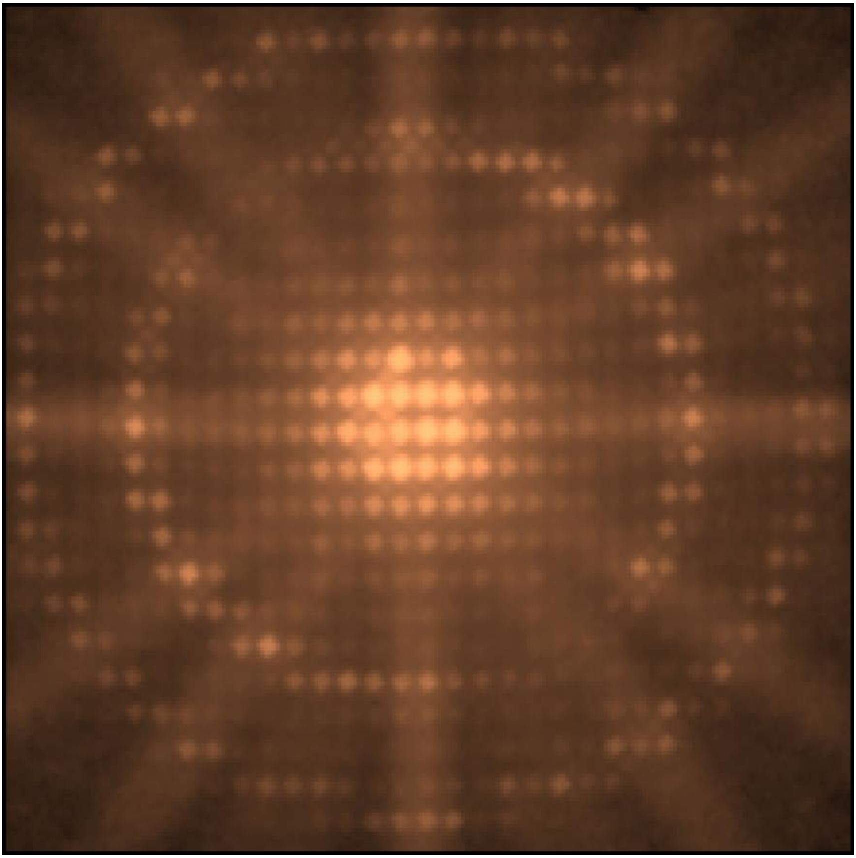
IOP Institute of Physics
Electron Microscopy and Analysis Group

NEWSLETTER January / February 2022



Small convergence angle HOLZ ring pattern from ordered La_2CoMnO_6

Image kindly provided by Dr Ian MacLaren of the University of Glasgow.
For further information on research relating to the image, see
doi: 10.1063/5.0026992 & doi: 10.1016/j.ultramic.2021.113296

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EMAG COMMITTEE

Chair
Jun Yuan
Department of Physics, University of York, Heslington, York, YO10 5DD
Email: jun.yuan@york.ac.uk

Secretary & Honorary Treasurer
Donald MacLaren
School of Physics & Astronomy, University of Glasgow, Glasgow G12 8QQ
Email: donald.maclaren@glasgow.ac.uk

Ordinary Members
Miryam Arredondo (Queen’s University Belfast, m.arredondo@qub.ac.uk)
Laura Clark (University of Leeds, L.Clark@leeds.ac.uk)
Michele Conroy (Imperial College London, m.conroy@imperial.ac.uk)
Alex Eggeman (University of Manchester, alexander.eggeman@manchester.ac.uk)
Sarah Harper (JEOL UK, Sarah.Harper@jeoluk.com)
Emanuela Liberti (Rosalind Franklin Institute, emanuela.liberti@rfi.ac.uk)
Catriona McGilvery (Imperial College London, catriona.mcgilvery@imperial.ac.uk)
Joanne Sharp (University of Huddersfield, J.H.Sharp@hud.ac.uk)
Tom Slater (Cardiff University, SlaterT2@cardiff.ac.uk)

Co-opted Members
Andy Brown (University of Leeds, A.P.Brown@leeds.ac.uk)

LETTER FROM THE CHAIR

Dear Friends and Colleagues,

Welcome to the EMAG Newsletter at the beginning of 2022. I wish you a glorious beginning to the New Year.

In this newsletter, you will have a review of activities undertaken by the EMAG group last year. Central to this is our biannual 'do' with the Royal Microscopical Society (RMS) in July. Despite a COVID-induced shift from our usual place, the beloved Manchester Central, to somewhere virtual in the metaverse, the MMC/EMAG2021 conference attracted more people than ever, and ever more international, as testified by the international nature of the prize winners awarded at the conference, many of whom chose to contribute to describe their findings to this newsletter. For an overall report of the meeting, our departing committee chair, Andy Brown provided a fitting summary. From the feedback received, you have told us that you all enjoyed the scientific program of the conference. This is largely down to your tremendous support, with contributions of abstracts, talks, and poster presentations or just being an enthusiastic audience with your questions and comments. We are also grateful for the seamless behind-the-scenes support provided by the Royal Microscopical Society staff, with the additional innovation of recorded sessions for people to re-watch if they missed the 'live' session, and online archiving of abstracts (https://www.mmc-series.org.uk/mmc2021/abstract-database.html). As ever, we are grateful for the generous support by our commercial partners and the technical presentations and discussions they generated. Also thanks are due to Laura Clark and Jo Sharp for organizing the pre-congress EMAG workshop.

In this newsletter, you will find an overview of a number of electron microscopy and related access schemes available in the U.K. These are opportunities not only to access the state-of-art electron microscopy facilities, but also avenues for collaboration and transfer of expertise and technical knowhow. I personally would like to thank our group Secretary Donald MacLaren for putting together this excellent Newsletter.

Soon after the successful conclusion of EMAG2021, our EMAG group held a successful election for the renewal of the committee membership. We are grateful for all the members who put themselves forward to serve the EMAG community. We welcomed five successful candidates, Michele A Conroy, Alex Eggerman, Emanuela Liberti, Catriona McGilvery and Thomas Slater, as new members of the committee in October. At the same time, we thank the outgoing committee members, Ana Sanchez, Cornelia Rodenburg and Michael Dixon, for their long service to the community. A special thanks is due to Andy Brown who extended his tenure as the group chair to ensure

EMAG2021 was such a success despite the pandemic and now continues to share his vast experience as a co-opted committee member.

The new EMAG committee has begun to plan our activity for 2022. We are actively preparing for a possible in-person focused EMAG meeting with the theme of 'Multi-dimensional Electron Microscopy', so watch out for the announcement of this meeting soon. As part of this meeting, we will also hold a celebration for the more than 75 years history of the EMAG group. Yes, EMAG has such a long history! Back in September, 1946, the inaugural meeting of the EMAG's predecessor, electron microscopy group was held in Oxford, with Darwin's grandson, Charles Galton Darwin of National Physical Laboratory as its first chair and Vernon Cosslett of Cambridge University as its first Secretary. The group was formally accepted by the Board of the Institute of Physics in October 1946. This is the start of the remarkable history of the group, later incorporating microanalysis to become the EMAG group we know today. For those of you interested in the early history, I found Cosslett's 1971's recollection (Phys. Bulletin, vol.22(6), 339-341https://iopscience.iop.org/article/10.1088/0031-9112/22/6/015) the easiest to start. EMAG has prospered throughout its history as one of the larger special interest groups in the Institute of Physics, recording many developments of electron microscopy and electron microscopists in the U.K. and beyond. I welcome all of you to celebrate this rich history by sharing with each other our own experiences of association, especially during the forthcoming activity associated with the EMAG focus meeting.

