XMaS Annual Report for 2023/24

Facility Name: XMaS the UK Materials Science Beamline at the ESRF.
Director: Prof. Tom Hase (University of Warwick) and Prof. Chris Lucas (University of Liverpool).
Start/End Dates: 15th November 2018 through 14th November 2023, extended to 31st January 2024.
Funds awarded: £6,909,720: split between Liverpool (£3,427,966) and Warwick (£3,481,754).

Value Proposition

XMaS is the UK Collaborating Research Group (CRG) beamline at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France, with a focus on *materials science*. The beamline remains an integral part of the UK's synchrotron radiation (SR) infrastructure and has been supporting UK materials scientists since 1997. XMaS has recently undergone extensive modifications and delivers a state-of-the-art facility, fully exploiting the capabilities of the new ESRF lattice. It delivers a brilliant X-ray beam with an operational energy range from 2.1 to 40 keV. The upgraded facility provides a combination of techniques from X-ray diffraction to small angle X-ray scattering as well as X-ray absorption and emission spectroscopy.

The science portfolio on XMaS continues to evolve, embracing a broad spectrum of scientific disciplines under the generic theme of materials science and cutting across research activities in physics, chemistry, biosciences, healthcare, engineering, and energy. XMaS continues to provide access to tender X-ray spectroscopy but the new high energy capability (25 – 40 keV) opens up new possibilities for *operando* experiments as well as accessing technologically important X-ray absorption edges. New multi-modal studies at all available energies allow materials to be characterised in terms of composition (XAS, XRF, TXRF) as well as spatial ordering on local (XAS, WAXS, XRD), and larger (SAXS, XRR) length scales. It is only by exploring materials across multiple length and time scales, whilst simultaneously following their functional properties, that fundamental insight into material properties can be obtained. A unique feature of the beamline is that these techniques can be applied to the same sample volume simultaneously (or within a short time) without changing the sample environment.

XMaS has strong links with the Diamond Light Source (DLS) as it has been operational throughout the development of the DLS beamline portfolio and there is frequent transfer of expertise between both facilities. Synchrotron studies tend to be collaborative, and it is essential that UK scientists can maintain, develop, and nurture links with international colleagues to increase the range, quality, and impact of their research. XMaS plays a pivotal role in this by providing a key access point to the ESRF, enabling UK users to develop international collaborations with both academic institutions and industry. These partnerships ensure the future competitiveness, resilience, and creativity of the UK materials sector, which relies on the development, characterisation, and exploitation of novel functional materials. The overall objectives of XMaS are:

- Deliver internationally leading science based on X-ray metrologies across a range of temporal and spatial length scales;
- Implement and develop instrumentation needed to keep the UK at the forefront of materials science;
- Attract world-class researchers to the facility and the UK;
- Train early career scientists and students in advanced X-ray methodologies;
- Operate the facility efficiently, sustainably, and resiliently within a framework of equality, diversity, and inclusion (EDI).

Scientific Excellence

Over the reporting period, XMaS operations have returned to pre-pandemic levels. Here we summarise the scientific excellence as evidenced by papers published within the reporting period and grouped around several of the main challenge themes:

Energy: *Catalysis, Chemical Reaction Dynamics, Energy Storage, Fuel Cells, Materials For Energy Applications, Nuclear and Photonic materials*

Energy materials span a wide range of scientific disciplines with studies on XMaS focusing on understanding catalysis and catalytic reactions, photovoltaics, new battery materials, electrochemical energy conversion and the storage of nuclear waste. There is a growing demand for *operando* and *in situ* studies to correlate functional properties and efficiencies with structure. Studies often rely on small devices or spatially resolved regions in complex sample environments.

Pt-based bi-metallic electrocatalysts are promising candidates to convert surplus glycerol to value added alternative organic compounds as well as a process to generate hydrogen. The nature and concentration of the second element affects the reaction kinetics and influences the product selectivity. Ex situ XAS at the Ni and Pt edges was used to investigate the electrochemical oxidation of glycerol from a series of PtNi nanoparticles (ACS Catal. 12, 14492 (2022)).

Organic semiconductors have broad applications in electronic devices with performance mainly determined by the morphology and crystal structure of the thin films. GI-WAXS was used to study the morphology and microstructure of as deposited and aged n-dialkyl side-chain-substituted thiophene DPP end-capped with phenyl groups (Ph-TDPP-Ph) thin films prepared under different deposition methods and onto different substrates (Langmuir, 39, 12099-12109 (2023)). Grazing incidence XRD was also used to explore the structure of organic solar cells with BTIC-H as A–D–A non-fullerene acceptor (APL Mater. 11, 061128 (2023)). Similar experimental configurations supported a study on the interface energetics of bulk organic heterojunctions and their impact on the external quantum efficiency of devices (APL Mater. 11, 061105 (2023)). In lead-halide perovskite solar cells, monovalent cation engineering has been one of the key drivers for the steep rise in the device efficiency. A study on the impact of chemical modifications at the A-site cation as well as addition of monovalent cation passivation agents on the optoelectronic, charge transport was supported with structural studies on XMaS (Energy Technol. 11, 2300358 (2023)).

Ni and Co *K*-edge XANES and EXAFS were used to support a broad study of the potential for crystalline MOFs to stabilise the electrocatalytic oxygen evolution reaction under near-neutral conditions Two isostructural two-dimensional (2D) MOFs were investigated and their performance in a neutral environment evaluated (Inorg. Chem. Front. 10, 2961-2977 (2023)).

In the field of materials for the nuclear industry, the handling and treatment of radioactive legacy waste requires a transformation into a glass for long-term storage. The Waste Treatment & Immobilization Plant located at Hanford in the USA will process a large amount of low yield waste in the near future. The glass-forming melts can trap gases that are evolving during the feed-to-glass reactions and this causes foaming that insulates the melt pool, reducing heat transfer to the reacting material. Carbon-based reductant additives are usually added to reduce foaming, but the amount and efficacy of some carbon-based solutions was unclear. Spectroscopic studies on XMaS explored in simulated, inactive Hanford high-iron HLW-NG-Fe_2 feeds the redox characteristics and foaming (J. Non-Cryst. Solids 608, 122240 (2023)).

Physical Sciences: *Polymer Materials, Magnetism and Magnetic Materials, Multiferroics, Ferroelectrics, Electronic Structure, Spintronics, Skyrmions, Superconductivity, Surface Science, Supramolecular Chemistry, Chemical Engineering*

Functional, quantum and soft matter materials underpin a plethora of emerging technologies requiring studies which use combinations of X-ray techniques in suitable sample environments. XMaS is one of the few beamlines in which magnetic scattering and diffraction can be performed, attracting user groups exploring element specific magnetic profiles in heterostructures. Resonant reflectivity on a CoFeTaB/Pt bilayer was performed at the Pt L_3-edge and used to characterise both field and spin-orbit torque driven magnetisation reversal in this system. Soft Co *L*-edge data recorded at Diamond suggested an important interface effect which was resolved on XMaS with studies of the proximity-induced magnetism (PIM) (Phys. Rev. B 106, 094429 (2022)).

As part of the EMPIR work, XRD measurements were performed on the offline X-ray laboratory on $BaTiO_3$ crystals sparsely doped with iron. Giant electrostrain was found, but degradation observed with electrical cycling. XRD studies identified four distinct phases as a function of temperature. Lattice parameters and symmetries were determined, and the diffraction data mapped to the functional properties of the films (APL Mater 11, 041109 (2023)). A different user group explored the integration of ferroelectric thin films into the silicon infrastructure, which is key to exploiting their functional potential, since the properties of the ferroelectric films strongly depend on their texture. A diffraction study explored the crystalline texture in a $La_2O_2CO_3$ thin film using a scalable inkjet printing method (J. Mater. Chem. C 11, 7705-7713 (2023)).

A new area of research on XMaS has been in the spectroscopic study of minerals and specifically the chemical state of the metals embedded within them. Showing the potential for such studies on the enhanced beamline, studies at the Ag *K*-edge explored in synthetic Ag-bearing pyrrhotites ($Fe_{1-x}S$) where the concentration of Ag ranged from 0.08 wt. % to 1.3 wt. % at 750 – 760 °C (ACS Earth and Space Chem. 7, 1648-1660 (2023)).

Healthcare Technologies: *Biomaterials, Biological Materials, Tissue Engineering, Biophysics and Soft Matter Physics*

In this reporting period, GI-WAXS studies were undertaken to understand the degree of crystallinity in semiconducting polymer in nanoporous thin films which were designed to be able to stimulate and promote the intracellular concentration of reactive oxygen species in human umbilical vein endothelial cells (HUVECs) (ACS Appl. Mater Interfaces 15, 30, 35973 (2023)).

The expertise of the XMaS team impacts activities at other central facilities. Nanobeams which are produced at 4th generation synchrotrons have coherence properties that make them ideal for 3D diffraction-based microscopy with unique sensitivity to strain and defects in crystalline materials. A short overview on the use of nanobeams for diffraction experiments, and perspectives for their use in spectroscopy studies were provided to the community (Nucl. Instrum. Meth. B 539, 127-135 (2023)).

Science performed on XMaS continues to be singled out for inclusion in the ESRF's outreach publications. In the upcoming 2023 version of the *ESRF highlights*, the work done on XMaS exploring columnar liquid quasicrystals with a honeycomb structure consisting of triangular, square and trapezoidal cells by X. Zeng *et al.* is emphasised (Nat. Chem. 15, 625-632 (2023)). The study by H. Luo *et al.* on the role of Ni in PtNi bimetallic electrocatalysts for the reduction of glycerol via glycerol electro-oxidation and the concomitant production of hydrogen is also included (ACS Catal. 12, 14492 (2022)).

New methodologies that have been developed

OpMetBat: XMaS, through the University of Liverpool, is a partner in the Joint Research Project within the European Partnership on Metrology "Operando metrology for energy storage materials" – OpMetBat which was launched in 2022. The project focuses on the development of new battery materials and specifically on improving the performance, lifetime, safety, and cost of energy storage technologies. Innovation is currently hampered by the inability for industry to characterise the chemistry of new batteries under *operando* conditions. The project is building a metrological framework at XMaS that will support traceable *operando* characterisation of state-of-the-art battery materials under dynamic charge/discharge conditions and establishing a good practice guide for *operando* spectroscopy and diffraction methods.

Figure 1: OpMetBat cell developed for transmission and grazing incidence XRD measurements at XMaS.

The first part of the programme, which started in 2022, began with studying pristine samples prior to any electrical cycling. All studies were performed under argon atmosphere. Several consortia meetings have occurred through the reporting period. We have undertaken preliminary XRD studies of pristine/virgin battery materials and collaborators successfully applied for beam time at XMaS through the ESRF (University of Rome). The next step is to study the structure of ex-situ cycled batteries using a new cell that can perform powder XRD under inert atmosphere (Figure 1). The cell has been designed by one of the consortium members – HZB, Berlin, and will be commissioned into the XMaS control system in February 2024 to then be available for the wider user community.

KB Mirror

In the last Annual Report, we detailed the decision to add micron-sized beams to the facility through the installation of a custom KB system. Over this reporting period, the system was designed, manufactured and commissioned by IDT. Delivery to the facility happened in August. Installation (Figure 2) and commissioning happened in October and December 2023. Routine user access is expected in 2024.

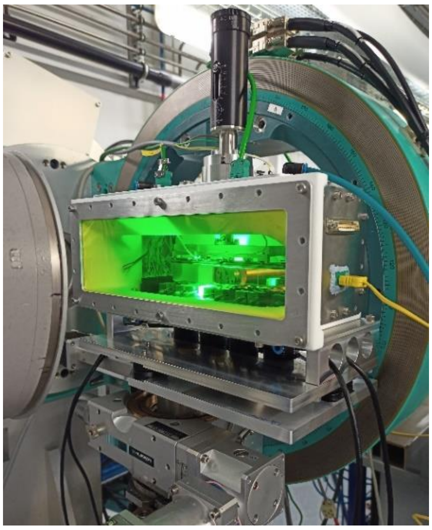
Figure 2: The IDT KB mirror system installed on the beamline.

The system is housed on the unfocused beam path and does not impede normal focused beam operations as this beam passes uninterrupted through the final housing. The design of the system also allows it to move out of the beam on a rotation. The reproducibility of the movement is still to be determined, but preliminary results indicate that the focused beam remains at the same place when the focused KB assembly is switched. Beam sizes < 5.5 (H) x 7 (V) μm^2 have already been measured in the first trials.

New sample environments for WAXS/SAXS experiments

A challenge for soft-matter studies and SAXS/WAXS studies in general is background signals arising from the various apertures and windows, which are inherent in the beamline and many of the sample environments. Over the reporting period, we have designed and successfully tested different sets of apertures and windows to continue to reduce the background and improve the signal to noise ratio for measurements on samples with low scattering power.

