Adding Electrons to Synchrotron Imaging Synergies

Dr Cecilia Sánchez-Hanke, Dr Christopher Allen

Now celebrating its tenth year of research and innovation, Diamond Light Source, the UK's synchrotron radiation facility, is building on the synergies with electron microscopy with the goal of allowing researchers to study samples at a large range of length scales.

Two new centres, known as the electron Physical Sciences Imaging Centre (ePSIC) and the electron Bio-Imaging Centre (eBIC), have both been constructed with the goal of making Diamond a key facility for high-resolution imaging in the UK. Diamond also has at present 28 operational beamlines that make use of synchrotron light to investigate a range of scientific challenges across the physical and life sciences, as well as contributing to cultural heritage and food science.

Aerial view of Diamond Light Source.

ePSIC is a collaboration between industry partner Johnson Matthey, the University of Oxford and Diamond Light Source. The facility incorporates two high-end JEOL instruments, an ARM 200 and a GRAND ARM 300. Dr Cecilia Sánchez-Hanke, discusses some of the opportunities this new facility presents Diamond and Dr Chris Allen explains the priorities for his research.

"Combining synchrotron microscopy techniques with these fantastic electron microscopes is truly exciting, as it means that at Diamond, our microscopy imaging instruments will cover length scales from the micrometre range to the Ångstrom - an impressive five orders of magnitude in length scale. We're hoping to pioneer combining these different experimental methods and help to drive the field forward," said Dr Cecilia Sánchez-Hanke, Operations Director at ePSIC and Scientific Operations Coordinator at Diamond Light Source.

"Although synchrotrons and electron microscopes have been around for some time, we're optimistic that grouping them in the same facility will promote new interactions and collaborations between both user communities, and lead to collaborations where the use of both instruments is required to provide the final solution.

Ideally, this will mean that experiments at ePSIC and across Diamond will be carried out on the same sample, under the same environments. This is a complicated challenge – but Diamond and industry partner Johnson Matthey are already collaborating together with specialized specimen holder manufacturer DENS solutions on the development of suitable new technology.

Open Science

Diamond has a long-established process whereby the facility is free at the point of access for researchers who will openly publish their results. In order to get instrument time at Diamond, researchers submit a proposal that is scientifically assessed by a panel of independent peer reviewers. ePSIC follows the same access model, meaning that we've been able to effectively organise time for our potential users and provide them with access to all the support facilities Diamond offers.

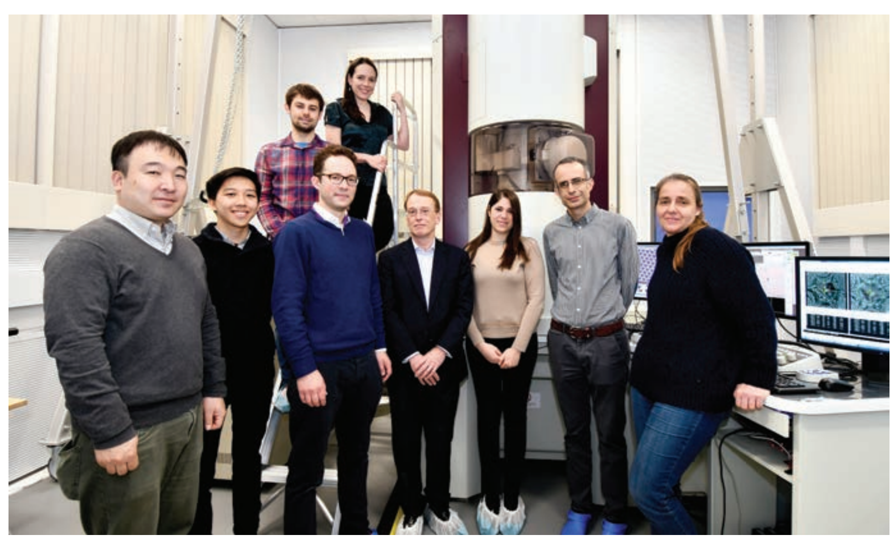
Both instruments are housed in a building external to the main synchrotron building, along with eBIC and the I14 Hard X-ray Nanoprobe beamline. In order to image at atomic length scales the instrumentation has to be incredibly stable and a great deal of work has gone into the design of the building to ensure the microscopes sit in the best possible environment and can operate at their highest spatial resolution.

The microscope rooms are vibration isolated from the rest of the building and equipped with radiant panels to control the room temperature, acoustic panels, and coils are installed in the walls as part of an active field cancellation system.

ePSIC is a unique opportunity for experts from both the electron microscope and the X-ray user communities to work together to solve new scientific challenges and to drive technical innovations in both fields. The similarities in the signals that electrons and photons generate when interacting with a sample provides a great opportunity for synergies between the two fields, including detector technologies, sample holders, and sample environments.

In the area of detector development, experience is already being used in a number of projects. Johnson Matthey have worked with Diamond's I14 beamline and DENS Solutions to develop a sample holder that allows them to investigate the same catalytic processes on the same sample using electron microscopes and an X-ray beamline with identical sample environment conditions.