Best wishes and keep safe,

Jun YUAN
Chairman of the EMAG group, Institute of Physics, January 2022.

FORTHCOMING EMAG EVENTS

EMAG2022: Multidimensional Microscopy

Incorporating a celebration of 75 years of EMAG.

Save the date:	Abstract submission deadline - April 13th 2022
	75th birthday celebrations – 5th July 2022
	EMAG 2022 conference – 6th & 7th July 2022
Submit through:	To be confirmed by email to all members in February 2022

Organised by the Institute of Physics Electron Microscopy and Analysis Group (EMAG), the 2022 EMAG Conference will focus on ***Multidimensional Microscopy***, and span microscopy across the length-scales, correlative, time-resolved and 4D STEM techniques. It is planned to be an in-person event held at Imperial College London. Look out for a first call for abstracts and further details soon!

EMAG Annual General Meeting 2022

It is expected that the EMAG AGM will be held in person during the EMAG2022 conference. If you cannot attend the AGM but have any issues you would like raised at the meeting, please contact the honorary secretary (donald.maclaren@glasgow.ac.uk).

SuperSTEM Summer School

The highly-regarded SuperSTEM Summer School has been postponed to summer 2023. Due to public health and travel restrictions worldwide, efficient planning for the summer school was unfortunately not possible for 2022. Please check the facility's website regularly for further updates, or sign up to receive regular updates: https://www.superstem.org/signup

Remote Hyperspy Workshop

Date TBC (April or May 2022)**.**

This workshop is aimed to be an introduction to the Hyperspy python package for the analysis of multidimensional data (primarily in electron microscopy). Topics will include an introduction to Hyperspy and analysis of spectroscopic, scanning diffraction and atomic resolution data. Due to COVID-19, we are aiming to run this year's workshop fully remotely. Participants will need to have their own computer and will be assisted over Zoom. Further details will be released shortly, but please contact mohsen.danaie@diamond.ac.uk if interested in attending.

NEWS

- It is excellent to note the awards of Honorary Fellowships of the Royal Microscopical Society to Prof. Alan Craven (University of Glasgow, nominated by the EMAG committee) and Prof. Ed Boyes (University of York). These awards are very much deserved and we extend our congratulations!
  For further information, including brief biographies, see the RMS announcement.
- Within the last few days, Professor Sarah Haigh (University of Manchester and recent EMAG Chair) has been honoured in this year's Blavatnik Awards for Young Scientists in the United Kingdom.
- Early Career Researchers may note that the Journal of Microscopy plans a special issue where the first and/or last author is an ECR and the paper is based on development of a microscopy technique, or where use of a particular microscopy technique has enabled an interesting application: https://www.rms.org.uk/study-read/news-listing-page/open-call-to-all-next-generation-microscopists.html
- Follow us on Twitter @IoP_EMAG
  Find us online:
  https://www.iop.org/physics-community/special-interest-groups/electron-microscopy-analysis-group

SuperSTEM Facility Updates

New instrumentation: SuperSTEM4 Hitachi SU9000

SuperSTEM, in partnership with Hitachi High-Tech, are delighted to announce the installation of a Hitachi SU9000 low voltage Ultra-High-Resolution FE-SEM/ STEM instrument. ***SuperSTEM4***, a cold field emission in-lens STEM/SEM operating at beam energies from 10eV to 30keV, boasts an impressive <3Å resolution. The instrument is equipped with an arsenal of imaging detectors for correlative surface and transmission imaging, including energy filtered in-lens SE detection, energy and angular filtered in-lens BSE detection and angular adjustable and segmented ADF/HAADF detection, as well as analytical capabilities with an ***Oxford Instruments Ultim100TLE*** windowless high solid-angle EDX detector for light element analysis.

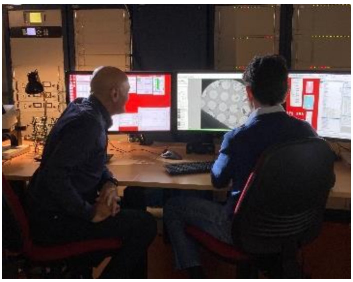
SuperSTEM 4: Hitachi SU9000

An array of available side entry holders is fully compatible with the facility's **Hitachi Ethos NX5000 triple beam FIB** instrument for seamless specimen exchange and observation.

Instrument upgrades

The monochromated SuperSTEM3 instrument has been retrofitted with a **Nion IRIS** high energy resolution EELS spectrometer, equipped with a Dectris ELA electron-counting detector. As of autumn 2021, the energy resolution of the instrument has improved to below 5 meV. SuperSTEM2 has been upgraded with an electrical pole piece for in-situ heating/ biasing and a new ***Bruker XFlash 6T-100*** large collection angle EDS detector for efficient chemical analysis. The delivery and installation of a ***Quantum Detectors Merlin*** direct electron detector upgrade for EELS is scheduled for February 2022.

SuperSTEM 3: Nion IRIS EELS spectrometer & Dectris ELA



Other News: BBC's "Big and Small"

In November, we had the pleasure to host Prof. Jim Al Khalili filming for BBC4's new series "**Big And Small"**. We had the opportunity to talk about the wonderful progress in electron microscopy instrumentation and look at single atoms dancing in graphene. The series will air on BBC4 in 2022.

Check www.superstem.org for news and further updates.

MEETING REPORTS

EMAG2021 (held as part of mmc2021), online 4-6 July 2021

By Andy Brown & Jun Yuan (outgoing and incoming EMAG chairs, respectively).

EMAG holds a biennial UK meeting covering all aspects of physical science microscopy applications, including instrumental or technique development. As has been the case in recent years, the 2021 EMAG meeting was incorporated into the Royal Microscopical Society's (RMS) *mmc*2021 meeting, which took place virtually for the first time. *mmc*2021 was an international scientific virtual conference of six parallel streams, covering an enormous range of microscopy, cytometry and imaging topics with more than 1,300 participants logged on from across the world.

Notably, *mmc*2021 required the creation of a bespoke conference website, which broke new ground for the RMS and we were extremely grateful for all their efforts in developing this. The website featured an integrated abstract admissions system, the embedding of both Vimeo and Zoom for the live conference streams and included an online commercial exhibition with a range of interactive options for delegates and visitors. The EMAG scientific programme consisted of 10 separate conference sessions with invited speakers, posters and a plenary session covering 'Adventures in 4D-STEM' by Prof J. Etheridge of the University of Monash (Australia). The programme was arranged by the EMAG committee to reflect the breadth and quality of the submissions of ~ 130 delegates and to ensure it is representative of the current community's activities. A copy of the programme and abstracts submitted is available here - https://www.mmc-series.org.uk/general-information/previous-congresses.html.

As ever, the range of science presented was truly excellent and an added advantage of the online format was significant growth in contributions and participation from international delegates. While the natural buzz and footfall of an in-person event can never truly be replicated, this was the best possible alternative; it was an exciting meeting with good attendance and engagement at the online scientific sessions. A reflection on EMAG2021 by Natalia Koniuch, a PhD student at the University of Leeds is given below.