One of the sources of performance instability in organic solar cells (OSC) and photoactive devices is the light itself, what is called light soaking. To help in unravelling the structural changes that are correlated with light soaking, a GI-WAXS chamber to characterise high-efficiency OSCs under *operando* conditions has been designed and fabricated (Figure 3). Four samples can be illuminated with four different wavelengths or 'chromatic sections', corresponding to different regions of the solar emission spectrum (ultraviolet, white, green and near infrared), and the efficiency of the devices measured, simultaneously, through the implemented electronics. Additionally, the chamber allows increasing the temperature of the samples up to 100 °C to perform accelerated aging experiments.



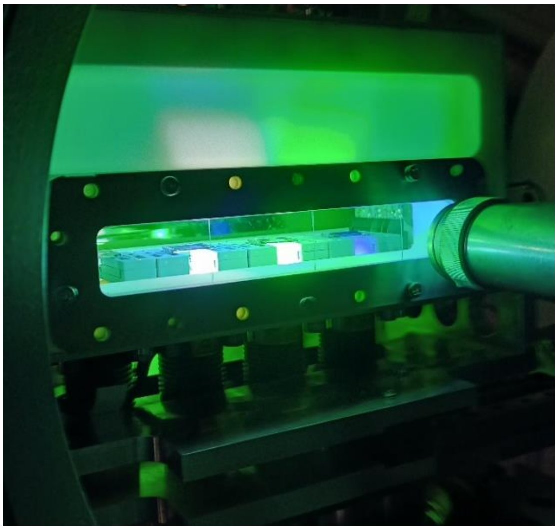


Figure 3: A new GIWAXS chamber for energy materials with an integrated sample support for simultaneous *in situ* and *operando* measurements of organic solar cells and photoactive devices under different illumination conditions.

PyXscat

We have developed an open-source software, PyXscat (Figure 4), with a graphical user interface for quick visualisation and reduction of two-dimensional data collected during X-ray scattering/diffraction experiments. PyXscat is especially focused on grazing-incidence geometries to assist both beginners and experienced synchrotron users. It provides a limited but powerful toolkit, including integration methods, background subtraction, frame-averaging and grazing-incidence corrections. It can be used during data collection, as a live visualisation tool, to assess the quality of the signal during the synchrotron experiment and as a complement, to analyse the data after the experiment. Its main assets are: (a) Intuitive, fluid browsing of data files and metadata; (b) Quick assessment of data quality and tuneable background subtraction; (c) Automatic data reduction and visualisation; (d) Implementation of grazing-incidence functionalities. PyXscat is fully coded in Python, open-source and built upon open-source code dependencies. The program, when possible, reuses many already existing and well-established packages and methods, specifically some data handling packages developed at the ESRF. The code and the manual are publicly available at https://gitlab.esrf.fr/xmas-bm28/data_analysis/pyxscat.

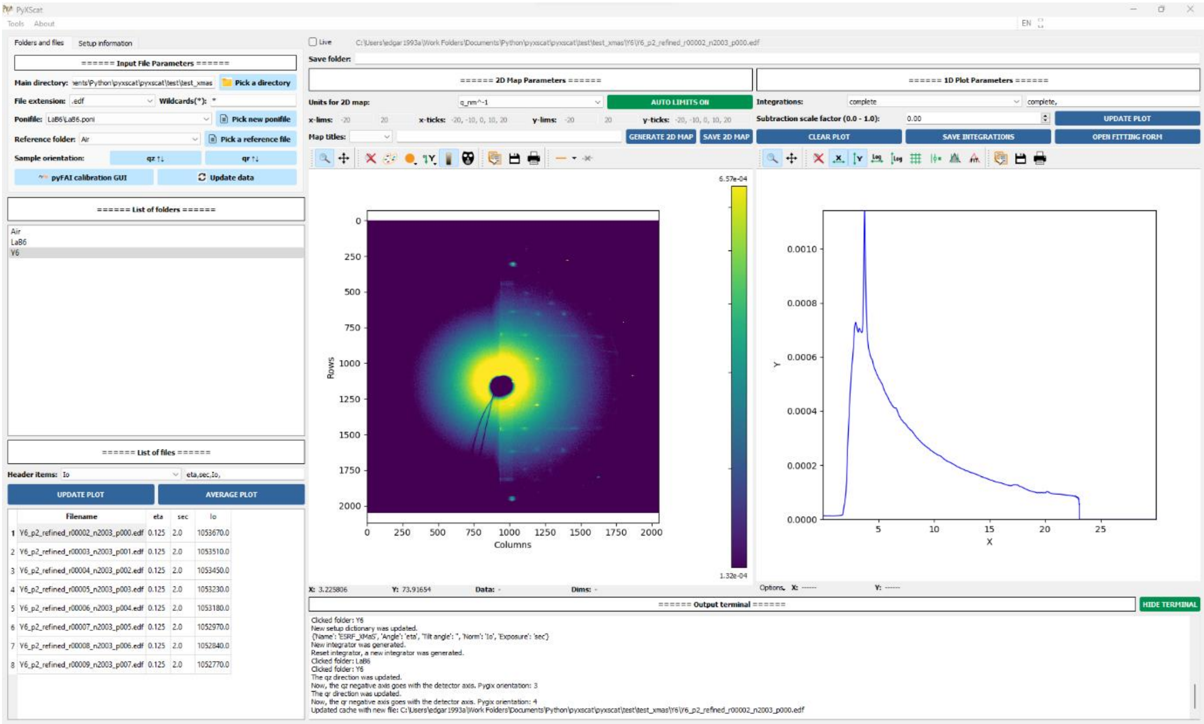


Figure 4: Screenshot of the PyXscat main window. The central image shows a 2D GIWAXS pattern. The azimuthally integrated intensity is plotted on the right panel.

Future Metrologies

The XMaS facility is consolidating the upgrades in infrastructure, hardware and sample environment that have been developed during the past few years. The plan over the next few years is to build capabilities in correlative studies and expand the synergistic use of different X-ray techniques. As identified by the quinquennial ESRF review (which took place in May 2023), the uniqueness of the facility is the ability to rapidly change energy whilst maintaining beam position on the sample. This will be exploited initially through fast energy scanning and spectroscopy but, with improvements in background and data handling, potentially allows rapid switching between scattering and spectroscopy measurements on a sample being held under technologically relevant conditions. This approach will require investment in suitable controls and protocols to be developed, especially as the current control system will be replaced by the BLISS control system in the next 2 years.

Publications

A full list of the publications is maintained on our website. We track publications by direct engagement with our users; an online submission portal is also available through our website and we perform regular searches of Web of Knowledge. Here we provide a full list of the publications associated with the XMaS NRF published in peer-reviewed literature over the past 3 years. The cumulative effects of COVID and the replacement of the ESRF lattice and corresponding dark period from late 2018 through to the gradual resumption of user operations during 2020 naturally impacted the number of published outputs throughout 2022 and 2021. The 2022 ESRF highlight publications from XMaS are marked (*). Publications that have been prepared specifically for a wider audience are marked (†).

2023

Carbone D, Bikondoa O,
"Focused and coherent X-ray beams for advanced microscopies",
Nucl. Instrum. Meth. B 539, 127-135 (2023). DOI: 10.1016/j.nimb.2023.03.036

Costley-Wood LG, Carravetta M, Decarolis D, Goguet A, Kordatos A, Vakili R, Manyar H, McPake E, Skylaris C-K, Thompson P, Gibson EK, Wells PP,
"Towards the Operational Window for Nitridic and Carbidic Palladium Nanoparticles for Directed Catalysis",
ChemCatChem 15, e2023008, 9 pp (2023). DOI: 10.1002/cctc.202300870

Criado-Gonzalez M, Bondi L, Marzuoli C, Gutierrez-Fernandez E, Tullii G, Ronchi C, Gabirondo E, Sardon H, Rapino S, Malferrari M, Cramer T, Antognazza MR, Mecerreyes D,
"Semiconducting Polymer Nanoporous Thin Films as a Tool to Regulate Intracellular ROS Balance in Endothelial Cells",
ACS Appl. Mater Interfaces 15, 30, 35973-35985 (2023) and supplem. DOI: 10.1021/acsami.3c06633

Dremann D, Kumar EJ, Thorley KJ, Gutierrez-Fernandez E, Ververs JD, Daniel Bourland J, Anthony JE, Ram Srimath Kandada A, Jurchescu OD,
"Understanding Radiation-Generated Electronic Traps in Radiation Dosimeters Based on Organic Field-Effect Transistors",
Mater. Horiz. 00, 1-8 (2023) and supplem. DOI: 10.1039/d3mh01507f

Eales JD, Bell AMT, Cutforth DA, Kruger AA, Bingham PA,
"Structural changes in borosilicate glasses as a function of Fe2O3 content: A multi-technique approach",
J. Non-Cryst. Solids 622, 122664 (2023) and supplem. DOI: 10.1016/j.jnoncrysol.2023.122664

Filimonova ON, Wermeille D, Kvashnina KO,
"State of Ag in Pyrrhotite: Insights from X-ray Absorption Spectroscopy",
ACS Earth and Space Chem. 7, 1648-1660 (2023) and supplem. DOI: 10.1021/acsearthspacechem.3c00002

Fynbo C, Huss-Hansen MK, Bikondoa O, Gangadharappa C, Filho DADS, Patil S, Knaapila M, Kjelstrup-Hansen J,
"Structural Study of Diketopyrrolopyrrole Derivative Thin Films: Influence of Deposition Method, Substrate Surface, and Aging",
Langmuir 39, 12099-12109 (2023) and supplem. DOI: 10.1021/acs.langmuir.3c01378

Guo X, Briscoe WH,
"Molecular interactions, elastic properties, and nanostructure of Langmuir bacterial-lipid monolayers: Towards solving the mystery in bacterial membrane asymmetry",
Curr. Opin. Colloid Interface Sci. 67, 101731 (2023). DOI: 10.1016/j.cocis.2023.101731

Habib I, Kaienburg P, Xia D, Gough O, Zhu M, Spruce J, Li W, Riede M,
"Vacuum deposited organic solar cells with BTIC-H as A–D–A non-fullerene acceptor",
APL Mater. 11, 061128 (2023) and supplem. DOI: 10.1063/5.0148208

Inyang O, Swindells C, Rianto D, Bouchenoire L, Morris RJH, Merkulov A, Caruana A, Kinane C, Hase TPA, Atkinson D,
"Non-uniform Gd distribution and magnetization profiles within GdCoFe alloy thin films",
Appl. Phys. Lett. 123, 122403 (2023). DOI: 10.1063/5.0165423
and Erratum: *"Non-uniform Gd distribution and magnetization profiles within GdCoFe alloy thin films"*
Appl. Phys. Lett. 123, 199901 (2023). DOI: 10.1063/5.0180988

Jungbluth A, Kaienburg P, Lauritzen AE, Derrien T, Riede M,
"Probing the energy levels of organic bulk heterojunctions by varying the donor content",
APL Mater. 11, 061105 (2023). DOI: 10.1063/5.0148191

Liang X, Wang S, Feng J, Xu Z, Guo Z, Luo H, Zhang F, Wen C, Feng L, Wan C, Titirici M-M,
"Structural transformation of metal–organic frameworks and identification of electrocatalytically active species during the oxygen evolution reaction under neutral conditions",
Inorg. Chem. Front. 10, 2961-2977 (2023). DOI: 10.1039/D2QI02436E

Lin Q, Senanayak SP, Nia NY, Alsari M, Lilliu S, Abdi-Jaleb M,
"Impact of A-Site Cation Modification on Charge Transport Properties of Lead Halide Perovskite for Photovoltaics Applications",
Energy Technol. 11, 2300358 (2023). DOI: 10.1002/ente.202300358

Patterson EA, Finkel P, Cain MG, Thompson P, Lucas C, Staruch M,
"Rejuvenation of giant electrostrain in doped barium titanate single crystals",
APL Mater 11, 041109 (2023). DOI: 10.1063/5.0142103

Picavet E, Rijckhaert H, Solano E, Bikondoa O, Gutierrez-Fernandez E, Paturi P, Van Bossele L, Vrielink H, Beeckman J, De Buysser K,
"The self out-of-plane oriented La2O2CO3 film: an integration tool for fiber textured ferroelectric thin films",
J. Mater. Chem. C 11, 7705-7713 (2023) and suppl. DOI: 10.1039/D3TC01046E

Rigby JC, Dixon DR, Kloužek J, Pokorný R, Thompson PBJ, Scrimshire A, Kruger AA, Bell AMT, Bingham PA,
"Alternative reductants for foam control during vitrification of high-iron High Level Waste (HLW) feeds"
J. Non-Cryst. Solids 608, 122240 (2023) and supplem. DOI: 10.1016/j.jnoncrysol.2023.122240

Xia T, Wemyss AM, Salehiyan R, Heeley EL, Hu X, Tang F, Sun Y, Hugues DJ, McNally T, Wan C,
"Effective and Fast-Screening Route to Evaluate Dynamic Elastomer-Filler Network Reversibility for Sustainable Rubber Composite Design",
ACS Sustainable Chem. Eng. 11, 17857−17869 (2023) and supplem. DOI: 10.1021/acssuschemeng.3c06752

(*) Zeng X, Glettner B, Baumeister U, Chen B, Ungar G, Liu F, Tschierske C,
"A columnar liquid quasicrystal with a honeycomb structure that consists of triangular, square and trapezoidal cells",
Nat. Chem. 15, 625-632 (2023) and supplem. DOI: 10.1038/s41557-023-01166-5

