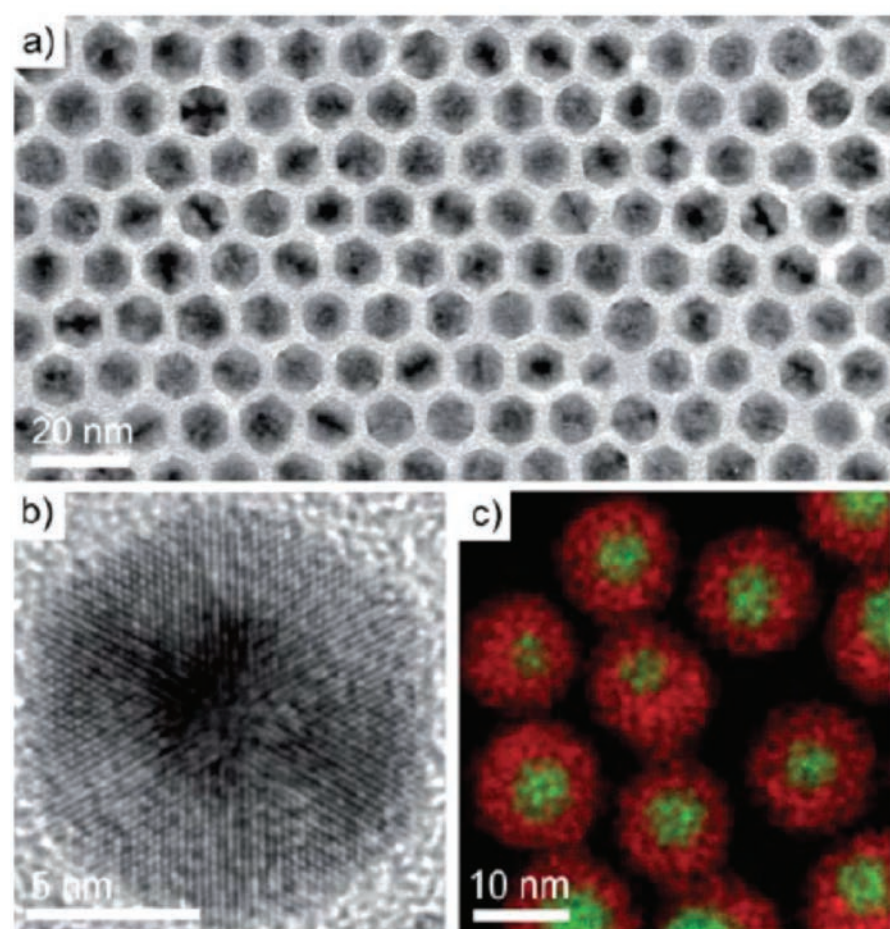


Figure 1. a)

TEM image of an array of 11.4 nm highly faceted Au-Pd core-shell nanocrystals. b) High-resolution TEM image of a single Au-Pd core-shell nanocrystal, showing a clear contrast difference between the dark core (gold) and lighter shell (palladium). Lattice fringes correspond to an icosahedral structure oriented along  $\langle 111 \rangle$ . c) Chemical map shown as an overlay of gold and palladium signals, confirming the core-shell structure.

From: Gold-Palladium Core-Shell Nanocrystals with Size and Shape Control Optimized for Catalytic Performance

Anna M. Henning, John Watt, Peter J. Miedziak, Soshan Cheong, Marco Santonastaso, Minghui Song, Yoshihiko Takeda, Angus I. Kirkland, Stuart H. Taylor, and Richard D. Tilley, *Angew. Chem. Int. Ed.* 2013, 52, 1477. Reprinted with permission from Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim.