"Over the last year or so we've managed to get used to remote working due to the COVID-19 pandemic, therefore the decision to deliver mmc2021 incorporating EMAG virtually came as no surprise. As a third year PhD student in the electron microscopy field, EMAG is an invaluable conference for my progression, offering the opportunity to interact with experts in the field and learn more about current research and development. EMAG offered an inspiring plenary about 4D-STEM (by Prof Etheridge) and a series of impressive invited talks and interesting contributed and flash talks, divided into several themed streams. I had the fantastic opportunity to present my research entitled '**Using transmission electron microscopy to monitor hydration of theophyllin**e' in a supportive environment and get constructive feedback from experts in my field. I found EMAG2021 to be a very informative and engaging event, especially for newer and younger researchers, and even with the online format I felt it was full of interesting discussions that may lead to collaboration in the future and definitely helped my identify emerging areas of research interest such as the wider application of STEM techniques."

Going forwards, planning is underway for *mmc*2023 incorporating EMAG2023, with the intention of returning to an in-person meeting in Manchester, July 2023 and hopefully retaining some positive aspects of the virtual format. We very much look forward to seeing some of you there…

EMAG2021 Student Prize Winners

Oral Talks

1. Waynah Lou Dacayan, DTU Energy, Denmark
   In situ EIS-TEM characterization of electrospun nanofibers for solid oxide electrolysis cells
2. Sharmin Sharna, Institut de Physique et de Chimie des Matériaux de Strasbourg, France
   Understanding Cu-Alumina interactions in redox conditions for Chemical Looping Combustion (CLC) application – A multi-scale correlative electron and X-ray microscopy study

Posters

1. Ruomu Zhang, University of Oxford
   High resolution imaging and spectroscopy of interfaces in solid-state Li-ion batteries
2. Gabriel Ing, University College London
   Liquid-phase Electron Microscopy in Structural and Molecular Biology

Flash Talks

1. Tamsin O'Reilly, Queen's University, Belfast, UK
   Exploring metastable domain configurations in BaTiO3: an in-situ study
2. Till Domröse, University of Gottingen, Germany
   Ultrafast nanoimaging of the order parameter in a structural phase transition

EMAG2021 Published doi references

EMAG2021 did not have a published proceedings; however, the Royal Microscopical Society provided permanent digital object identifier (doi) links to all presenters upon request. Here, we collate those doi links relating to EMAG presentations.

4D-STEM

Design of electron ptychography experiments through simulations, M Danaie, D Batey, T Slater & C Allen, doi:10.22443/rms.mmc2021.338

In-situ biasing and temperature influence on the electric fields across GaAs based p-n junction via 4D STEM, A Pokle, D Heimes, A Beyer & K Volz, doi:10.22443/rms.mmc2021.11

Optimal experiment design for characterising structures containing multiple types of elements using 4D scanning transmission electron microscopy, DG Sentürk, A De Backer & S Van Aert, doi:10.22443/rms.mmc2021.151

Ptychography on dynamically scattering samples, L Clark, G Martinez, C O'Leary, T Petersen, S Findlay & P Nellist, doi:10.22443/rms.mmc2021.240

Quantification of light elements in ABF and 4D STEM electron ptychography, E Liberti, CM O'Leary, KP Treder, JS Kim, PD Nellist & AI Kirkland, doi:10.22443/rms.mmc2021.119

Energy and Energy Storage Materials

Direct Imaging of Oxygen Sub-lattice Deformation in Li-rich Cathode Material Using Electron Ptychography, W Song, M Osorio, J-J Marie, E Liberti, X Luo, C O'Leary, R House, P Bruce & P Nellist, doi:10.22443/rms.mmc2021.111

In situ EIS-TEM characterization of electrospun nanofibers for solid oxide electrolysis cells, WL Dacayan, C Chatzichristodoulou, W Zhang, K Mølhave, I Aziz & SB Simonsen doi:10.22443/rms.mmc2021.125

Nanoscale origins of degradation of Ni-rich NMC Li-ion battery cathodes, J Morzy, W Dose, A Mahadevegowda, C Grey, M de Volder & C Ducati, doi:10.22443/rms.mmc2021.75

Understanding Cu-Alumina interactions in redox conditions for Chemical Looping Combustion (CLC) application – A multi-scale correlative electron and X-ray microscopy study, S Sharna, V Rouchon, A-L Taleb, C Legens, S Stanescu, A Lambert, A-S Gay, D Chiche & O Ersen, doi:10.22443/rms.mmc2021.92

Understanding the degradation of Be tiles in the JET tokomak reactor using EELS and DFT, X Liu, C Makepeace, R Nicholls, S Lozano-Perez & J Yates, doi:10.22443/rms.mmc2021.107

2D Materials
Electron-beam manipulation of lattice impurities, T Susi doi:10.22443/rms.mmc2021.6

Functional Materials
Correlative Tomography for micro- and nano- scale porosity reduction analysis in Additive Manufactured healable aluminium alloy, J Gheysen, B Winiarski, A Chirazi, A Brinek, Y Zhu, G Pyka & A Simar, doi:10.22443/rms.mmc2021.329

Inclined twins related non-radiative defects in vertical III-V nanowires grown in unconventional directions , HA Fonseka, J Gott, N Gächter, X Yuan, R Beanland, HH Tan, C Jagadish, M De Luca & A Sanchez, doi:10.22443/rms.mmc2021.316

Quantitative STEM for Bimetallic Catalyst Nanoparticles, X Luo, A Varambhia, D Ozkaya, S Lozano-Perez & P Nellist, doi:10.22443/rms.mmc2021.88

Utilising correlated EDS and 4DSTEM to investigate the structural ordering of Full-Heusler Co_2FeSi alloy, E Duran, I Azaceta, V Lazarov & A Eggeman, doi:10.22443/rms.mmc2021.309

Soft and Hybrid Materials
Expanding Performance and Usability of High-speed / Low-dose STEM Scanning, T Mullarkey, JJP Peters, C Downing & L Jones, doi:10.22443/rms.mmc2021.19

Study of beam damage effect on CPO-27-Ni Metal Organic Framework by Cs-corrected STEM imaging and EELS, T Tran & D Ozkaya, doi:10.22443/rms.mmc2021.95

To Monochromate or not to Monochromate: Balancing Electron Dose and Energy Spread Requirements, F Quigley, P McBean, P O'Donovan & Lewys Jones, doi:10.22443/rms.mmc2021.37

Using transmission electron microscopy to monitor hydration of theophylline, N Koniuch, M S'ari, N Hondow, R Drummond-Brydson & A Brown, doi:10.22443/rms.mmc2021.255

Automated Control, Advanced Data Processing
Quantification of dynamic structural changes of nanomaterials via atom-counting from sequential ADF STEM images, A De wael, A De Backer, L Jones, A Varambhia, P Nellist & S Van Aert, doi:10.22443/rms.mmc2021.135

Understanding Trainable Segmentation for Inorganic Nanoparticle Images, C Bell, K Treder, C Huang, M Schuster, J Kim, A Kirkland & T Slater, doi:10.22443/rms.mmc2021.134

Using Bayesian inference to improve three-dimensional atomic reconstructions from a single projection using Z-contrast imaging, A De Backer, S Van Aert, PD Nellist & L Jones, doi:10.22443/rms.mmc2021.60

Spectroscopy & Advanced SEM

Fast generation of calculated ADF-EDX scattering cross-sections under channelling conditions, Z Zhang, A De Backer, I Lobato, S Van Aert & P Nellist, doi:10.22443/rms.mmc2021.113