Engineering and Physical Sciences Research Council

2022

Burn DM, Fan R, Inyang O, Tokaç M, Bouchenoire L, Hindmarch AT, Steadman P,
"Spin orbit torque driven magnetization reversal in CoFeTaB/Pt probed by resonant X-ray reflectivity",
Phys. Rev. B 106, 094429 (2022). DOI: 10.1103/PhysRevB.106.094429

Finkel P, Cain MG, Mion T, Staruch M, Kolacz J, Mantri S, Newkirk C, Kavesky K, Thornton J, Xia J, Currie M, Hase T, Moser A, Thompson P, Lucas C, Fitch A, Cairney JM, Moss SD, Nisbet AGA, Daniels JE, Lofland SE,
"Simultaneous Large Optical and Piezoelectric Effects Induced by Domain Reconfiguration Related to Ferroelectric Phase Transitions", Adv. Mater. 2106827, 1-8 (2022). DOI: 10.1002/adma.202106827

Gruender Y, Lucas CA, Thompson PBJ, Joly Y, Soldo-Olivier Y,
"Charge Reorganization at the Adsorbate Covered Electrode Surface Probed through in Situ Resonant X-ray Diffraction Combined with ab Initio Modeling",
J Phys. Chem. C 126, 9, 4612–4619 (2022) and supplem. DOI: 10.1021/acs.jpcc.1c09857

(†) Hase TPA, Wermeille D,
"*The power of X-rays in materials science",* Futurum, DOI: 10.33424/FUTURUM245

Li Y, Gao H, Zhang R, Gabana K, Chang Q, Gehring GA, Cheng X, Zeng X, Ungar G,
"A case of antiferrochirality in a liquid crystal phase of counter-rotating staircases",
Nat. Comm. 13, 384, 1-11 (2022) and supplem. DOI: 10.1038/s41467-022-28024-1

(*) Luo H, Yukuhiro VY, Fernández PS, Feng J, Thompson P, Rao RR, Cai R, Favero S, Haigh SJ, Durrant JR, Stephens IEL, Titirici M-M
"Role of Ni in PtNi Bimetallic Electrocatalysts for Hydrogen and Value-Added Chemicals Coproduction via Glycerol Electrooxidation"
ACS Catal. 12, 14492 (2022) and supplem. DOI: 10.1021/acscatal.2c03907

Swindells C, Głowinski H, Choi Y, Haskel D, Michałowski PP, Hase T, Stobiecki T, Kuswik P, Atkinson D,
"Magnetic damping in ferromagnetic/heavy-metal systems: The role of interfaces and the relation to proximity-induced magnetism", Phys. Rev. B 105, 094433 (2022). DOI: 10.1103/PhysRevB.105.094433

Swindells C, Atkinson D,
"Interface enhanced precessional damping in spintronic multilayers: A perspective",
J.Appl. Phys. 131, 170902 (2022). DOI: 10.1063/5.0080267

2021

Al Kindi M, Joshi G, Cooper K, Andrews J, Arellanes Lozada P, Leiva-Garcia R, Engelberg D, Bikondoa O, Lindsay R,
"Substrate Protection with Corrosion Scales: Can we Depend on Iron Carbonate?",
ACS Appl. Mater. Interfaces 13, 48, 58193–58200 (2021). DOI: 10.1021/acsami.1c18226

Bikondoa O, Carbone D,
"On Compton scattering as a source of background in coherent diffraction imaging experiments",
J. Synchrotron Rad. 28, 538-549, (2021). DOI: 10.1107/S1600577521000722

Brewer A, Lindemann S, Wang B, Maeng W, Frederick F, Li F, Choi Y, Thompson PJ, Kim JW, Mooney T, Vaithyanathan V, Schlom DG, Rzchowski MS, Chen LQ, Ryan PJ, Eom CB,
"Microscopic piezoelectric behavior of clamped and membrane (001) PMN-30PT thin films",
Appl. Phys. Lett. 119, 20, 2903, (2021). DOI: 10.1063/5.0068581

Brandt RL, Salvati E, Wermeille D, Papadaki C, Le Bourhis E, Korsunsky AM,
"Stress-Assisted Thermal Diffusion Barrier Breakdown in Ion Beam Deposited Cu/W Nano-Multilayers on Si Substrate Observed by in Situ GISAXS and Transmission EDX",
ACS Appl. Mater. Interfaces 13, 5, 6795-6804 (2021). DOI: 10.1021/acsami.0c19173

Dowsett M, Wiesinger R, Adriaens M,
"X-ray diffraction",
Spectroscopy, Diffraction and Tomography in Art and Heritage Science, Chapter 6, 161-207 (2021).
DOI: 10.1016/B978-0-12-818860-6.00011-8

Fox LJ, Slastanov A, Taylor N, Wlodek M, Bikondoa O, Richardson OM, Briscoe WH,
"Interactions between PAMAM dendrimers and DOPC lipid multilayers: Membrane thinning and structural disorder",
Biochimica et Biophysica Acta (BBA) - General Subjects 1865, 129542 (2021).
DOI: 10.1016/j.bbagen.2020.129542

Hussain M, Nagaraj M, Cayre OJ, Robles ESJ, Tantawy H, Bayly AE,
"Aqueous Phase Behavior of a NaLAS−Polycarboxylate Polymer System",
Langmuir 37, 17, 5099–5108 (2021). DOI: 10.1021/acs.langmuir.0c03280

Korsunsky AM, León Romano Brandt LR,
"The Effect of Deposition Parameters on the Mechanical and Transport Properties in Nanostructured Cu/W Multilayer Coatings"
Functional Thin Films Technology, Ed. by Sam Zhang, Jyh-Ming Ting, Wan-Yu Wu, chap. 11, (2021).
DOI: 10.1201/9781003088080

O'Neill CD, Abdul-Jabbar G, Wermeille D, Bourges P, Krüger F, Huxley AD,
"Field-Induced Modulated State in the Ferromagnet PrPtAl",
Phys. Rev. Lett. 126, 197203 (2021) and supplem. DOI: 10.1103/PhysRevLett.126.197203

(*) O'Neill CD, Schmehr JL, Keen HDJ, Pritchard Cairns L, Sokolova DA, Hermann A, Wermeille D, Manuel P, Krüger F, Huxley AD,
"Non-Fermi liquid behavior below the Néel temperature in the frustrated heavy fermion magnet UAu_2", PNAS 118, 49, e2102687118 (2021). DOI: 10.1073/pnas.2102687118

Swindells C, Głowinski H, Choi Y, Haskel D, Michałowski PP, Hase T, Kuswik P, Atkinson D,
"Proximity-induced magnetism and the enhancement of damping in ferromagnetic/heavy metal systems",
Appl. Phys. Lett. 119, 152401 (2021). DOI: 10.1063/5.0064336

Tierney GF, Alijani S, Panchal M, Decarolis D, Briceno de Gutierrez M, Mohammed KMH, Callison J, Gibson EK, Thompson PBJ, Collier P, Dimitratos N, Crina Corbos E, Pelletier F, Villa A, Wells PP,
"Controlling the Production of Acid Catalyzed Products of Furfural Hydrogenation by Pd/TiO_2",
ChemCatChem 13, 5121-5133 (2021). DOI: 10.1002/cctc.202101036

Engineering and Physical Sciences Research Council

Scientific Excellence - Highlighting the role of XMaS

Figure 5: Front cover of the 2022 XMaS Newsletter.

XMaS Newsletter: XMaS activities are widely disseminated in our annual newsletter which is written for a scientifically interested reader. The 2022 version was available from March 2023 and circulated widely (> 600 copies). The newsletter highlights a range of science that is undertaken on the beamline and showcases its capabilities. We provide below a short paragraph describing the research and the role of the facility in each of the scientific articles below:

- **Kondo charge accumulation** (PNAS 118, 49, e2102687118 (2021)): High resolution diffraction tuned close to and away from the U M_4-edge was used to study the magnetic and charge modulations in UAu_2 down to 2 K. The combination of a suitable sample environment, beamline capabilities and good signal to noise allowed a significant contribution to the charge scattering to be observed at temperature below which the magnetic order had saturated, suggesting charge accumulation driven by Kondo screening below the Néel temperature.
- **Near interface magnetisation reversal in spintronic bilayers** (Phys. Rev. B 106, 094429 (2022)): Resonant reflectivity measurements tuned to the Pt L_3-edge and exploiting the phase retarder explored the spatially induced magnetic polarisation of Pt in a CoFeBTa/Pt bilayer as a function of both applied field and electrical current. XMaS was required for the *in-situ* studies and resonant polarisation control. The fitted data are used in conjunction with similar soft X-ray studies probing the Co *L*-edges at Diamond and the combined data set provides new insights into reorientation of the magnetisation through the application of external stimuli.
- **Probing magnetism with X-ray Standing Waves**: This thesis work describes a novel approach to the study of magnetic order at the atomic level using a combination of site-selectivity from the diffraction-based X-ray standing waves (XSW) technique coupled to the sensitivity of the magnetic properties through X-ray magnetic circular dichroism (XMCD). Preliminary data exploiting the ability of XMaS to combine scattering, spectroscopy and polarisation control were necessary.
- **TXRF on XMaS: Environmental Quantification:** The flexibility of the diffractometer and the suite of XMaS detectors was used within a COST Action to demonstrate the ability of XMaS to perform quantitative analysis of noxious materials. Preliminary data on $PM_{2.5}$ particulate matter was collected for inclusion into a new ISO standard and the preparation of suitable reference standards.
- **Spontaneous self-assembly of straight- and bent-rod molecules into a liquid crystal phase of counter-rotating chiral columns** (Nat. Comm. 13, 384, 1-11 (2022)): The flexibility to measure simultaneously both wide and small angle scattering under grazing incidence on XMaS was used to probe the ordering of columnar liquid crystals in thin films. In conjunction with data from I22 and I16 at Diamond, a phase transition on cooling from hexagonal columnar ordering to a more complex symmetry was observed. The low temperature phase consists of 8 chiral helical columns to form an antiferrochiral structure.
- **Surface resonant X-ray diffraction probing the charge distribution at an electrochemical interface** (J Phys. Chem. C 126, 9, 4612–4619 (2022)): *In situ* resonant surface X-ray diffraction studies were performed to assess the charge distribution and bonding mechanism at the electrochemical active interface during the adsorption of bromide anions onto a single crystal

Cu(001) electrode surface. The results concluded that charge reorganisation within the interface atoms occurred, rather than a charge shift between atoms. Such information and the developed metrological approaches are key to understanding a wide range of energy materials including the next generation of battery materials and their response to charge/discharge cycling.

- **Understanding interactions between metals and ionic liquids**: The unique ability of XMaS to conduct spectroscopic studies at both the Cu and Cl *K*-edges was used to explore the interaction between an aqueous-ionic liquid system and $CuCl_2$ as a function of concentration. The data were contextualised using Tafel plots from the laboratory and provide new insight into corrosion mechanisms.
- **X-ray absorption spectroscopy study of the state of Pd in pyrite**: A detailed study of the local structure in Pd doped pyrites was facilitated by the extended X-ray energy range at XMaS, allowing X-ray absorption spectroscopy studies at the Pd *K*-edge (25.514 keV). The measured XANES spectra allow the oxidation state of the Pd to be determined along with details of the nearest neighbours. The results provide new insights in to the physio-chemical modelling of the Pd migration and highlight potential techniques for ore refinement.

The 2023 XMaS Newsletter is being finalised for a release in mid-February 2024 and will be available online. It will be detailed in the next annual report.

Case studies

Case study 1 is the result of our collaboration and links with the Advanced Photon Source at Argonne National Laboratory. It used beam time awarded during the APS dark period to offset similar access that was granted to sector 4 at the APS for XMaS users during the 2019/20 XMaS shutdown. Case study 2 is XMaS in-house work performed by our PDRA in the field of geoscience using spectroscopic techniques to understand metals embedded in mining waste. The third case study is copied from the UKRI commission impact assessment of the ESRF by Technopolis in which the XMaS scientist experience was presented as a science highlight.

1. Deploying Low Temperature Single Crystal Uniaxial Strain at the XMaS Beamline at the ESRF.

(Dr. Phil Ryan, Advanced Photon Source, USA)

We used high-resolution X-ray diffraction and near edge X-ray fine structure (NEXAFS) spectroscopy to study the temperature dependence of uniaxial strain-induced ferroelectric order in quantum paraelectric nano-membrane $SrTiO_3$ free-standing films. By integrating a large displacement tri-piezoelectric strain device into the standard 4K ARS cryostat, we could apply > 2% in-plane strain by displacing the strain gap by up to 40 microns. XRD measured the tetragonality imposed on the unit cell along with accurate strain measurements. In addition, Ti *K*-pre-edge measurements indicate the onset of Ti driven central symmetry breaking. This measurement allowed a full temperature-strain phase diagram to be plotted in both structure and symmetry response to strain. The induced transition from paraelectric to ferroelectric behaviour was mapped out as a function of strain and temperature.