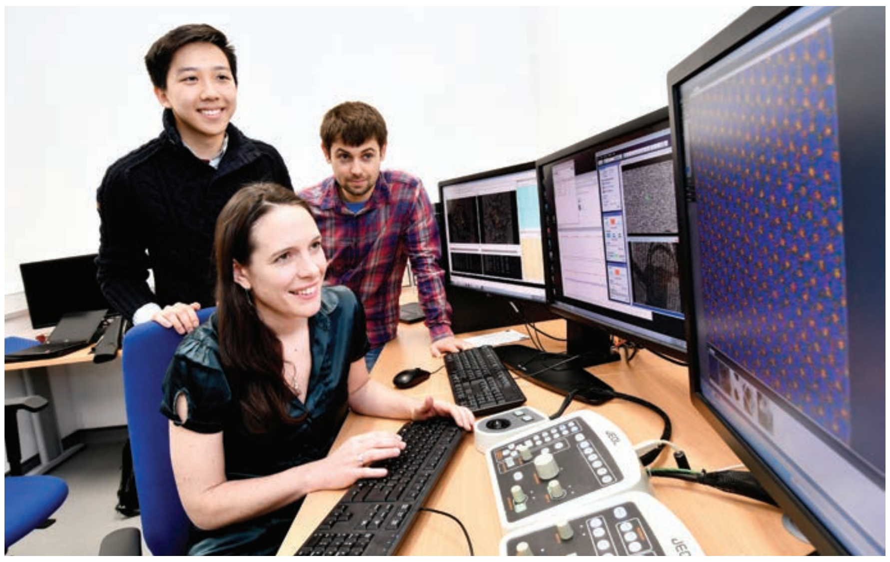
The synergies between photons and electrons mean that Diamond's Detectors and Data Acquisition groups, who have previously worked on the Medipix3 detector, a product of an international collaboration that Diamond is part of, are now working with ePSIC. Medipix3 detectors are used on a number of Diamond's X-ray beamlines in mapping-style experiments, so many commonalities are present with the types of experiment performed using the electron microscopes at ePSIC.



First users at ePSIC, from the National Graphene Institute, pictured with the team from ePSIC. Left to right: Hidetaka Sawada, Lan Nguyen, Aiden Rooney (rear), Christopher Allen (front), Sarah Haigh (rear), Angus Kirkland, Emanuela Liberti, Mohsen Danaie and Cecilia Sánchez Hanke.

Preliminary work has managed to allow researchers to utilise many of the software tools developed at Diamond to analyse their results, with the aim of extending this to deal with the unique features of electron data in the future.

More electron microscope facilities have already, or are in the process of, developing 'user facility' models similar to that which we operate here at Diamond. As we see these developments quickly expand, collaborations between the facilities will also increase how much the facilities share their experiences in sample preparation, sample environments, and data acquisition and analysis, leading to further technological evolution.



ePSIC (electron Physical Sciences Imaging Centre) welcomes first users from The National Graphene Institute at The University of Manchester. Left to right: Lan Nguyen (rear), Sarah Haigh (front) and Aiden Rooney (rear).

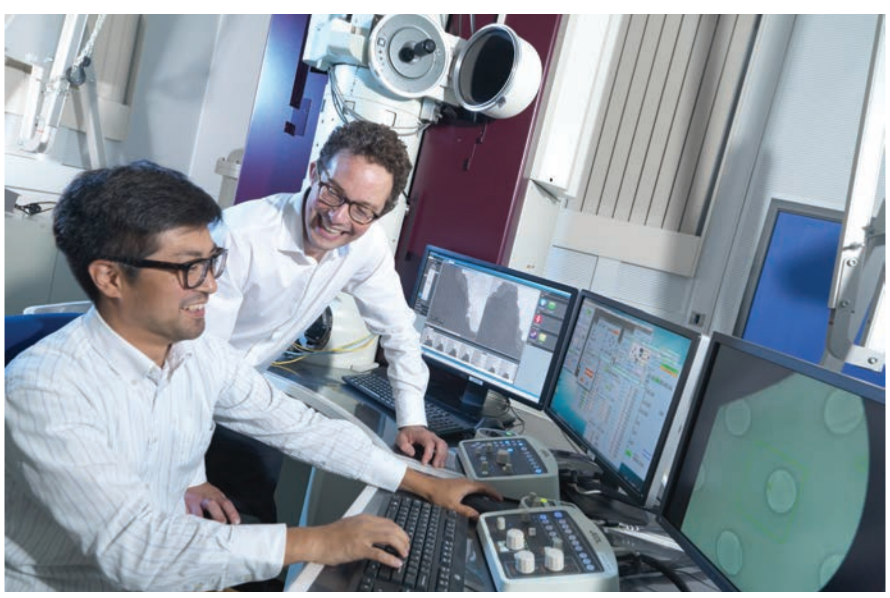
Establishing collaborations between the experts in their fields, who have solved challenges with particular instruments, is vital to continuing this rate of development. Moreover, we're hoping that Diamond opens the possibility for our users to find collaborators in the electron microscopy community and that new collaborations are established, building an even wider community," added Dr Sánchez-Hanke.