Secondary Electron Hyper Spectral surface imaging for beam sensitive biomaterial characterisation, N Farr, A Quade, J Schäfer & C Rodenburg, doi:10.22443/rms.mmc2021.270

Electron Crystallography and Diffraction

A new sample preparation workflow in the FIB-SEM for rapid, in-situ TKD analyses, P Trimby & J Lindsay, doi:10.22443/rms.mmc2021.131

Comparative analysis of continuous rotation electron diffraction (cRED) data using Bloch-wave simulations, A Cleverley, Y Carter, W Roberts & R Beanland, doi:10.22443/rms.mmc2021.340

Democratisation of dynamical 3D ED: structure analysis using dynamical diffraction applied to all types of 3D electron diffraction data, P Klar & L Palatinus, doi:10.22443/rms.mmc2021.331

Electron diffraction studies of commensurately modulated structures in bismuth transition metal oxide, S Choudhury, V Mohan, H Ghosh, A Pal, M Singh, R Mandal & J Basu, doi:10.22443/rms.mmc2021.79

In-situ microscopy

Assessing structure and dynamics of biological systems via Liquid Phase Electron Microscopy, L Ruiz-Perez, C De Pace, S Acosta-Gutiérrez, G Ing, G Marchelo, S Pilotto, F Werner, FL Gervasio & G Battaglia, doi:10.22443/rms.mmc2021.110

Heat Treatment of Steel: A Novel High Temperature In Situ SEM Imaging Approach, R Heard, doi:10.22443/rms.mmc2021.284

Instrumentation Development (inc. Detector technology)

Extreme Light-Matter Interactions in the Ultrafast Transmission Electron Microscope, I Kaminer, doi:10.22443/rms.mmc2021.260

On the Dependence of the Sensitivity of EELS vs XEDS in the AEM with Thickness and Beam Energy, AB Yankovich, L Zeng, E Olsson & NJ Zaluzec, doi:10.22443/rms.mmc2021.257

Towards a 100,000 frames per second direct detector for 4D STEM, B Levin, D Chatterjee, J Wei, A Kvit, B Bammes, R Bilhorn & P Voyles, doi:10.22443/rms.mmc2021.93

EMAG2021 Prize Winners' Reports

In situ EIS-TEM characterization of electrospun nanofibers for solid oxide electrolysis cells

By Waynah Lou Dacayan, Technical University of Denmark

Attending the EMAG 2021 conference was a great experience as, albeit held online, interaction and exchange of ideas among organizers, presenters, and attendees were well facilitated. On top of this, being incorporated in the MMC 2021 conference, there was also an access to even more great talks and presentations in the field of microscopy for different applications. It is therefore indeed a pleasure for me to be a part of it and to have given an oral presentation on what we are working on in the ERC starting grant project "High-temperature Electrochemical Impedance Spectroscopy Transmission electron microscopy on energy materials" (HEIST).

In this project, we are integrating electrochemical measurements, primarily solid-state electrochemical impedance spectroscopy (EIS) with TEM for in situ characterization of functional materials used in energy applications. In such applications, the overall efficiency of the process mainly relies on the electrochemical and structural properties of the component materials. As these two properties are interdependent, it is critical to simultaneously perform their characterizations to establish a direct structure-activity relationship. The in situ EIS-TEM method is facilitated in an Environmental TEM while exposing the materials to elevated temperature, reactive gases, and applied electrical potential using a chip-based TEM holder and a heating and biasing MEMS chip.

In the work presented, the in situ EIS-TEM method was used to characterize $Ce_{0.9}Gd_{0.1}O_{1.95-\delta}$ (CGO) which is a popular electrolyte-electrode barrier layer material in solid oxide electrolysis cells (SOEC) and is also used as a multi-functional component in the cells' composite electrodes. In particular, an electrospun nanofiber of CGO was characterized as electrospun ceramic nanofibers are known to have good functional properties that are critical in SOEC. In addition, since such nanofibers have diameters ca. 100 nm, no further thinning is needed thereby avoiding surface damage in the material.

Figure 1 shows a coupled EIS and TEM dataset obtained from an in situ characterization of CGO. By taking such coupled dataset at different times and conditions, real-time changes in the structural and electrochemical properties of the material can be acquired which can be correlated with each other. Further information from these datasets can also be used to determine other relevant properties including the structure-dependent conductivity of a material.

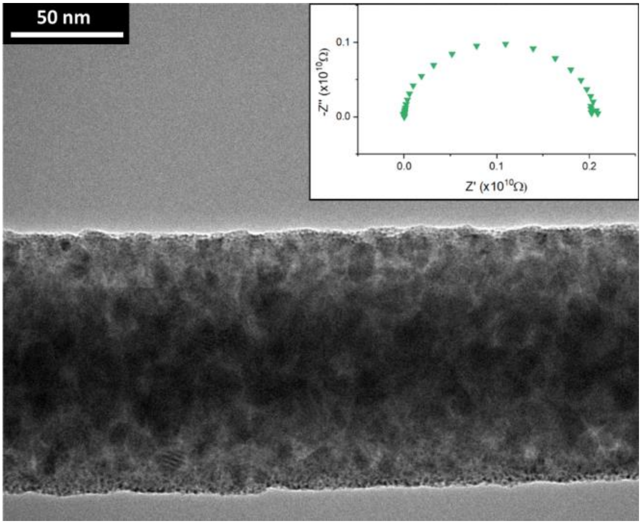


Figure 1: TEM image of a $Ce_{0.9}Gd_{0.1}O_{1.95-\delta}$ nanofiber in 2.5 mbar pO_2 recorded at 500°C and its corresponding EIS data presented as Nyquist plot (insert).

We are also currently expanding the presented EIS-TEM method where other characterizations are done simultaneously including Electron Energy Loss Spectroscopy (EELS) and dark-field imaging to determine possible reduction and grain migration, respectively, together with other electrochemical measurements. With these, we will be able to provide a more complete picture of the critical functional properties of energy materials.

Acknowledgment
This project has received funding from the European Research Council (ERC) under the European Union's Horizon 2020 research and innovation programme (grant agreement No 850850).

Understanding the CuO-Alumina interaction in redox condition using X-ray and Electron microscopy

By Sharmin Sharna, Université de Strasbourg, France

It was a delight and enriching experience to attend the EMAG conference in 2021. Although it was online, the quality and the flow were excellently maintained throughout the conference. I am very thankful to have received the award of runner-up oral for my talk on the correlative utilisation of X-ray and Electron microscopy. My research focusses on the understanding of the interaction between the CuO and Alumina for Chemical Looping Combustion (CLC) application. CLC is an alternative combustion process with low energy penalty carbon dioxide capture solution. The process utilises an oxygen carrier (ceramic supported metal oxide) to provide oxygen to the hydrocarbon for combustion which eliminates direct contact between the air and the fuel, generating a readily separable stream of CO_2. Copper oxide supported on alumina (CuO/Al_2O_3) has been widely considered as a promising oxygen carrier (OC) for industrial use in CLC. Despite having flexible redox behaviour and high oxygen carrying capacity, CuO/Al_2O_3 materials undergo chemical, structural and morphological changes after multiple redox cycles at 900°C, leading to degradation in their overall reactivity. The evolution in the cycled material is attributed to the migration of the Cu-based phases at the grain scale.