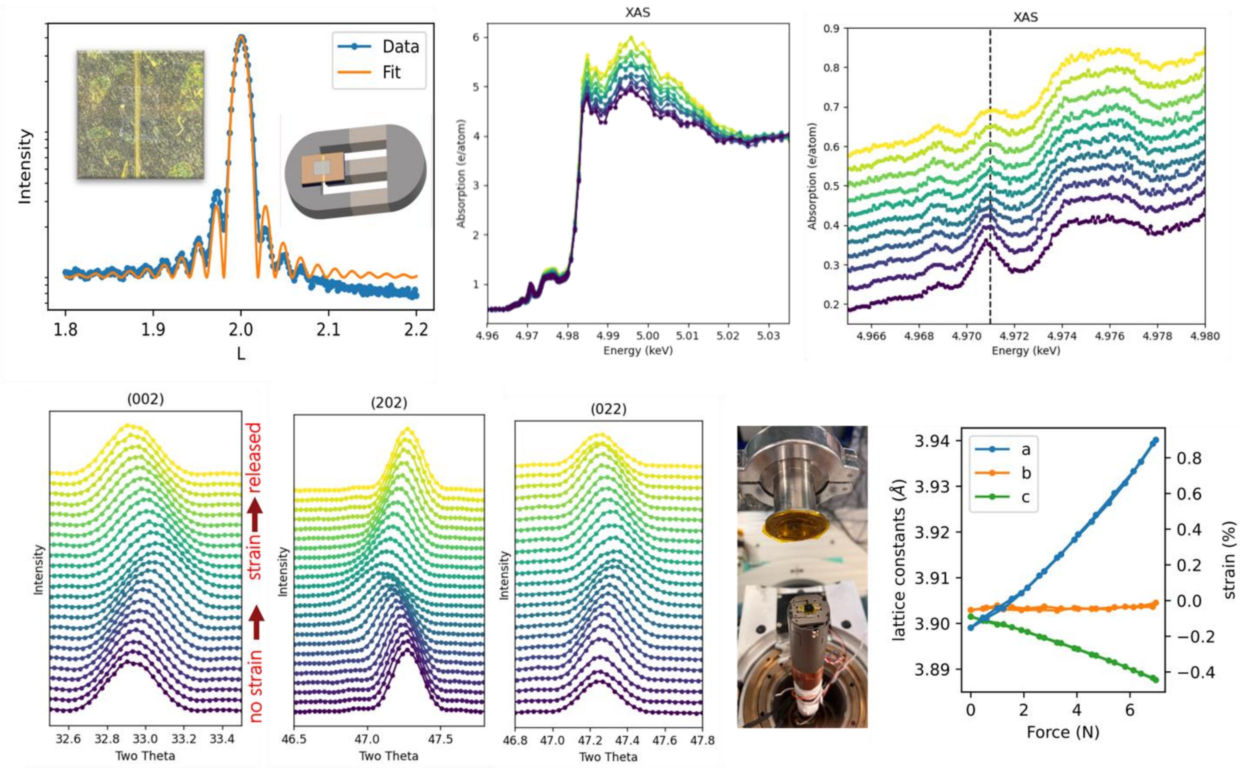


Figure 6: Top left; Theta – Two Theta scan of a 10 nm freestanding $SrTiO_3$ membrane on a Kapton sheet affixed to a tri-piezo stack uniaxial strain cell as pictured (inset). Bottom left; Strain cycle of the (002), (202) and (022) STO reflections at a fixed temperature. Bottom right; Compilation of lattice responses to the applied strain cycle of all three lattice parameters with strain percentage. Top centre & right: Ti *K*-edge spectroscopy illustrating pre-edge signatures of Ti related central symmetry breaking within the octahedral oxygen cage.

2. Spectroscopic studies in the Geological Sciences

(Dr. Olga Filimonova, XMaS beamline, University of Liverpool)

The fast rise in demand for 'critical' minerals, coupled with the political vulnerabilities linked to the geopolitical supply of these materials, may create potential obstacles to the widespread adoption of low-carbon energy technologies. To minimise the risks, the Critical Minerals Strategy recommended accelerating the growth of the UK's domestic mining capabilities. Our approach was aimed at developing new extraction and leaching technologies for exploitable deposits and mining tailings from what is traditionally thought of as exhausted deposits.

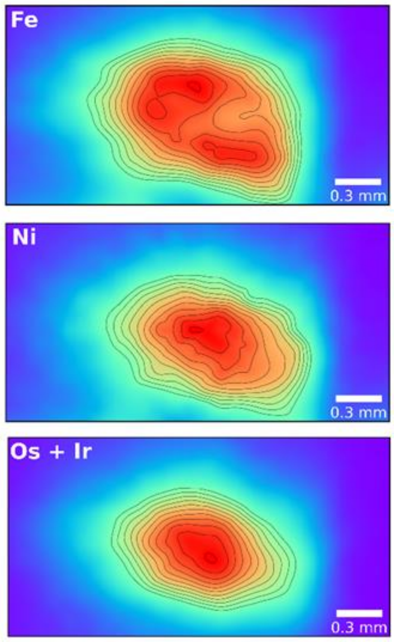


Figure 7: XRF mapping at 11.7 keV, signal for each ROI is normalised to 1.

Despite the low Clarke value (average abundance of elements), the contents of Pt, Pd, Os, Ag, Au, In, etc. in natural Cu, Zn, Fe, and Pb sulphides can reach a few tens and even hundreds of ppm. The form of occurrence, which, in addition to the micro-inclusions, their intermetallic compounds, or compounds with chalcogens, can be represented by so-called "invisible", or, in other words, a refractory form. The latter is a solid solution with sulphides or micro-inclusions/clusters in their matrixes and composes an economically important part of the total critical metals budget. Comprehensive knowledge of the state of the critical metals within the sulphide matrices, including local atomic environment and charge, is essential for the development of sustainable technologies for their extraction. The state of critical metals in sulphide matrices can be reliably determined only by spectroscopic methods and X-ray absorption spectroscopy (XAS) in particular. XAS analysis of natural sulphides and their synthetic analogues has experimental challenges including the overlap of emission lines, high matrix to metal signal ratios, sample heterogeneity, small grain sizes and low temperatures of decomposition.

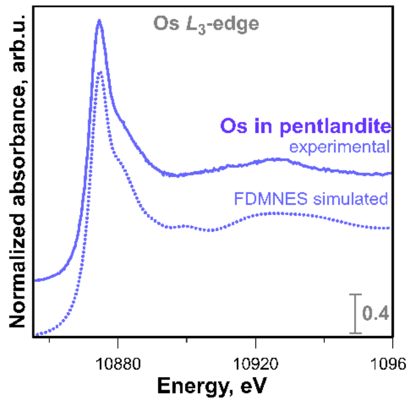


Figure 8: Experimental and simulated Os L_3 spectra of pentlandite with Os in a tetrahedral position.

Several experiments were conducted to address these challenges and to establish a protocol for future studies of minerals containing critical metals at the XMaS beamline. Natural pyrrhotite often contains exploitable amount of Ag, however, the contents of Ag in natural pyrrhotite are often too low for XAS analyses. We therefore studied synthetic analogues of pyrrhotite enriched with critical metals. The experiment was performed *in situ* using a capillary technique adapted from Raman spectroscopy where silica capillaries were filled with the Ag-bearing pyrrhotite and heated whilst simultaneously recording the fluorescence signal. The unique capability of the XMaS beamline to measure XAS at high energies, coupled with the availability of the Ge detector, will enhance the efficiency of these types of experiments. Another crucial aspect of geological analyses is elemental mapping and subsequent µ-XAS analyses. In collaboration with the University of Cardiff, we conducted a preliminary experiment involving the mapping of Os-bearing pentlandite $(Fe,Ni)_9S_8$ (Figure 7, 8). The KB mirrors and the capability to use µ-XRF mapping and µ-XANES/EXAFS will be extremely beneficial for the geological community over the next years and will further exploit the protocols developed to date.

3. XMaS Scientist Experience Case Study as reported by Technopolis for UKRI

(Impact evaluation of ESRF Final Report Neil Brown, Vivek Seth, Nadya Mihaylova, Charlotte Glass, António Neto, Aaron Vinnik, Julie D'Hont, Adebisi Adewusi, Tia J'Nae Murray, and Reda Nausedaite)

Included as a Vignette in the main report:

The XMaS Scientist Experience Scheme

Background

Since 2015, the XMaS national research facility staff have successfully developed an outreach programme aimed at tackling gender bias in Physics. They run an annual national competition, resulting in a group (typically numbering sixteen) of 16-18 year old female students taking a trip to visit the European Photon and Neutron campus in Grenoble, and touring both the ESRF and the XMaS beamline. Participants conduct mini research projects, give a presentation, tour the campus, and meet female scientists working at the facility.

Impacts seen

According to programme representatives, the main benefit for the participants is being surrounded by other like-minded science enthusiasts, helping to build up confidence in their own aspirations. The initiative also gives the students a positive image of a career in science, giving them something to aim for. The students create a WhatsApp group where they chat to each other and stay connected even after the event.

According to the programme staff, as a result of the trip, the students' perceptions and stereotypes of people working in STEM careers is challenged. They see, often for the first time, the collaborative nature of working in the sciences first hand, in contrast to the perception that many hold of life as a scientist being an isolating experience. The opportunity also provides an inspirational setting to see science 'beyond the classroom', but with direct relevance to the school curriculum on the one hand and research in the 'real world' on the other.

Additionally, the students see how the ESRF provides a space for international collaboration, co-working and interdisciplinarity, which the XMaS team is keen to showcase to young female students contemplating a career in science.

Included as an Appendix to the report with more details:

Summary

XMaS is an EPSRC funded National Research Facility located at the ESRF which supports the broad UK materials science communities. It provides free at the point of access to synchrotron radiation at the ESRF.[1] Since 2015, XMaS has successfully applied a growing outreach programme aimed at tackling gender bias in Physics. They run an annual national competition, resulting in a group (typically numbering sixteen) of 16-18 year old female students taking a trip to visit the European Photon and Neutron campus in Grenoble, and touring both the ESRF and the XMaS beamline. Participants conduct mini research projects, give a presentation, tour the campus, and meet female scientists working at the facility.

[1] https://www.xmas.ac.uk

Background

The initiative's origins came from the XMaS team discussing ways of encouraging and enthusing more young women into science, particularly physics. The team realised there was a need to inspire young women into careers in science, and to show them that success in a science career for women is possible. They developed a programme centred on practical experiments at the ESRF, but with the primary aim of introducing the young students to older female role models and the work they do. The idea is to allow the young students to talk freely to female scientists about their work, their research, and their careers; letting them ask any questions they may have in an informal manner. Ultimately, the programme looks to inspire and enthuse the next generation of scientists.

Originally the initiative focused on students from around Warwick and Liverpool but has now expanded to include the whole of the UK. To win a place on the trip, the students are challenged to conduct research on a famous female scientist. During their time at the facility, they are asked to conduct a mini research project and then prepare a presentation, which they present to their peers. They are taken around the beamlines at the ESRF and explore the facility more widely. They are then invited to eat lunch and socialise with the scientists at the facility as well as take up the opportunity to explore the city of Grenoble.

Skills benefits

According to programme representatives, the main skills benefit for the participants, is being surrounded by other like-minded science enthusiasts, which has helped to build up confidence in their love of science. The initiative also gives the young students a positive image of a career in science, being something within reach and to aspire to. The students have a WhatsApp group where they chat to each other, and stay connected even after the initiative.

The importance of the facility in achieving impacts

According to the programme staff, as a result of the trip, the student's perceptions and stereotypes of people working in STEM careers is challenged and changed. They see the collaborative nature of working in the sciences first hand, in contrast to the perception many of the students held that life as a scientist was a somewhat isolated experience. The opportunity also provided an inspirational setting to see science 'beyond the classroom', but with direct relevance to the school curriculum and international research in the 'real world'.[2]"

Additionally, they see how big research infrastructures like the ESRF provide a space for international collaboration, co-working and interdisciplinarity, which the XMaS team is keen to showcase to the young female students. In interviews, leaders stated that the ESRF provides a unique opportunity to showcase the benefits of international partnerships.

[2] https://www.materialstoday.com/materials-chemistry/articles/s1369702115002205/

Training, Outreach and Non-Scientific Impacts

Facility staff training: Recognising the increasing need for technical computing and to ensure suitable support can be given to data reduction and user needs, beamline staff attended Python training courses at the ESRF. French language courses are provided to new staff to better integrate them into working and living in France. Staff can also make use of training courses provided by the Universities.

Activities to promote the facility: Our aim is to continually grow and diversify the user community. This happens through our annual newsletter but also our annual user meeting which has returned to being in person (Figure 9). Despite a rail strike, the 2023 User meeting attracted some 40 users, about 25% of which were not currently facility users. Excellent discussions, centred around user science, enabled participants to consider future strategies for sample environments and scientific need.

Figure 9: Attendees at the 2023 XMaS User meeting in the Spine building at the University of Liverpool.