Atomic Structure Imaging

Explaining more about his own research work, Dr Chris Allen, Acting Principal EM Staff Scientist at ePSIC commented: "Our strategic objective is to add value to the electron microscopy landscape in the UK and our design has ensured one of the most stable electron microscopes in Europe. Using state of the art aberration-corrected transmission electron microscopes (TEM), we can now routinely achieve sub-Ångstrom resolution in a variety of samples. At ePSIC we are using this technology to address a wide range of challenges by directly imaging the atomic structure of materials.

Our work includes studying catalyst particles to establish links between catalytic action and atomic structure. This not only helps us understand the fundamental mechanisms behind catalytic processes but also provides information for the design of more efficient future catalysts.

There is much current fundamental and commercial research interest in battery technologies. By performing atomic resolution imaging of battery materials at various points in their charge-discharge cycle we can attempt to answer questions pertaining to both precisely what makes a high capacity battery and what causes the degradation of this capacity over time.



Acting Principal EM Staff Scientist, Christopher Allen (right), with Akira Yamagishi from JEOL UK (left), working with one of the microscopes at ePSIC.

In order to form an image within the TEM, samples have to be electron transparent, limiting their thickness to a maximum of a few hundreds of nanometres at most, with ideal thickness of a few tens of nanometres. Consequently, quality sample preparation is crucial and can be technologically challenging.

The formation of images using electrons is a complex process. As such to get the highest quality data from the microscopes requires an excellent understanding of the instrument and an expert operator – our JEOL ARM300 microscope has 19 round lenses, 4 dodecapole elements and 24 deflection coils, each of which has to be adjusted correctly depending on the precise nature of the data required.

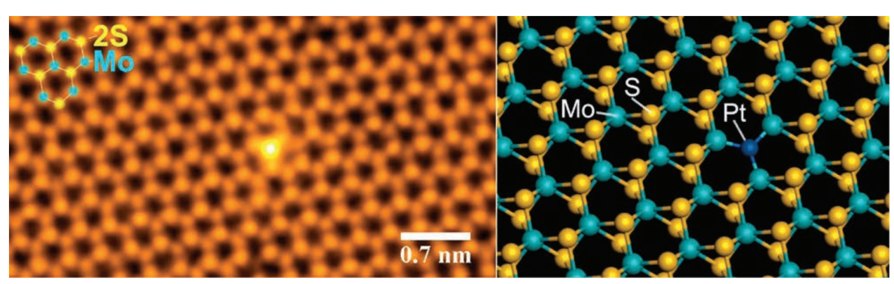
In order to interpret the data from our microscopes in a quantitative manner requires a detailed understanding of the quantum mechanical process that occurs both during the electron – sample interaction and as the electrons propagate through the various lenses of the microscope. We have spent a great deal of time carefully calibrating our instrument to enable us to correctly model these processes and therefore extract the most information from our data.

First users on the I14 beamline (housed in the same building as ePSIC), Manfred Schuster and Peter Ellis (Johnson Matthey), with Principal Beamline Scientist, Paul Quinn.

A large part of my personal research involves studying the structure of low dimensional materials. A tiny concentration of defects or dopants in a material can have a huge consequence on their physical properties. For example, semiconductor technology, which enables all of modern computation and telecommunication, relies on the change in electronic properties of crystalline silicon due to the substitution of as few as one in every billion silicon atoms for a metal atom. If we are to exploit the potential of the new generation of low dimensional materials, such as graphene, we must similarly understand the structure and stability of defects and dopants in these materials.

Within the TEM we can image defects and dopants in low dimensional materials with atomic resolution making it the most powerful tool for their study. Using the state of the art microscopes at ePSIC we are able to record atomic resolution movies of defects and dopants in low dimensional materials with frame rates up to 1000 fps. From this we are developing a deep understanding of defect stability and energetics which will help us to further exploit the fascinating and unique properties of low dimensional materials.

A fast evolving field within electron microscopy is the development of sample holders which allow us to perform dynamic experiments inside the microscope. For example we perform experiments in which we image a material as it is heated or cooled, as an electric current is passed through it, or in a changing liquid or gaseous environment. These 'in-situ' techniques present further challenges in sample preparation and experimental design but with them we are beginning to explore not only static atomic structure but to also image the dynamics of chemical processes on the atomic scale.



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ePSIC will provide a world class capability for materials imaging and the collaboration with Johnson Matthey will bring technologically important problems into focus and answer fundamental research questions. The combination of electron microscopes for both the life and physical sciences, together with the I14 Hard X-ray Nanoprobe beamline, will provide a unique capability, and the location at Diamond will provide the best possible environment to promote scientific interaction."

To find out more about Diamond, or ePSIC, visit their website: **www.diamond.ac.uk**

For further details of the University of Oxford's Department of Materials, visit **www.materials.ox.ac.uk**

For further information about this article contact: **cecilia.sanchez-hanke@diamond.ac.uk**

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