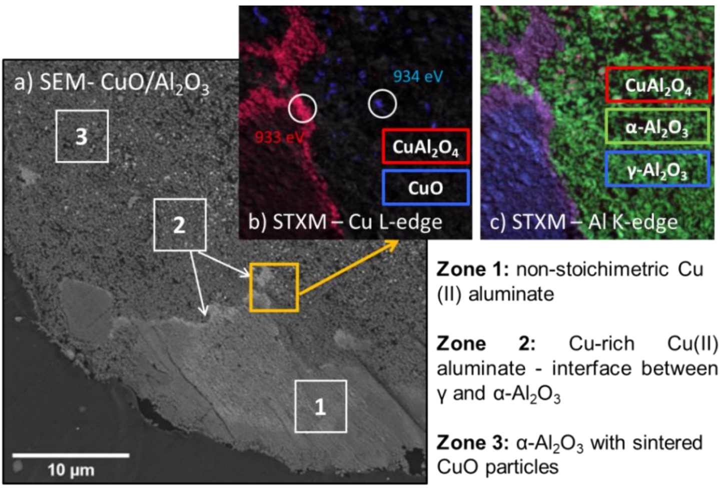


Figure 1: (a) SEM image of ultramicrotomy cut of CuO/Al_2O_3 after 50 cycles; 10 × 10 μm STXM mapping of the area represented by the yellow square at (b) Cu L-edge and (c) Al K-edge

Herein, we bridged the gap in understanding between the observed µm-scale migration of Cu-based phases and nanoscale transformations of the Cu nanoparticles (NPs) by employing a multi-scale characterization approach using Scanning Transmission X-ray (STXM) and Electron (STEM) Microscopies, respectively.

Correlative spectro-microscopic (SEM-EDX and STXM-XAS) techniques helped to understand how the evolution of both Cu and alumina phases and their interaction after different aging times under successive redox cycles are linked to copper migration inside the alumina grain (Figure 1). Furthermore, in-situ STEM, mimicking redox cycling, is used to achieve a nanoscale view of the phase/morphological evolution of the Cu NPs and to identify the sintering mechanism of the copper phases during successive reduction and oxidation steps (Figure 2).

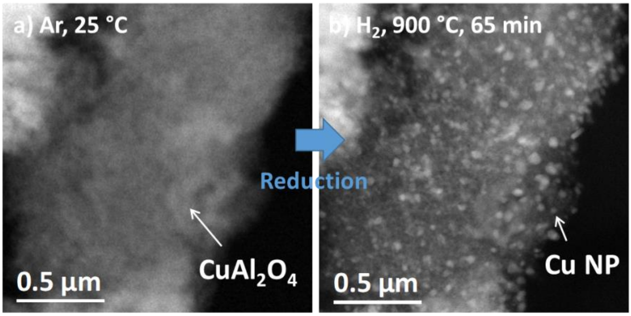


Figure 2: In-situ STEM characterization of fresh CuO/Al_2O_3 OC (a) in Ar atmosphere at 25° and (b) reduction by H_2 at 900° after 1 hr.

High resolution imaging and spectroscopy of interfaces in solid-state Li-ion batteries

By Ruomu Zhang, University of Oxford

EMAG2021 was definitely the most enjoyable conference in 2021 with the efficient arrangement of different streams and many high-quality oral talks and posters. I had a lot of fruitful conversations with researchers across the world that have provided insights for my future works. I would like to take this chance to thank EMAG for providing such a great platform for research ideas exchange, especially during this pandemic situation where communications between people are not as frequent as before. It was a great honour for me to receive the award of 'EMAG Poster 1st Prize' for my poster titled 'High resolution imaging and spectroscopy of interfaces in solid-state Li-ion batteries'.

My research focuses on using (S)TEM to study the spontaneous interfacial reactions in All Solid-state Li-ion batteries (ASSLIBs). ASSLIBs are emerging energy storage devices that are highly expected to revolutionise the electrical vehicle industry. However, the main challenge lies in the side reactions at the electrolyte/cathode interface which cause capacity fading and poor stability. Being an indispensable high-resolution characterisation technique, TEM is however limited in the study of SSLIBs due to the materials' high sensitivity towards both air and beam.

In our work, we pioneered using (S)TEM techniques to study the effect from both air exposure and beam damage on the solid-state electrolyte (SSE) argyrodite. It was found that air exposure leads to inhomogeneous distribution of O and Cl and elemental losses of S. Besides, we observed amorphization and tilting of electrolyte particles as a result of beam damage. By carefully taking diffraction patterns under minimum beam dose, we are able to obtain the critical dose threshold based on the decay of diffraction spots intensity (Fig. 1). We observed a wide distribution of crystallite sizes in argyrodite. The results are free of air exposure and with minimised beam damage benefitting from the long-distance anaerobic transfer procedure and the critical dose. More importantly, small crystallites which significantly limit the ionic conductivity show S loss at specific sites. Furthermore, we are currently using simultaneous ADF/EDS/EELS to characterise the crystal structure, elemental distribution, and Ni oxidation states at cathode/SSE interface. Our work can contribute to the further improvement of ASSLIBs performance by focusing on the composition tailoring and interface design.

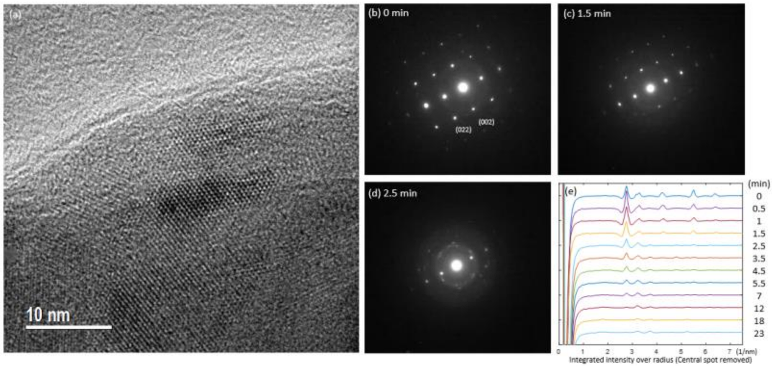


Figure 1: (a) Atomic resolution HRTEM image of argyrodite LPSCl at room temperature at 200kV; ex-situ diffraction pattern of argyrodite LPSCl at 200kV at (b) 0 min; (c) 1.5 min; (d) 2.5 min; f) the integrated intensity over radius at different time (central spot removed)

Liquid-phase Electron Microscopy in Structural and Molecular Biology

By Gabriel Ing, University College London

I thoroughly enjoyed attending the EMAG/MMC conference in July 2021. It was fascinating to see the breadth of microscopy research. I am pleased I was able to hear cutting edge talks on subjects both highly relevant to my research and more distant. My highlight was the poster session where I was able to interact with other researchers and discuss our respective projects, I look forward to being able to do so in person at future conferences! I was delighted to receive the runner up poster prize.