We publicise meetings and facility activities through X, formally twitter, and continue to actively engage with the Diamond Light Source (DLS) and the ESRF as well as the Catalysis Hub and the Royce Institute. Staff also attend national and international meetings and conferences:

- ESRF User Meeting, February 2023, Grenoble (booth for XMaS with slideshow).
- Winter school on Molecular Simulations (MolSim2023), January 2023.
- IOP Condensed Matter and Quantum Materials (CMQM2023), June 2023, Birmingham.
- International platinum symposium 2023, July 2023, Cardiff.
- ESRF Science and Student Days, October 2023.
- Physical Aspects of Polymer Science, September 2022, London
- Society of Glass Technology Annual Conference (SGT), September 2023, Cambridge.
- MML Conference, Seoul Korea
- IUCr Congress, Australia

Public Engagement: **Scientist Experience:** Our annual nationwide competition for Year 12 female students to visit XMaS and the ESRF attracted 166 applications. In Grenoble the girls spent two days touring the ESRF facility, taking part in the ESRF Synchrotron@Schools session and talking to numerous female scientists based at the ESRF, gaining the inside track on how far Physics can take you as a career (Figure 10). The competition remains a wonderful way to take learning out of the classroom and into the real world. We have just launched our 2023 competition, so if you know someone who fits the brief or you have contacts with Science Teachers, please encourage them to submit an entry. We also hosted a visit of 30 sixth former students from the University of Liverpool Maths School to the ESRF and XMaS during which they visited the site and interacted with staff, especially around the different career pathways that may lead to jobs in synchrotron radiation research and at other large scale international facilities.

Figure 10: Winners of the 2023 Scientist Experience discussing with female scientists at the ESRF canteen.

Societal, economic and environmental impacts: The impact of the ESRF and XMaS is included in an impact evaluation commissioned by UKRI and written by Technopolis.

Cost Recovery

UK access to synchrotron radiation is provided through the ESRF and the DLS and follows the "free at point of use to the best science" model. The Elsy report, submitted to BEIS in 2017, stress-tested this access mechanism in an independent review of National Large Facilities at Harwell and strongly supported it. XMaS is currently funded with the same user access model and there is, therefore, limited scope to generate direct cost recovery through user access. The upgraded facility has been designed with increasing efficiency as a key driver, ensuring that we can support more users and therefore be more sustainable by reducing the cost per user/experiment.

Cost recovery covering some staff expenses and instrument development has been secured through European funding in partnership with the University of Liverpool. Through Horizon2020, the European Metrology Programme for Innovation and Research EMPIR has funded several projects in which the facility has been a partner – Nanostrain (2013-2016), ADVENT (2017-2020) and OpMetBat (2022-2025). These projects have provided some limited funds to cover staff costs (60%) associated with the project work and ensured additional capabilities could be developed (40%), which are now freely available for the wider user community. The funds have contributed marginally to the actual running costs of the facility.

Year	Running Costs	Grants	Other Academic	Students	Industry	% Recovered
2019	£1,741,944	ADVENT: £124,489	£0	£0	£0	4%
2020	£1,141,944	£0	£0	£0	£0	-
2021	£1,141,944	£0	£0	£0	£0	-
2022	£1,141,944	OpMetBat: £103,417	£0	£0	£0	5%
2023	£1,141,944	£0	£0	£0	£0	-

Table 1: Additional Income through other funding sources.

Some cost recovery is achieved from our 11 commercial licences, generating a few k£ per annum. Following renegotiations between the Universities and the staff who developed the designs, much of this is returned to the original IP holders. The remainder is reinvested into supporting the NRF staff activities.

Further cost recovery can only feasibly be achieved by direct investment from industrial users. This access mechanism is handled through the ESRF but its uptake is traditionally low in the physical sciences.

Users

There was no user access to the facility throughout 2018-19 due to the ESRF shutdown for its EBS upgrade. Access was further impacted by COVID through 2020-21 with first users only having limited access from January 2021. In this reporting period however, we have been operating normally.

The user statistics compiled from the CRG access route are shown in Table 2 below:

Year	Student	RA	Academic	Industry	Other	No. Repeat	No. unique
2019	0	0	0	0	0	-	-
2020	0	0	0	0	0	-	-
2021	26	14	18	0	0	19	39
2022	31	22	52	0	0	10	80
2023	36	23	42	0	0	12	86

Table 2: User statistics over the grant period. We report the statistics by calendar year and not the reporting period.

Our user community reaches across the UK and internationally. The CRG users in 2023 came from 33 UK and 9 international research groups with 35% of visits being from new users. For the remaining ESRF allocated time, there were an additional 32 users from 5 UK and 7 international groups of which 6 were students and 5 were early career researchers. The percentage of these ESRF users who were new to the facility was 28%. Historical trends and a further more detailed breakdown of our user statistics can be found on our website.

The scientific area of the research is identified by users in their end of run survey. Research areas in this reporting period is compared to the facility average in Figure 11.

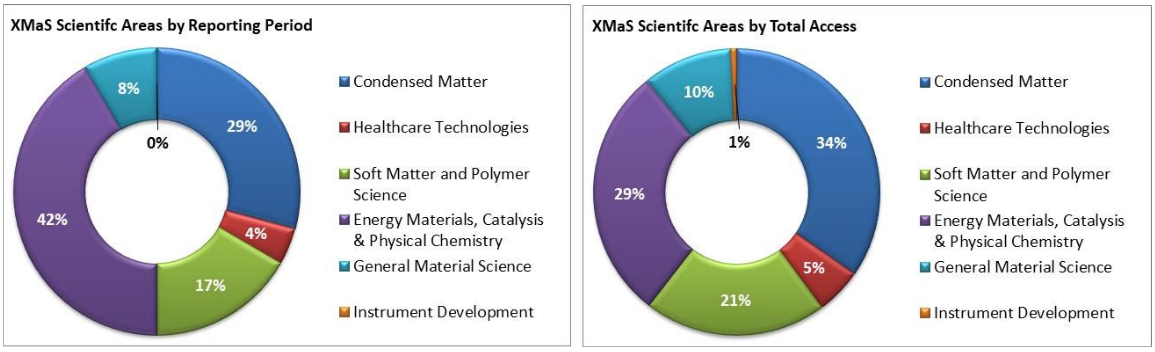


Figure 11: Research areas as reported by the users in their end of run survey. Data from this reporting period (left) compared with the facility average (since 2012, right).

Future Plans and Trends: A clear trend over the last few proposal rounds is for more proposals to make use of new co-modalities offered by the facility. This is reflected in the community vision, derived during the last statement of need: "*to build on the new enhanced capabilities, is for XMaS to deliver the correlative characterisation that is needed to understand ever more complex and heterogeneous materials and devices under technologically relevant conditions*." In the last proposal round 35% of applications requested *operando* studies with 20% requiring more than one X-ray technique to be applied simultaneously. Users have also requested a mechanism to facilitate rapid and high throughput experiments through a Block Allocation Group (BAG). Following advice from the PMC, we plan to implement a BAG in energy materials focusing on XAS spectroscopy and using our newly develop fast scanning modes. This BAG will work in conjunction with similar BAGs at DLS, bringing new users to XMaS and preparing for the DLS dark period.

User Surveys/Satisfaction

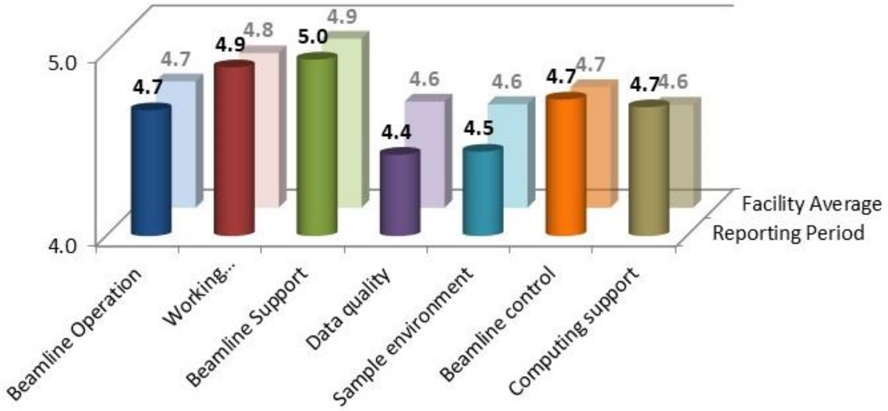


Figure 12: User survey results for the current reporting period (foreground) with historical averaged data in the background.

The principal investigator for each experimental run at the facility is required to complete, on behalf of the experimental team, an end of run survey scoring against operational performance as shown in Figure 12. Our return rate is over 95% for CRG beam times and direct follow-up with the users occurs whenever the satisfaction is lower than expected or issues are specifically identified in the free form text boxes which accompany the survey. User satisfaction in this reporting period is in line with the historical average and is generally very positive. User support by the beamline staff was highlighted by the external ESRF quinquennial review panel who noted in their report the "stellar levels of user satisfaction".

Some free form comments from users over the reporting period include:

> *"Excellent support was given from our contact who worked extremely hard to get our experiment to work."*

> *"Beamline staff support definitely was way more than excellent!"*

> *"The support provided on this beamline is exceptional. It is particularly beneficial to me as a relatively inexperienced beamline user. Making it possible for groups to come with less experience is such a fantastic opportunity and made things run very well."*

> *"Our beamline scientist was terrific, helping us immensely setting up the beam and kept checking on us throughout the experiment."*

> *"The beamline scientist was extremely helpful and dedicated."*

> *"Everything to do with the facility, equipment and support offered by XMaS, was fantastic."*

> *"Our local contact provided excellent support and made our beam time such a beautiful experience for all of us. We sincerely appreciate the support we received at the beamline and are always grateful to the team."*

> *"The support by the staff was excellent to ensure data were of the quality we needed to fit the EXAFS and support to make sure we could cycle the operando battery."*

> *"XMaS was, as usual, a great place to work. Thank you for the time and effort put in to producing our special sample environment."*

> *"This is an excellent facility with outstanding beamline scientists! The support was in place from beam time scheduling to experimental planning, to data collection and post-experiment help."*

Service Demand

Access to the facility is governed by a Collaborating Research Group (CRG) contract with the ESRF. This contract stipulates that 30% of the full flux beam time is allocated through public ESRF review panels in a worldwide call. The remaining beam time, which is available only to the UK community, is allocated through our independent Peer Review Panel (PRP) and assessed on scientific merit only. Beam time is scheduled in six-month allocations and where possible, all electron bunch modes of the ESRF filling are used. As noted in the quinquennial ESRF review "The beam time is allocated in an efficient way, where all the available shifts are used to schedule experiments. Aside from a marginal number of shifts used for commissioning, all shifts are filled with very low downtime."

Since the EBS upgrade there have been some problems with the beam flux and stability in the 16- and 4-bunch modes of the ESRF machine. Unfortunately, we have not yet been able to schedule users into the limited number of 4-bunch operational modes (used for commissioning and techniques development) but have continued to schedule users into the 16-bunch operational modes in which the flux is slowly returning to pre-upgrade levels. It is not possible to provide a chart showing demand and capacity monthly, but Figure 13 shows the number of proposals received through the two separate access routes (1st April and 1st October) as a function of time. We note that the number of proposals submitted is slowly returning to pre-pandemic levels (shown by the dashed lines in Figure 13). This trend mirrors what has been seen at other ESRF beamlines and other synchrotron facilities over the past year or so.

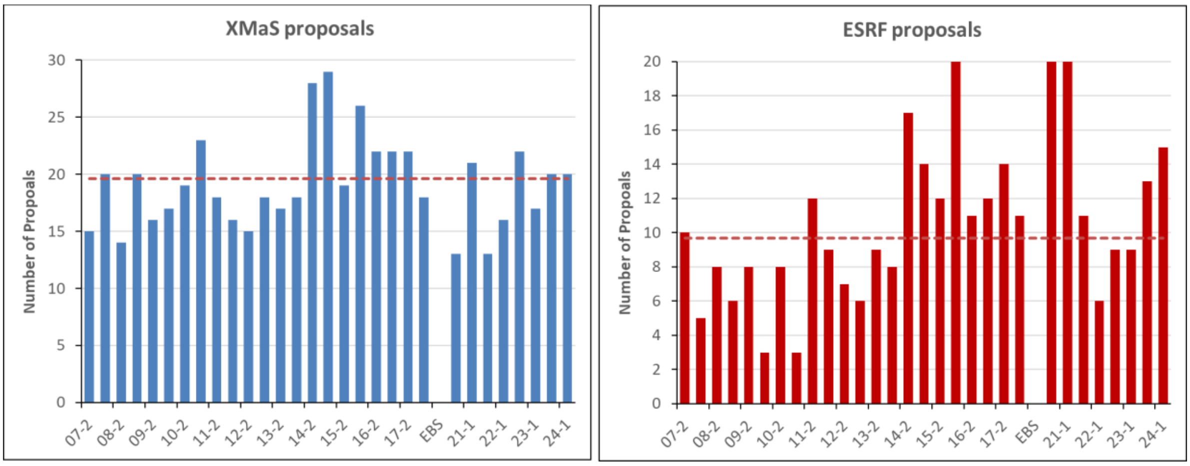


Figure 13: The number of proposals submitted to the XMaS NRF and reviewed by the independent review committees as a function of time. UK-based proposals (left) and international applications through the ESRF (right). The dashed lines are the averages calculated up to 2018 before the EBS shutdown.