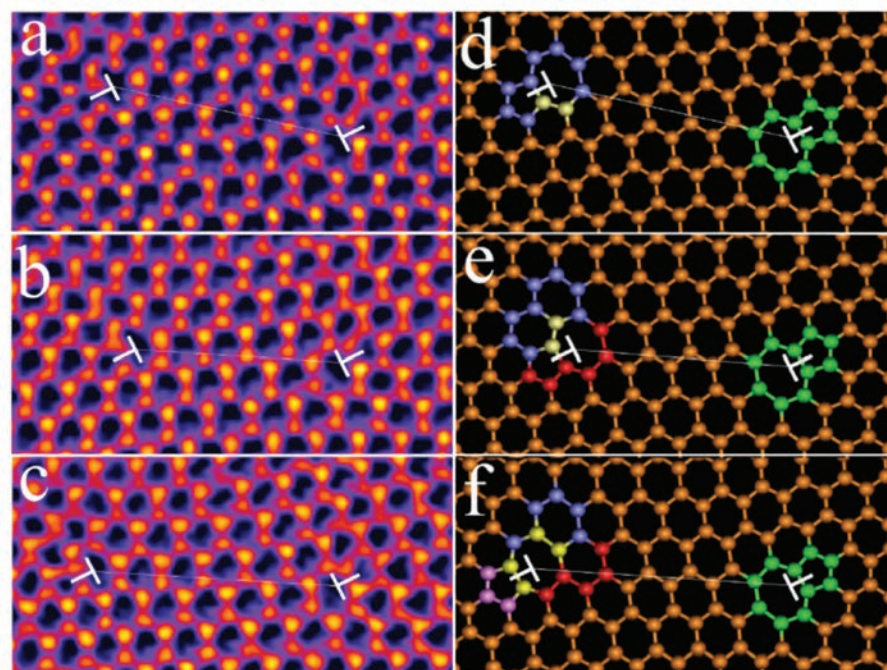


Figure 2. Real time dislocation dynamics in Graphene. TEM images showing changes in the position of an edge glide dislocation with time under continuous electron beam irradiation. (a) Time = 0 s. (b) Time = 141 s. (c) Time = 321 s. (d) - (f) Atomic models illustrating the structures inferred from (a)-(c) respectively.

From: Dislocation-Driven Deformations in Graphene, Jamie H. Warner, Elena Roxana-Margine, Masaki Mukai, Alexander W. Robertson, Feliciano Giustino, Angus I. Kirkland, *Science*, 337, 209, 2012. Reprinted with permission from AAAS.

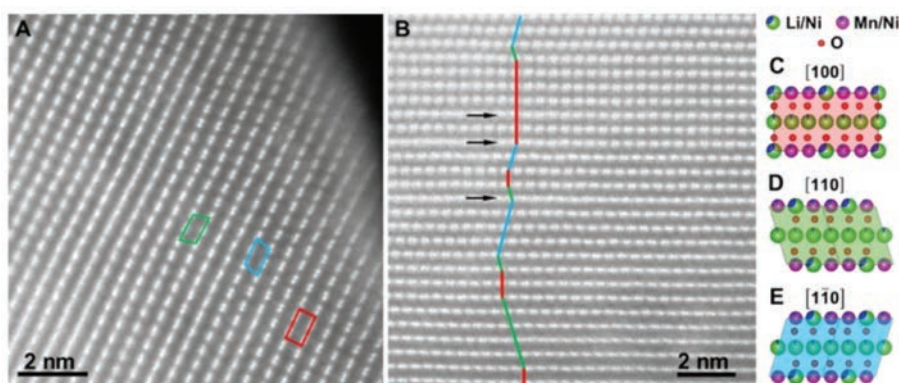


Figure 3. Experimental TEM images, showing the structure of  $\text{Li}[\text{Li}_{0.2}\text{Ni}_{0.2}\text{Mn}_{0.6}]\text{O}_2$  consisting of different stacking of three main projections of the monoclinic unit cell [100] in red, [110] in green and in blue. The crystal domains of these three main variants are shown in (b) by colored lines. Flips along the (001) planes, reflection twins (flips between [110] and) and disordered transition metal layers (arrows) are generally observed. The unit cells in the different orientations are shown in (d-e).

"The global catalysis market is currently worth ca. \$20 billion and is predicted to grow by between 5-8% to 2020. The instruments at ePSIC will enable the study of advanced catalyst structures including those under operating conditions. In turn this will enable UK industry to lead in the development of new catalytic materials that maximise performance whilst minimising our reliance on scarce resources. A specific example is the development of 'core shell' materials, grown under controlled conditions which optimise the use of precious metals such as platinum in controlled geometry nanoparticles (Figure 1).

"Two dimensional materials including graphene offer new possibilities for advanced structural materials display technologies and electronic devices. The UK leads the world in this research area and ePSIC will provide the means to provide atomic resolution images of the structures of defects and their motion in real time which determine many of the key device properties in these materials (Figure 2).

"Materials for energy storage are essential for the commercial development of new battery technologies and for applications in fuel cells. Many of the most promising materials are complex oxides which undergo subtle structural changes during charging and discharging cycles, directly impacting their performance. Many of these changes can only be followed by direct imaging at atomic resolution and the instruments at ePSIC are already providing new data in this area (Figure 3).

"Finally, ePSIC aims to act as a focus for new developments in electron microscopy ensuring its long term sustainability as a National resource. In this area we already support scientists from industry and academia working together to develop new instrumentation, control and automation and theory and computation and the unique co-location of these at a National facility represents a new model for the development of electron microscopy in which the UK has taken the lead."

For further information email: [diamondcomms@diamond.ac.uk](mailto:diamondcomms@diamond.ac.uk)



Read, Share and Comment on this Article, visit: [www.labmate-online.com/articles](http://www.labmate-online.com/articles)