My research focuses on using liquid-phase transmission electron microscopy to study proteins in solution. Proteins are the molecular workforce in cells, as well as the most common type of drug target, therefore it is vital to understand their structure and function. Using liquid-phase electron microscopy on protein solutions, we can start to understand dynamic processes in real time, in ways which are difficult to achieve by other methods. One example of an ongoing project I am working on is imaging amyloid-beta aggregation. Amyloid-beta is a small peptide disordered protein that can aggregate into higher order structures including globular oligomers and long fibres (**Figure 1**). This aggregation pathway is highly associated with Alzheimer's Disease as these structures are commonly found in brains affected by the disease. Thus, current and future therapies aim at finding potential drugs that can target amyloid-B aggregated structures. The details of the aggregation pathway remain elusive, despite previous research attempts, with much of current knowledge arising from computational simulations. My aim is to image different stages of the aggregation pathway of amyloid-beta in real time, in liquid environments by means of transmission electron microscopy.

Liquid-phase transmission electron microscopy is an exciting technique which promises to revolutionise the understanding of many nanoscale processes. There are numerous challenges associated with the technique, starting from experimental difficulties creating a thin, airtight liquid-cell with clean electron transparent window. Often specimens will not show what we are aiming for or we simply cannot understand the phenomena taking place in front of our eyes The images and videos collected are typically obscured by high levels of noise and require significant computational image processing, denoising and analysis before hidden structure features are unveiled and conclusions can be drawn. The results justify the efforts however, as the biological processes being imaged have never be seen in this way before, and the initial results are promising. In this regard our research efforts could offer significant cues for understanding diseases like Alzheimer's disease. There are many challenges associated with LTEM of proteins but also exciting opportunities ahead. I look forward to seeing what I can achieve in my remaining two years of my PhD.

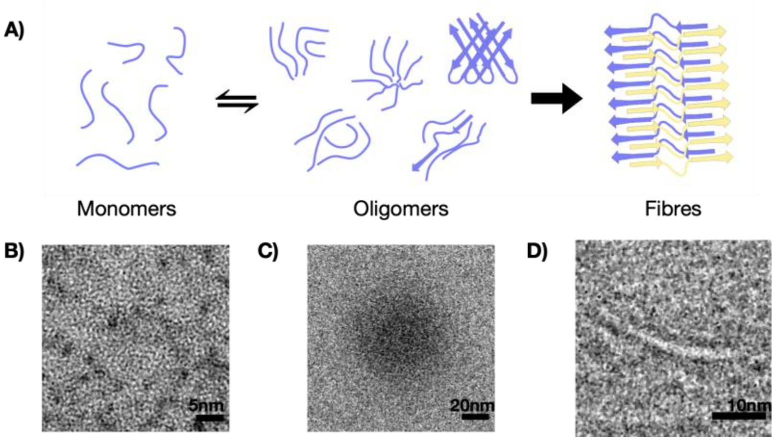


Figure 1: A) *The aggregation pathway of amyloid-beta peptide monomers into globular oligomers followed by amyloid fibrils.* ***B-D)*** *Liquid-phase transmission electron microscopy images of a monomers or small oligomers* ***(B)****, a large oligomer* ***(C)*** *and a fibril* ***(D)****. These structures are fully hydrated and unstained.*

Ultrafast nanoimaging of the order parameter in a structural phase transition

By Till Domröse, Max Planck Institute for Multidisciplinary Sciences, Göttingen

The EMAG conference 2021 has particularly remained in my memory owed to the lively participation of the attendees in this well-organized virtual format. It was a great pleasure to be introduced to the fascinating and multifaceted contributions ranging from material-focused research to method and instrumentation development in talks and direct interaction in the poster sessions. I was delighted to be given the opportunity to present our own work in this productive atmosphere and I am very grateful to have been awarded the second prize for my flash talk entitled *"Ultrafast nanoimaging of the order parameter in a structural phase transition"*.

The experimental results integral to my contribution are facilitated by ultrafast transmission electron microscopy (UTEM), which, as a method, adds an ultrafast temporal component to conventional TEM. In a stroboscopic laser pump/electron probe approach, we record snapshots of non-equilibrium dynamics in heterogeneous structures (see Fig. 1A for the experimental setup). Here, we employ this technique to image a structural phase transition between two charge-density wave (CDW) phases in the prototypical van der Waals material 1*T*-TaS_2 on its intrinsic time and length scales. Both CDW phases and their respective accompanying structural counterpart, an additional periodic modulation of the underlying crystal lattice, are distinctly characterized by the wavelength and the direction of the respective distortion, translating into the occurrence of additional spots in electron diffractograms at the corresponding wave vector.

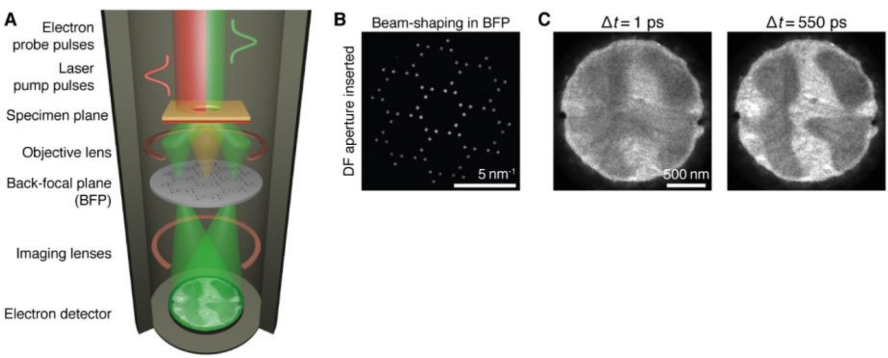


Fig. 1. (A) Experimental setup. (B) Diffractogram of the room-temperature CDW phase with the dark-field (DF) mask inserted into the BFP. (C) DF images displaying CDW (bright) and high-temperature CDW domains (dark contrast) after laser excitation at Δt = 0 ps.

The decisive component of our measurement setup is a uniquely-designed dark-field mask enhancing sensitivity to the local amplitude of the CDW room-temperature state of the material, shaping the electron beam such that only electrons scattered by the CDW periodicity are transmitted further to contribute to the image contrast. With this mask inserted into the back-focal plane of the microscope's objective lens (cf. Fig. 1B), we achieve an unprecedented combined 5 nm spatial and sub-picosecond temporal resolution to local variations of the CDW amplitude. Driving aforementioned phase transition with a spatially heterogeneous ultrashort laser pulse allows us to track the evolution of the emergent pattern of domains of the high-temperature phase of the material, and, furthermore, gives access to dynamics at interphase boundaries (see Fig 1C). With our work, we hope to inspire beam shaping schemes with other materials or more complex devices in the future. Our results have been published in Science:

https://dx.doi.org/10.1126/science.abd2774

Microscopy Infrastructure Access Schemes

A conversation with the Henry Royce Institute Access team

By Jo Sharp

The Henry Royce Institute (Royce) is a partnership of institutions leading in advanced materials research and innovation. The Royce Hub operates at The University of Manchester, with the partner institutions being the universities of Cambridge, Imperial College London, Liverpool, Leeds, Oxford and Sheffield, the National Nuclear Laboratory, UK Atomic Energy Authority, and two associate organisations, the universities of Cranfield and Strathclyde.

Funded by the EPSRC, Royce coordinates over £200 million of facilities and is open to academia, industry and the public, with a joined-up multidisciplinary approach which enables access to leading facilities and capabilities across a number of advanced materials research areas. The Royce equipment catalogue can be viewed online at http://www.royce.ac.uk

Research at Royce tackles some of the most pressing challenges facing society and a number of access schemes have been designed to open up their facilities to students, academic researchers and industry across the UK.

Q: How can researchers and PhD students apply for funded access in 2022?