Demand for the facility can be gleaned from our application statistics. Figure 14 shows the demand of the facility since 2013 in terms of shifts. We plot the ratio of the requested to available shifts in the left panel of Figure 14. A ratio of over 1 means that there is more demand than we can meet, and hence we cannot accommodate all user requests but must reject some proposals. The right-hand panel of Figure 14 shows the percentage of shifts applied for that were rejected. We note that the PRP regularly comments that the vast majority of applications have strong scientific merit and, in most cases, would warrant beam time. The open data points in Figure 14 correspond to a time when the statistics were dominated by the impact of the COVID pandemic with some experiments not possible due to remote working and access rules. This necessitated some accepted experiments having to be deferred as well as last-minute scheduling to accommodate both operational and user needs. Such actions skewed the statistics but even so, the average demand has remained at a manageable level.

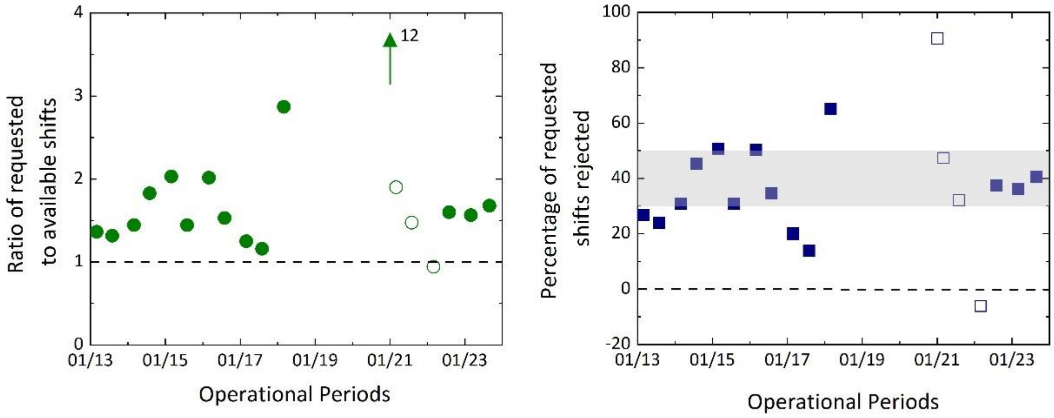


Figure 14: The demand for the facility represented by the ratio of requested to available shifts (left) and the same information but presented as the percentage of requested shifts that could not be accommodated. The open symbols on both plots are data that are compromised due to the COVID pandemic.

We aim for a rejection rate of between 30 – 50%, and in this reporting period were rejecting ~40% of the proposals in terms of shifts. This ensures that only the best scientific challenges are addressed whilst at the same time not discouraging users from applying for time.

Unfortunately, we cannot provide the same statistical analysis of the applications from our international colleagues through the public ESRF review panels. Typically, however, the rejection rate through this route is more than double that for the UK CRG time and approaches 80% in some application rounds. This indicates that the beamline is in considerable demand by non-UK users.

In addition to the synchrotron beamline itself, the XMaS facility also provides access to our “offline” facilities (the diffractometer based on a fixed tube X-ray micro-source and the facilities for electrical characterisation). These activities restarted in 2022 and are handled *ad hoc* by the beamline staff. Often, this instrumentation is used in tandem to provide support for some of the experiments scheduled on the main beamline.

Engineering and Physical Sciences Research Council

Risks

A risk register is maintained for the facility and reviewed every two months. It consolidates the risks into:

1. Operational Risks
2. Financial Risks
3. Data Risks
4. Ongoing Projects

A section specifically related to Risks associated with Covid was retired in February 2023 removing 11 risks from the register. A more generally defined risk associated with a shutdown of society and/or university research was added.

The Risk Register is reviewed, circulated, and discussed at each project management committee meeting, although it is updated more frequently as part of the day-to-day management of the facility. The discussions and input from the management committee have helped to define the risk register and keep it relevant and responsive to operational challenges. Monitoring the changing risk profile regularly has enabled project completion deadlines and targets to be agreed upon as well as highlighted several areas of action. The project management committee uses the risk register to effectively stress-test current operations as well as ensure short, medium, and long-term risks are appropriately managed and mitigated.

Summary of Risk Register

The general trend over the reporting period is for a reduction in overall risk associated with the facility, with many individual risks reducing.

Risk Management

- Risk Assessments – remains high and a concern, but progress is ongoing.
- Scenario planning – updated local "wiki" pages exist (currently being moved to Confluence), but formal scenario test yet to be completed.

Strategic Risks

- Unchanged high-level risks associated with long-term failure or access to the ESRF.
- Decreasing risk to long-term operations and user engagement with continuation funding from EPSRC.

Operating Risks

- 2 new risks associated with IT and data security.
- 2 increasing risks recognising the reliance on staff availability and expertise.
- 2 new risks - Data analysis computers and wider access to high performance and high throughput computing.

Project Risks

- 1 new risk recognising the exposure of the facility to accessing long-lead time items.
- 6 closed risks removed.
- 2 new projects added.

Currently, the risk register is not made public, but the latest version is always available upon request and is appended to this report.

Key Performance Indicators (KPIs) and Service Level (SLs)

XMaS operates under the following **Key Performance Indicators (KPIs)**:

A) The Number of Individual Researchers and University Research Groups [“users”] that have made use of the XMaS beamline in that Period. This should be expressed as a Total Number for that period.

B) Number of User Complaints received during the period. This should be expressed as a percentage of the Total Number of User Approvals made within the period.

C) The Uptime of the beamline within the period. This should be expressed as a percentage of the Total Available Time within that Period.

D) The Number of research outputs. This should be expressed as a Total Number for the period.

and **Service Level Agreements (SLAs)** with colour code based on meeting targets:

Facility Users will have access to facility staff for assistance on site.	
The facility will be operational and available for use for 80% (eighty percent) of the maximum possible operational time.	
The facility will train all new Users in the safe and effective use of the beamline.	
The facility will perform a minimum of 2 (two) publicity activities per year.	
The facility will generate a minimum of 15 (fifteen) research outputs per year.	
The facility will respond to all User enquiries clearly and quickly in line within 5 (five) working days for emails and 2 (two) working days for telephone enquiries.	
The facility will respond to User complaints within 10 (ten) working days.	
The facility will treat all proposals equally, fairly and in confidence.	
The facility will treat all Users equally and fairly.	
The facility will uphold high standards of integrity in all operations and in contact with Users.	

We now provide the underpinning data recorded against each of the KPIs and SLAs. Up to date data is provided on the website.

A) The number of Individual Researchers and University Research Groups visits: The facility hosted 101 user “visits” from 33 UK and 9 international University groups over this reporting period. These statistics cover the CRG access only and are compared with historical data in Figure 15.

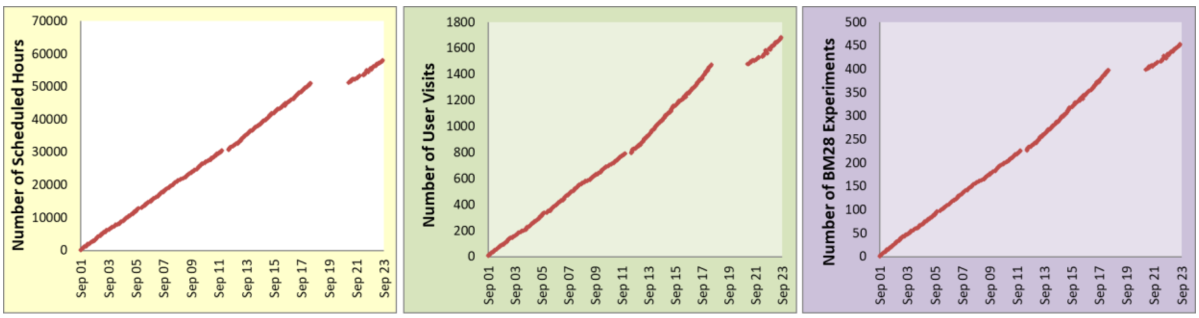


Figure 15: Historical performance metrics for XMaS including the number of scheduled hours (left), the number of user visits to the facility (centre) and the number of individual experiments performed on the beamline (right).

Further analysis of the type of user show that over the reporting period 36% of the users were students and 23% were early career researchers. 35% of the visits were from users attending XMaS for the first time.

B) **Number of user complaints:** XMaS has received zero (0) complaints over its lifetime of > 25 years.

C) **Uptime of the facility: 95.4%:** In this reporting period, the machine and operations were generally very good. We unfortunately lost 72 hours due to a failure of a sample environment which impacted a single user group. This experiment has been rescheduled in the next operational cycle and the faulty system immediately sent for repair. As a result of the incident we have reviewed our procedures for preventative maintenance and explored options for enhanced redundancies.

D) **The number of research outputs**: We track research outputs primarily by published papers and year of publication.

In 2023, there was a total of 19 published papers with the cumulative output exceeding 488 (Figure 16, top). The publication rate has returned to levels close to that seen pre-COVID. The dark period and the impact of the COVID pandemic is evident in the output statistics throughout 2019-2023.

Papers published in the last year continue the trend of increasing publications in journals with impact factors > 7 (Figure 16, bottom) which is now approaching 40%. Approximately 60% of the papers are co-authored with XMaS staff, which agrees with the historical average. In addition to peer reviewed publications, there are many conference and seminar presentations, which are more difficult to capture, and are *not* included in our KPI reporting metric.

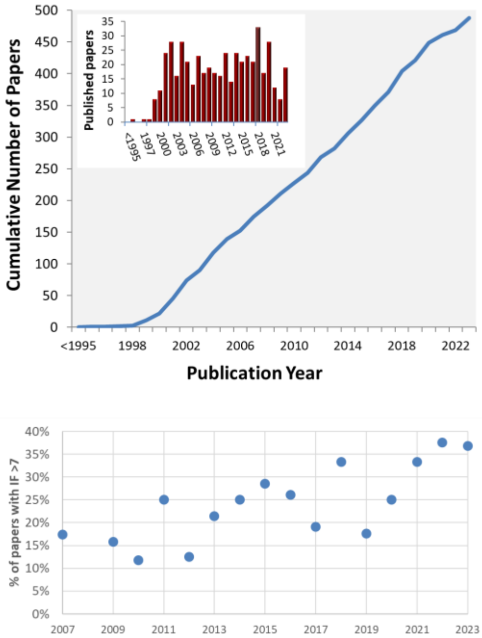


Figure 16: Publications from XMaS in peer reviewed journals. Top: cumulative and annual outputs and bottom the % of papers published in journals with impact factors >7.

Service Level Agreements

All SLAs have been met. The 2022 newsletter was disseminated in March 2023 and regular updates are communicated on the webpage and through twitter, now X. Direct engagement with the user community continued with the XMaS User meeting held in person at the University of Liverpool on the 31st May 2023.

Links

The UK has been at the forefront of synchrotron radiation (SR) provision for decades, building the world's first dedicated SR facility in 1981. Third generation machines (pioneered by the ESRF and Diamond Light Source (DLS)) increased the flux and beam quality, greatly enhancing the impact of SR across the physical and life sciences. Now, new technologies deliver diffraction-limited sources, hugely increasing the brilliance and coherence of the X-ray beams. These transformative designs are redefining the SR landscape with all major facilities upgrading - the ESRF upgrade was completed in 2021, the Advanced Photon Source (APS) in 2023-24 and DLS in 2027-29. SR facilities have a range of beamlines catering to their user communities and these are built up to be complementary, both within and between facilities. Access is based on peer-reviewed scientific excellence, with the ESRF limiting UK access to 10%, aligned to the UK's budget contribution. XMaS provides UK access to the ESRF over and above this limit. DLS has commissioned its beamline suite with XMaS operational and, as such, XMaS, DLS and the ESRF together constitute the UK research infrastructure for SR. As an integral component of the UK Synchrotron Radiation infrastructure, XMaS naturally has strong links with the DLS and has been operational throughout the development of the DLS beamline portfolio. DLS executives are fully cognisant of the capabilities of XMaS with senior Diamond staff chairing our Project Management Committee and providing help and support more generally. Representing the complementary neutron scattering community, Prof. Sean Langridge, the head of the ISIS Diffraction and Materials Division is also a member of the PMC. XMaS directors likewise sit on, and have chaired, DLS review panels and Prof. Tom Hase is currently chair of the DLS Science Advisory Committee and attends Diamond Board meetings. There is a free flow of information about current capabilities and opportunities for collaboration. Access to XMaS is major part of the DLS mitigation strategies for the physical science for the DLS upgrade and its concomitant dark period. We are actively working with Diamond stakeholders is preparing capabilities and ensuring Diamond users are aware of the opportunities at XMaS. Planned initiatives include working with the Energy Materials and Catalysis BAGS at Diamond and providing complementary capabilities and enhanced capabilities and throughput to the UK community.