Royce can provide funds for UK students (doctoral and research masters) and researchers (from PDRAs through to tenured academics), to access their world-class materials science facilities and expertise. Funding is available through national open calls, such as the Royce Student Equipment Access Scheme (for students) and the Royce Research Equipment Access Scheme (for researchers), as well as through facilities access funding held locally within partners. Royce funding has enabled students and researchers to utilise equipment and learn new techniques, in support of single packages of work which they may not otherwise have been able to carry out within their own institutions. Students and researchers are supported by experienced technical staff and Research & Facilities Managers across Royce, who support the opening-up of Royce facilities to the UK academic materials community, and help to build relationships and future collaboration opportunities.

The 2021 Royce Student Equipment Access Scheme handled 149 applications from 41 separate Higher Education Institutions across the UK. Work progressed from more than 80% of these applications, with the majority of projects now complete.

Details of the 2022 national Access Scheme calls have yet to be released, although funding is expected to be available in a similar format and local facilities access funding will continue to be available within Partners. Keep an eye on the Royce website for information when the schemes are announced, or contact access@royce.ac.uk

Q: Is there anything available for small-to-medium enterprises (SMEs)?

Royce is open to businesses of all sizes, to help tackle materials challenges and accelerate innovation. From equipment access for sensitive research to long-term partnerships, Royce can support a range of needs.

The Royce SME Equipment Access Scheme is ongoing and open to UK-based SMEs, spin-outs and start-ups. The scheme offers subsidised facilities access and is designed to overcome cost barriers, de-risk experimental materials-based R&D, and help pave the way for future collaborations with Royce. Companies can either identify a specific piece of equipment they require, or discuss which techniques might work best for them with experienced Business Engagement Managers and the facilities teams.

To apply, companies should complete the application form online at https://www.royce.ac.uk/sme-access/, which requires them to define how Royce facilities could help address materials-based analysis for their business. Technical experts will assist in developing the experimental project and schedule access to appropriate equipment. Project costs may be covered by the Henry Royce Institute up to a maximum of £10,000.

This scheme is an open call available to businesses under the Small Amounts of Financial Assistance Allowance, part of the UK Subsidy Control regime, and businesses must be eligible to receive this in order to apply.

Q: What electron microscopy or sample prep related equipment does Royce have?

Royce has a huge range of EM capability across the Partners including SEM with EDS and EBSD, TEM and STEM, with EDS and EELS, and a wide variety of EM sample preparation techniques. EPMA is available for more quantitative measurements of the composition of bulk samples (also NanoSIMS for trace element or isotopic analysis). There is FIB capability (gallium, plasma and laser) allowing sample preparation and cross sectioning across large regions of samples. Direct electron detectors and precession scanning capabilities enable 4D-STEM data acquisition. There is also a large suite of in-situ EM capability, often available in combination with these techniques including in-situ SEM nanoindentation, environmental liquid and gas cell TEM holders, and capabilities for in situ heating and biasing in both SEM and TEM.

Q: What do you count as a small work package, and how would an investigator approach you to collaborate on a bigger project?

Work packages are usually expected to take between a few days to a couple of weeks under the Royce Student and Researcher Equipment Access Schemes. Costs supported by Royce for these small packages are typically in the order of £5,000.

Further discussions can be held with Royce Facilities Managers around potential longer term collaborative work.

The Royce Hub team at the University of Manchester are gratefully acknowledged for answering these questions to make this article possible. Thank you!

SuperSTEM

The SuperSTEM facility is fully operational and welcomes the submission of new proposals and collaborations through the usual access schemes. Beamtime requests are considered on a rolling basis with no specific call, but it is highly recommended to first get in touch with facility staff (enquiries@superstem.org) to discuss your project and the instruments' capabilities in detail before submitting a proposal. This is so that your proposed experiments can be tailored to optimise the use of the facility and to ensure you get the most from your visit. More details are available online at https://www.superstem.org/facility/access.

Covid-19 measures: protocols for social distancing, including the use PPE are in place for the protection of facility staff and users. Facilities for web-based streaming of the live microscopy sessions, are also available with samples mailed in advance.

ePSIC

ePSIC, a national user facility for electron microscopy, is part of Diamond Light source and is located at the Harwell Science and Innovation Campus in Oxfordshire. The facility offers "free at the point of access" use of two aberration corrected electron microscopes. The centre is a collaboration between Diamond Light Source, Oxford University and Johnson Matthey and comprises two aberration-corrected transmission electron microscopes.

Capabilities at ePSIC include aberration corrected imaging in both TEM and STEM, high solid-angle EDX spectroscopy and imaging at both cryogenic and elevated temperatures. One of the key capabilities of ePSIC is 4D STEM diffraction imaging, enabled by the use of a direct electron detector with a frame rate above 1 kHz (Quantum Detector's MerlinEM). Experiments are supported by a team of staff scientists and instrument time is free at the point of access for academic use.

The main access route for ePSIC is through a "Standard Access" application. Scientists can apply through this route whenever calls for proposals are issued; this takes place twice a year with deadlines usually at the start of April and October. Calls are normally opened six weeks prior to the deadline. Applications are made for the allocation period in 6 months' time, e.g. proposals submitted for the April deadline will be scheduled October-March. However, due to COVID-related interruptions, a new call will go out in January for experiments April-September. Applications for Standard Access are made online via the User Administration System (UAS). Once submitted proposals are reviewed initially by the facility staff for technical feasibility. An independent Peer Review Panel then make recommendations on microscope time allocation based on the scientific merit of the applications.

ePSIC also has a "Rapid Access" route for proposals, which may be submitted at any time. These proposals must meet one of the following criteria:

- The proposal is a short feasibility study, the results of which will inform whether an application is submitted via the standard route for the next allocation period.
- The proposed experiment is necessary to complete a paper that would be ready for submission upon successful completion of the experiment.
- The proposed experiment will contribute towards a PhD thesis that will be completed before the end of the next allocation period and therefore could not wait for the standard access route.

For more information visit https://www.diamond.ac.uk/Instruments/Imaging-andMicroscopy/ePSIC/ or email christopher.allen@diamond.ac.uk

ESTEEM 3

ESTEEM3 is a network of laboratories and SMEs across Europe, which exists to enable access to electron microscopy facilities to academics in both EU and non-EU countries. ESTEEM3 is funded until the end of December 2022. Small amounts of funding for travel and subsistence may be available from some of the participating facilities. There are 15 facilities to which you can apply to use – with a broad range of specialities across sample prep, (S)TEM techniques and data analysis. Details of the available facilities and the application form are available at www.esteem3.eu. Access to UK facilities by UK users can be possible under certain circumstances, for more information on this, contact Angus Kirkland (Oxford).

Queens University Belfast

Facilities include the Climate DENS Holder for dedicated gas in-situ experiments, one of the two systems available in the UK. The holder allows for simultaneous heating and gas flow control. Additionally, we now also have the capabilities for static liquid cell experiments. We would like to invite those interested on in-situ experiments to contact Dr. Miryam Arredondo (m.arredondo@qub.ac.uk).

Kelvin Nanocharacterisation Centre (U. of Glasgow)

KNC Facilities include a plasma focused ion beam system and probe-corrected STEM-EELS instrument. It is now able to offer nano-analytical capabilities (EDS, EELS) alongside differential phase contrast microscopy, scanning precession electron diffraction and 4D STEM with a direct electron detector. Contact phas-knc@glasgow.ac.uk for further information on access.

UK Provision of electron microscopy facilities and services for nuclear research

Nuclear materials can pose specific health and safety concerns. Notably, if the inner workings of a chamber are made active by a sample, there may be a risk to technical officers and research engineers; and many centre staff simply do not have the training or equipment to deal with such samples. The same applies to materials that are highly toxic, for example beryllium, which will contaminate the room via the vacuum system if milled in an ordinary FIB. There are also potential implications for the instrumentation, such as radiation damage to sensitive electron detectors. There are a number of centres around the UK, however, which specialise in electron microscopy of radioactive, nuclear and toxic materials. In this article we highlight aspects of current provision across the UK. Funding schemes are available through the National Nuclear User Facility (NNUF) and Royce Institute.

NNUF funded access is available to projects at any UK university or other institution allowed to apply for UKRI funding. Costs covered: up to 6 months of project, travel, subsistence, sample transport, insurance, and recently added, PhD students' attendance at conferences to present results. Apply through the NNUF access scheme which has quarterly panels: the next one is February 28th 2022; funded access is available until the end of March 2023. To access this scheme, contact the facility you want to use directly via https://www.nnuf.ac.uk/home .

The Royce Institute also funds access to facilities at Royce Institute partner institutions. More details on this are in the article earlier in this newsletter.

The UKAEA Materials Research Facility (MRF) at Culham near Oxford has the following instrumentation within "hot cell" enclosures suitable for radioactive and toxic materials: a FEI Helios FIB, a Tescan Mira 3 SEM with EBSD, EDX, TKD and WDS, and a selection of TEM sample prep equipment including Gatan PIPS2 ion mill, spark erosion and other cutting equipment, and electropolishing. Access to UKAEA equipment is available through NNUF or Royce.

The NNL Central Laboratory at Sellafield in Cumbria has some excellent EM equipment: a FEI Helios FIB with EBSD & EDS and a cryo stage; a TESCAN XEIA3 FIB with EDX, EELS and time-of-flight SIMS (the only active FIB in the UK with SIMS); and a JEOL 2100 (S)TEM with EDS & EELS. An aberration corrected JEOL ARM 2100F STEM with EDS & EELS is due to be installed in Jan/Feb 2022. These facilities can be accessed through NNF or Royce.

The **Alpha-Active Material Manufacturing and Characterisation Facility** lives in the Nuclear Fuel Centre of Excellence at Manchester University, and has a huge variety of equipment concentrated on research into (as the name suggests) alpha-active material. The electron microscopy complement includes: a FEI Helios FIB with EBSD and EDS with an autoloader linked to glovebox, and a FEI Talos F200i TEM with EDS, EELS with an inert atmosphere holder that can be loaded in the glovebox. This can be accessed through NNUF.

MIAMI at Huddersfield University (Microscope and Ion Accelerator for Materials Investigation) is not a facility that deals with radioactive materials, but specializes in observing the effects of ion irradiation in a TEM sample in situ, including as a faster, safer proxy for the effects of e.g. neutron irradiation in fusion reactors. MIAMI2 can irradiate a sample with ions from H to Au at energies from 20-350 keV (inert gases and H can also be run from 1-20 keV from a separate source, in tandem or instead of the main source). Heating and straining holders are available, and the Hitachi LaB6 TEM provides EDX and EELS (but not STEM). Access is possible via the National Ion Beam Centre https://uknibc.co.uk but email miami@hud.ac.uk in the first instance to discuss the work you need.

IoP Research Students Conference Fund

If you are a student member and are looking for funding to attend a meeting or conference, please apply for an RSCF bursary, which may give you up to £300 towards your costs. We have several of these bursaries to give away each year. Check eligibility criteria and download the form at:
www.iop.org/about/grants/travel-bursaries/research_student/page_38808.html

No bursary requests were received in 2021 but funds are available for 2022.

The IoP also run an early career researchers' conference fund, a carers' fund and a benevolent fund that may EMAG members may also be able to access. Further information can be found here: https://www.iop.org/about/support-grants.

EMS Membership

EMAG members are reminded that they are all automatically members of the European Microscopy Society, at no additional personal cost. However, in order to receive information from the EMS, it is essential to send your e-mail address to the EMS secretary - this cannot be sent by the IOP due to the Data Protection Act. This is important, since almost all communications from the EMS are sent by e-mail, including information for voting for the next Executive Board.

Send your e-mail address (and preferably your other details, postal address, phone & fax numbers) to: secr@eurmicsoc.org and indicate whether you agree to include this information in the EMS Yearbook. If you do NOT wish to appear in the Yearbook, your e-mail address will be used solely for the dispatch of information by the EMS secretary (virginie.serin@cemes.fr).

The EMS web page can be viewed at: http://www.eurmicsoc.org/

EMAG members are also reminded of the availability of EMS Bursaries. For more details, see https://www.eurmicsoc.org/en/funding/scholarships/.

Additional Future Meetings of Interest

The Second Joint Meeting of the Microscopy Society of Ireland and the Scottish Microscopy Society
6-8 April 2022, *National University of Ireland Galway, Ireland*

17th European Workshop on Modern Developments and applications in Microbeam Analysis (EMAS 2022), 7-11 May 2022, *Krakow, Poland*

Advanced workshop on cryo-electron tomography
7 -13 May 2022, *Vienna, Austria*

Pico 2022,
8 - 12 May 2022, Kasteel Vaalsbroek, The Netherlands

Quantitative Electron Microscopy 2021/22
8 -20 May 2022, *Port Barcarès, France*

Cryo Electron Microscopy Course
6 - 10 June 2022, *Harpenden, UK*

ESTEEM3 Workshop on Electron diffraction for solving engineering problems
21-23 June 2022, Trondheim, Norway

Faraday Discussion on Challenges in biological cryo electron microscopy,
13-15 July 2022, Sheffield, UK

16th Multinational Congress on Microscopy (MCM)
4-9 September 2022, Brno, Czech Republic

For more microscopy events see:
http://www.eurmicsoc.org/en/meeting-calendar/calendar/
and
https://www.rms.org.uk/rms-event-calendar.html

Contact points

IOP: Institute of Physics, 37 Caledonian Road, London N1 9BU, UK
Tel: +44 (0)20 7470 4800, Fax: +44 (0) 20 7470 4848
For conferences:
Email: conferences@iop.org
http://www.iop.org/events/scientific/conferences/index.html

Group matters: Science Support Officer
Email: groups@iop.org

EMS: European Microscopy Society
Email: secr@eurmicsoc.org
http://www.eurmicsoc.org/index.html

MRS: Materials Research Society, 9800 McKnight Road, Pittsburgh, PA 15237, USA.
Tel: +1 412 779 3003, Fax: +1 412 779 8313
http://www.mrs.org/meetings-events

MSA: Microscopy Society of America, 12100 Sunset Hills Rd., Suite 130, Reston, VA 20190, USA.
Tel: +1 703 234 4115, Fax: +1 703 435 4390
http://www.microscopy.org/

RMS: Royal Microscopical Society, 37/38 St. Clements, Oxford, OX4 1AJ.
Tel: +44 1865 248 768, Fax: +44 1865 791 237
Email: meetings@rms.org.uk
http://www.rms.org.uk/events/

This newsletter is also available on the web

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Institute of Physics, 37 Caledonian Road, London N1 9BU, UK.

Tel: 020 7470 4800

Fax: 020 7470 4848