Beyond Diamond, XMaS has strong links with sectors 4 and 6 at the Advanced Photon Source (Argonne National Laboratory, US). These links have been developed over many years, including knowledge transfer on sample environments and metrologies for magnetic and high-resolution diffraction. XMaS users were granted some access to beamline 4-ID-D during the recent ESRF shut-down and XMaS has reciprocating, hosting APS users during the APS dark period. We are also developing close contacts at beamline 4-ID at the National Synchrotron Light Source-2 (Brookhaven National Laboratory, US) as well as the Brazilian Light Source. The engagement with other facilities is built on long-standing collaborations and mutual trust and support. We fully expect this to continue and develop further in the years ahead.

XMaS has been working closely with key stakeholders at the UK Catalysis Hub (Research Complex @Harwell) to provide sample environments that exploit the unique ability to access both tender (2-4 keV) and hard (20-40 keV) X-ray photons interacting with the same sample volume. The focus here is on developing sample environments for operando experiments, which are crucial for the understanding of material function in their working environment that drives advances in catalysis and battery technologies. Prof. Andy Beale (UK Catalysis Hub) sits on our PMC and helps to develop our industrial interactions. We are also engaged with the Royce Institute which advertises our proposal round calls to its science community.

Industrial and non-academic collaborators have included: APS, AXO Dresden, BP, CPI, DLS, DSTL, ElectroSciences, EU Institute for transuranic studies, Futurum, Huber, IBM, Johnson Matthey, Mary Rose Trust, NPL, Nuclear Decommissioning Authority, PragmatIC Printing, P&G, PSI, Siemens, Smart Solutions, Surrey 5G centre, Swiss Light Source, US Navy, Vienna Fine Art Museum and the Advanced Materials Catapult (WMG). Third party activity has been through EMPIR-funded projects including Nanostrain, ADVENT and recently OpMetBat. XMaS is also involved in standard developments through the ISO TC201 "surface chemical analysis" committee and the recently completed COST action CA18130 "ENFORCE TXRF". XMaS supports users with a current EPSRC grant portfolio conservatively in excess of £200M and in applying for fellowships and international research programmes. Our users, often with industrial partners, are involved in several Centres for Doctoral Training, either funded directly through EPSRC or at a university level. In the reporting period, 21% of the beam time allocations had an industrial partner involved with the non-proprietary research.

Of course, XMaS benefits tremendously from being part of the ESRF and based at the European Photon and Neutron (EPN) campus in Grenoble, France. The EPN campus is an international science hub hosting the ESRF and the world's most intense reactor neutron source, the ILL, along with joint partnerships. In normal times, it hosts a staff of 1500 people, including 500 scientists and postgraduate students, and typically welcomes more than 8000 guest researchers every year. There is strong interaction between XMaS and the ESRF. Prof. Chris Lucas has sat on and chaired several beamline review panels and ESRF upgrade steering groups. The XMaS beamline scientists engage in day-to-day interaction with the broad EPN international scientific community, for example benefiting from the technical expertise both at the ESRF and the ILL in the development of novel sample environments. Users to XMaS become part of this international community often developing new scientific ideas that lead to collaborative projects involving scientists from across Europe and beyond. Many of the UK driven experiments have international collaborators, some of whom also apply for time directly through the ESRF international route. The wider impact of the ESRF to the UK is detailed in a UKRI commission report published in March 2023.

XMaS staff are heavily involved in developing international standards through the ISO TC201 and BSI CII-60 committees and have worked with international partners with the COST action CA18130 (ENFORCE) to define terminologies and obtain reference spectra needed for total reflection X-ray fluorescence (TXRF). The benefit of these interactions are difficult to quantify but provide huge knowledge transfer to and from the facility and are of crucial importance in maintaining the scientific competitiveness of the UK.

Improvements and Future Plans

Facility Access

We are continuing to plan for a BAG access route in "energy materials" in partnership with DLS and the B18 beamline. This was planned for the first quarter of 2024 but will now begin in October 2024 due to scheduling demands. Following the successful refunding programme, we are now designing new sample mounts which will facilitate faster sample throughput, and which will enhance the BAG user experience and impact.

Although some 20% of the beam time allocations had some industrial engagement with the PIs, we recognise that there is a continuing need to attract new industrial users for proprietary research, as well as support research programmes at the academia-industry interface. To achieve this, we will, over the next year, generate a marketing pack specifically tailored to industry highlighting the capabilities, data and services that can be offered. We will devote several shifts to the collection of industrially relevant data for case studies and develop protocols for data reduction and analysis. We will necessarily work with the industrial engagement team at Diamond to ensure that we can accommodate any needs from industry during the Diamond dark period.

In terms of general user access, we are also planning to evaluate and significantly decrease the environmental impact of travel to the XMaS facility in a way that also improves the efficiency of the experimental programme and promotes green travel options to other stakeholders. Sustainable travel options based on trains will be encouraged along with the development of 'travel bundles' which encompass on-line training modules. The travel bundles will provide scientific training, software support and experimental planning as well as advocate the adoption of sustainable research practices more generally. The plan is that these training packages will use model data from XMaS, links to analysis resources as well as videos and real user experiences. Although a specific EPSRC grant application to support these activities was unsuccessful (December 2023) we hope to implement some of the ideas that were generated during the preparation of the application to encourage sustainable user travel options in the future.

Facility Management

XMaS is committed to implementing its published EDI policy. In partnership with the ESRF Safety Office, we will endeavour to ensure that the facility can accommodate any user, but this may require an individual needs assessment. We continue to monitor the EDI requirements and needs of our users through the project management meetings and update policies whenever they are needed. Many of these policies are informed by the EDI activities at the Universities of Liverpool and Warwick.

Technical Upgrades and Equipment Developments

The few years preceding this review period saw significant investment and capability uplifts to the facility that had been requested by the users. These were supported in part by bids to the EPSRC Capital Equipment Calls in 2020 and 2022. In this review period the new equipment and capabilities were consolidated and incorporated into the beamline control system.

Detectors

The new extended high-energy range of the beamline facilitates spectroscopic studies at X-ray energies up to 47 keV. To access these energies, new detectors were needed. To increase efficiency, both in terms of count rates and solid angle intercepted, detectors with multiple sensing elements are an advantage. A suite of enhanced high energy detectors are now arriving at the facility (Figure 17). Two Si drift diode systems (2 – 30 keV) including a single 2 mm thick sensor (commissioned on site and available for users later this year) and a 4-element with 1 mm sensors (due to arrive in Jan. 2024). A 7-element Ge cryogenic free detector has arrived but during its testing phase several serious issues have been identified (vibrations, port access for vacuum). Solutions have been proposed and we are in discussion with the manufacturer as to how proceed.

Figure 17: Energy dispersive detector suite.

The new detectors are supported by a second XIA Falcon-X high throughput counting chain which has been integrated into the beamline controls and data acquisition software.

Fast Energy Scanning

We have successfully modified the control software with support from the ESRF computing teams to allow fast scanning of the monochromator for spectroscopic measurements. Our preliminary tests used ion chambers before and after the sample to record the data as shown in Figure 18. The fast scans are fully consistent over a broad energy range with the step-by-step approach we had before. For comparison at the Ag *K*-edge (Figure 18, top), the step-by-step scan took 8 minutes whilst the fast scan took 15 seconds. To demonstrate the versatility of the facility, the data at the W L_3- and Au *K*-edges were taken back-to-back. The next stage is to validate the approach using the energy dispersive (SDD and Ge multi-element) detectors. Fast, multi-motor scans are possible in the future but will require the operating controls to be fully upgraded to the BLISS system.

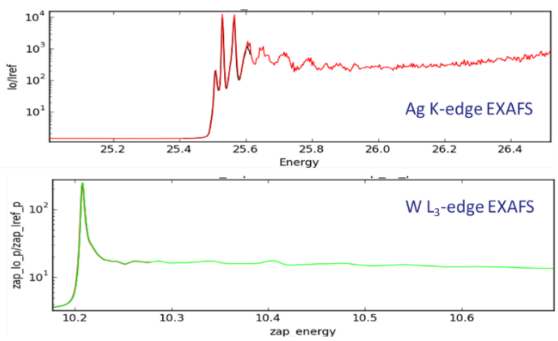


Figure 18: Comparison between EXAFS data recorded using the convention step scan (black) and fast scanning (red/green) at the Ag *K*- and W L_3-edges.

Upgrades of obsolete systems

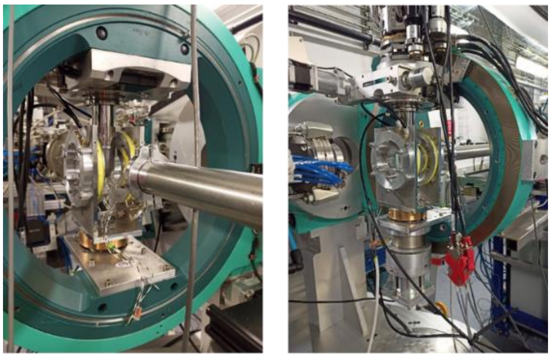
After operating the facility for over 25 years, there are various obsolete systems that have been identified through the risk register as being increasingly likely to cause a single point failure and concomitant downtime. There is therefore a rolling project to replace relatively small-scale items such as Keithley current meters to more modern equivalents over the next year.

BLISS

A major upgrade planned through 2024 is the replacement of the obsolete SPEC monolithic control and data acquisition software with the new ESRF standard - BLISS. The new BLISS standard separates controls from data acquisition and is Python-based rather than C-based. This will bring about a big change in the feel of the controls but is also an opportunity to make changeovers between different configurations easier. Current macros will all need to be rewritten in Python and commissioned. A one-year position is available within the new grant to help with this changeover and the post will be advertised in the spring of 2024. The X-ray laboratory source will be used as a sandbox and test bench for the integration of multiple pieces of equipment and to test the rewritten macros. This will ensure that downtime on the main beamline is kept to the bare minimum. As a result of the transition to BLISS, more exotic scanning protocols and fast data collection strategies will become possible. The facility will also move to the hdf5 “world standard” file format and allow use of analysis software developed at the ESRF, Diamond and other facilities. The transition to BLISS will be completed in 2025.

Low Field Magnet

There have been several user groups working with very magnetically soft materials. These typically saturate even in the remanent field of conventional magnets. To facilitate these experiments, a new magnet was designed and commissioned. Two pairs of water-cooled Helmholtz coils generate a uniform field (±2 mT) but with a zero remanent field (Figure 19). The system accommodates the standard cryostats and can be mounted to apply the fields parallel or perpendicular to the beam direction.



B orthogonal to the beam | B along the beam

Figure 19: New Helmholtz coils magnets mounted onto the diffractometer.

Gas Delivery System

With the finalisation of the KB system and its mounting onto the flight delivery pathways, we can now plan for the full deployment of the gas delivery system (this necessarily had to be paused while the KB mirror project was underway. The mass spectrometers are already available (Figure 20) and the mass flow controllers have been ordered. We are working with Lucy Costler-Wood (Johnson Matthey) and the Harwell Catalysis Hub in designing the complex gas system that will be able to provide gasses for both the ion chambers in the beamline and to the specialist sample environments. We have leveraged the expertise from BM23/ID24 at the ESRF for the preliminary piping and bottle storage configurations that are already in place.

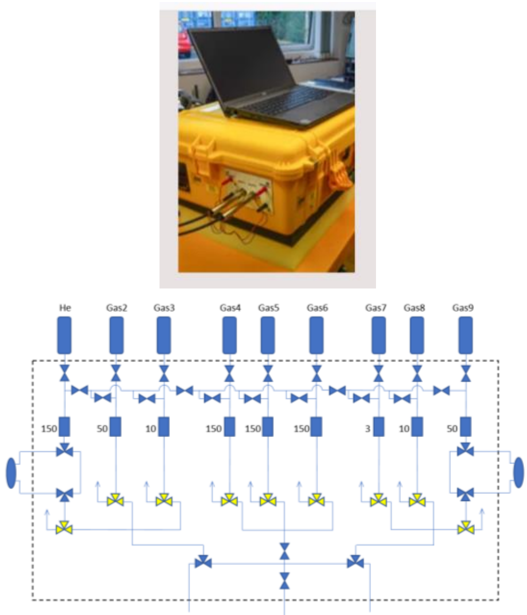


Figure 20: Mass spectrometer and potential gas flow schematic for the beamline.

Raman spectroscopy

Figure 21: Raman system from Renishaw.

Raman spectroscopy is widely used in the field of organic, polymer chemistry, polymer physics and physical chemistry. This technique gives 'fingerprint' information, which means that every peak can be associated with a specific bonding between specific atoms. That is why Raman spectroscopy can be applied to detect the presence of a particular molecule (additive) in the sample or to check chemical changes in the material. In this way, Raman is a suitable complement to Infrared spectroscopy and Nuclear Magnetic Resonance. Chemical mechanisms such as polymerisation, curing, oxidation and degradation; physical processes such as crystallisation, stretching or even conformational shifts have been studied with this technique. Raman spectroscopy also has a number of applications in the study of electrochemical systems, for example related to applications in batteries and hydrogen fuel cells. A dual wavelength Raman spectrometer will be arriving at the beamline in January 2024 (Figure 21). This will require laser designated laboratory space and fibre-optics coupling for delivery to the beamline so that the X-ray and Raman measurements can be performed simultaneously. Installation and commissioning of this system will take place in 2024 with user availability expected towards the end of the year.

Potentiostat/Galvanostat

The current potentiostat available at the beamline, a PAR263 potentiostat/galvanostat, is 20 years old. It is controlled and integrated into the SPEC software through a GPIB interface. The nature of this software communication significantly slows down the measurement time and excludes the possibility of fast (< 1 second) data acquisition. Furthermore, the current setup allows steps in the applied voltage/potential but does not fully support the more advanced potential cycling or scanning options that are required, for example, for the characterisation of batteries and fuel cells. Over the last 20 years the technology has much advanced (due to the fast development of renewable energy applications that are based on electrochemical systems and require more advanced potential cycling). Four Gamry potentiostat/galvanometer units have been ordered and will be delivered in 2024. One of these is a state-of-the-art instrument for electrochemistry and impedance spectrometry measurements that will be fully integrated into the beamline control system. The remaining three are more basic but will allow programmable charge/discharge cycling of battery materials in support of the OpMetBat project and user experiments, particularly as part of the future energy materials BAG.

Software for Users

The esaProject code has been rewritten by Prof. M. Dowsett and is available to users via the XMaS webpage. We continue to explore ways to project and present reciprocal space volume in reciprocal space as well as HKL using codes developed by Dr. Sam Seddon (IXIAN) in python and Prof. Markys Cain in IDL. Tests continue with an aim to make them more generic and user friendly. The IXIAN code is currently available on GitHub. Software links are being uploaded onto the webpage.

A process of identify and signposting software for user analysis will be implemented through 2024 as both a repository but also with training and model data. We have compiled a list of software for spectroscopy and are working with the Spectroscopy group at DLS to build the training modules.

An extensive and growing section of manuals and "how-tos" is now available to users via the XMaS wiki server which is accessible from the beamline or within the ESRF firewall.

Governance Structure

The organogram of the staff associated with the facility is shown in Figure 22. Governance and management are via the directors at the Universities of Liverpool and Warwick. External committees provide oversight and support (PMC) as well as independently rank access requests (PRP). This governance structure has been in place for over 25 years and has proven to be robust. To ensure continuity of management in case of unexpected events, there is a co-director at each University.

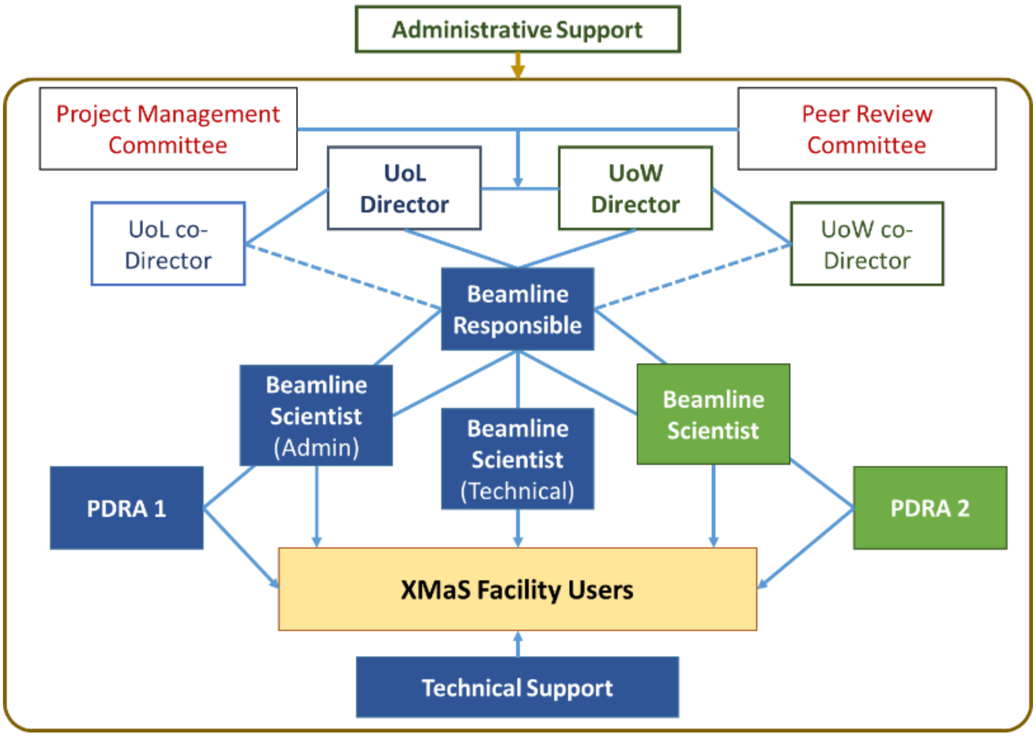


Figure 22: Organogram of the XMaS management and governance structure.

There were 4 permanent on-site staff members; 3 beamline scientists providing user support (one, acting as beamline responsible, one, covering on-site administration) and an additional scientist leading technical developments. In addition to the beamline scientists two on-site PDRAs are employed to further the scientific development, data analysis procedures and software development.

Project Management Committee (PMC)

The project management committee is drawn from experts in the use of synchrotron radiation and/or central facilities as well as our EPSRC liaison members. Dr. Chris Nicklin became chair of the committee at the start of the review period. A broad cross-section of experts ensures that roles span user and industry representatives as well as bringing operational expertise, sharing of best practices and fertilising inter facility collaboration. The facility works with the PMC to seek advice, help prioritise scientific projects and ensure activities are strategically aligned with the needs of the UK materials communities. The PMC reviews all KPIs and SLAs along with the budget and the risk register.

Dr. Chris Nicklin	Diamond Light Source [Chair]
Dr. Maria Alfredsson	University of Kent
Dr. Rosa Arrigo	University of Salford [Chair of PRP]
Prof. Andrew Beale	University College London
Prof. Markys Cain	Electrosciences Limited

Prof. Karen Edler	Lund University
Prof. B.J. Hickey	University of Leeds
Prof. Sean Langridge	ISIS, Rutherford Appleton Laboratory
Prof. Bill Stirling	Institut Laue Langevin and past director of ESRF

Peer Review Panel (PRP)

During the reporting period, Dr. Rosa Arrigo (Salford) took over as chair of the committee from Dr. Roger Johnson (UCL) who retired from the committee. His area expertise was replaced by a new member, Dr. Mark Senn (Warwick). The committee brings a broad range of expertise being comprised of extensive users of synchrotron and neutron facilities. The independent committee ranks the submitted proposals and prepares a recommendation for the XMaS management to schedule users. They report directly to the PMC and highlight trends and user needs seen in the application rounds. They act as the user voice of the facility and compiled the statement of need for the recent funding request.

Dr. Rosa Arrigo	University of Salford [Chair]
Prof. Andrew Hector	University of Southampton
Dr. Ellen Heeley	Open University
Dr. Karen Syres	University of Central Lancashire
Dr. Larissa Ishibe-Veiga	Diamond Light Source
Dr. Mark Senn	University of Warwick
Dr. Maximilian Skoda	ISIS Neutron and Muon Source

Any comments on the facility from your advisory board:

Chris Nicklin, Chair of PMC: The Project Management Committee (PMC) meet twice a year and are grateful to the directors and beamline team for all their efforts in preparing the documentation and presentations. We view the way that XMaS is run as exemplary and appreciate the openness of the discussions and how this is viewed as a partnership with the PMC. The PMC is well balanced with members representing relevant science areas or with technical insight that the beamline team utilise very well. One of the strengths of XMaS is its innovations in terms of instrumentation development and science delivery, most recently in working on correlative studies. We also appreciate the in-house science programme that we believe enhances the quality of the user science delivered and the end-to-end delivery including aid with proposals, analysis, and publication. It is a pleasure to help with this facility, the governance structure is proven and continues to work really well and the relationship with the directors ensures co-development of the processes (e.g. risk register) and the technical/science strategy. It is an important component of the UK synchrotron science strategy and I look forward to seeing it deliver and develop over many more years.

Engineering and Physical Sciences Research Council

Website

The NRF's website is www.xmas.ac.uk. The webpage has been continually refreshed and restructured to reflect the changes on the beamline and its new capabilities. Clearer signposting and a more logical layout have been developed and there is now a section dedicated to industrial users.

We continue to modify the design based on the latest accessibility criteria focusing on the use of colour, formatting, the overall page structure as well as the readability including how images and videos are embedded.

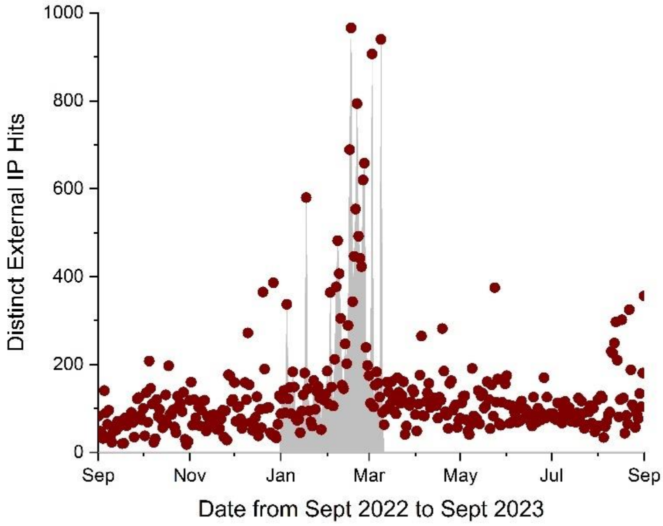


Figure 23: Distinct website views per day over the reporting period.

Web analytics are provided by the host server at the University of Warwick and have recently been transferred to Google analytics which will enable more a more detailed report in 2024 as more statistics are automatically collected. Excluding search engines, the website receives on average 132 page views per day, twice the number than in the previous reporting period. The daily website views from distinct IP over the reporting period are presented in Figure 23. The light grey shaded region corresponds to when applications to the 2023 Scientist Experience were open. The deadline was the end of February and accounts for the sharp increase in visits in the spring of 2023.

Additional Information

In addition to review by ESPRC, XMaS is also reviewed every 5 years by an external panel of experts initiated by the ESRF and its Science Advisory Committee (SAC). This review was held in May 2023 with a panel comprising Prof. P. Willmott (Swiss Light Source), Prof. J. Hastings (Stanford SLAC), Prof. K. Kvashnina (ESRF), Prof. B. Murphy (Kiel and ESRF council) and Prof. G. Portale (Groningen).

The report was very positive noting that “the beamline is in excellent shape”. Commenting on the uniqueness of the facility, it noted “the new source has almost doubled the available energy range and allows users swiftly change the incident energy range between tender and hard X-rays, which is very appealing for catalysis, energy, and materials science. This would be impractical for an insertion-device beamline.” They panel stated that “the beamline's unique features can be exploited to allow combinatorial and multimodal analysis in many fields of material science. The staff are very competent, with complementary scientific background and can provide excellent support for the user community.” In terms of capability, they found that “XMaS provides a combination of spectroscopic and diffraction techniques over a large photon energy range, making it complementary to many beamlines worldwide. The beamline has an impressive range of sample environments, including cryogenic environments, oven/heating, B-fields (up to 4 T), E-field up to ±10 keV, calorimetry, potentiostat/galvanostat, Raman spectrometer, and mass spectrometry. Indeed, we do not believe there is a need or even a desire to limit or concentrate the activities on specific fields. One of XMaS' main plus points is its flexibility to cater for a broad range of science.” The panel recognised “the focus of XMaS is to provide a successful user-oriented scientific support. The users are provided with a very high level of support reflected in the stellar levels of user satisfaction.”

Research by users: “We have judged as outstanding some of user output and note that the XMaS capabilities have attracted a broad cross-section of the UK and international science communities.”

Research by staff: “There are many scientific highlights and excellent publications from the beamline staff. The research activity being done collaboratively with users is a win-win scenario and a success of the beamline. The review committee was impressed by the output of the two young researchers.”

Future scientific directions: **“**The committee strongly encourages the beamline staff to pursue and optimise the unique capabilities of the beamline to smoothly change energies from the tender range (as low as 2.1 keV) to the hard X-rays (up to 47 keV) in the fields of catalysis, energy and environmental science (operando and in-situ experiments). New multi-modal capabilities allow materials to be characterised in terms of composition [XAS, XRF, TXRF] as well as spatial ordering on local [XAS, WAXS, XRD, xPDF (currently under development)], and larger [SAXS, XRR] length scales. This is a unique capability of this beamline. Moreover, the beamline will soon have also complementary Raman spectroscopy capabilities, which can enhance the chemical sensitivity during multimodal analysis.”

Summary of ESRF panel recommendations

1. Remain flexible and continue to use the complete energy range to provide multiple techniques on the same sample.
2. Clearly define the science drivers behind any novel instrument/technique developments and reassess these regularly based on user interest and results obtained.
3. Continue to develop sample environments and ancillary methods such as electric and magnetic characterisation in response to user demand.
4. Protect uniqueness of BM28 in the milieu of ESRF and DLS (and the latter's upgrade program).