Review

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XMaS @ the ESRF

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This paper describes the motivation for the design and construction of a beamline at the European Synchrotron Radiation Facility (ESRF) for the use of UK material scientists. Although originally focused on the study of magnetic materials, the beamline has been running for 20 years and currently supports a very broad range of science as evidenced by the research topics highlighted in this article. We describe how the beamline will adapt to align with the ESRF's upgrade to a diffraction limited storage ring.

This article is part of the theme issue ‘Fifty years of synchrotron science: achievements and opportunities’.

1. Introduction

In 1990, the European Synchrotron Radiation Facility (ESRF) in Grenoble, France was still being built—it did not take its first regular users until 1994. As a third-generation synchrotron source, the magnetic lattice was designed primarily around its suite of insertion devices which were the flagship X-ray sources for the facility. The bending magnets were simply there to steer the electron beam around the storage ring. However, the radiation from these bending magnet sources was, in fact, superior in terms of brightness and brilliance to that of most European national machines of the time, including the UK's SRS facility at Daresbury Laboratory. Thus, given the relatively large floor area of the ESRF ring the idea arose to make the bending magnet ports available to

national consortia with the proviso that the resulting beamlines should be operationally cost neutral to the ESRF with one-third of the beamtime (using the highest electron current mode) returned to the ESRF for its users as ‘rent’ for the use of the photon beam. The ESRF called these consortia Collaborating Research Groups (CRGs).

At about this time, Bill Stirling (then Head of Physics at Keele University) and Malcolm Cooper (in Physics at Warwick University) were led to believe that a bid to construct a CRG beamline for the physical sciences might gain support from the UK Research Council and from the ESRF itself. Both Stirling and Cooper were regular users of the SRS as well as other synchrotrons around the world for studies of magnetic materials. The first attempt at an estimate of the cost of building the beamline was based on little more than a perusal of equipment catalogues and discussions with Daresbury staff. This resulted in a figure of some £750 000, which turned out to be a gross underestimate. Fortunately, the Engineering and Physical Sciences Research Council (EPSRC) decided that a proper design study was necessary and gave Cooper and Stirling a grant which supported an X-ray engineer and a scientist, David Paul and David Laundy, both based at Daresbury. The remit was to design a diffraction beamline with a beam delivery and optics system which allowed for both focused and unfocused monochromatic beams, but also a white beam capability. The unfocused monochromatic beam was incorporated to allow higher wavevector resolution, but it was expected that the mainstay of the beamline would be the focused monochromatic beam. The actual focusing optics were the subject of much design work and debate with one contender design based on a parabolic pre-mirror to produce a parallel beam onto the monochromator (for higher wavevector resolution), with a second parabolic mirror after the monochromator to focus the beam onto the sample position [1]. The difficulty of aligning such a combination was brought home by a special Advisory Panel convened by EPSRC under the Chairmanship of Denis McWhan; the panel strongly advised that the optics design be as simple as possible and use just one focusing element—a post monochromator toroidal mirror. With hindsight it was absolutely the correct decision and this simple system has operated reliably for over two decades, although the more complex design has been deployed successfully on several other beamlines, including BM02 at the ESRF. What emerged from our design study was a beamline with a constant exit Si-111 water-cooled monochromator as the first optical element, followed by a toroidal mirror with a heavy duty, very versatile multi-axis (Huber) non-magnetic diffractometer (6 primary circles and 11 operational axes with a 30 μm sphere of confusion) at the focal point some 25 m downstream from the optics. The beamline was built facing the ‘soft’ end of the bending magnet which produced radiation with a critical energy of 9.8 keV, chosen to maximize the flux at low and conventional X-ray energies driven by the scientific interest in studying magnetic materials such as uranium for which the M edges of the actinides were of specific relevance [2]. The provision for white beam and for unfocused monochromatic beam were retained [3], although their use is somewhat of a rarity these days.

The financial bottom line came to an estimate of over two million pounds, including the salaries for staff seconded out to Grenoble to build it. The project proposal was accepted by EPSRC and the ESRF and in early 1996 a small team (David Paul, David Bowyer, David Laundy and Simon Brown) moved out to the ESRF to begin the daunting task of building the beamline from scratch. They were joined later that year by Malcolm Cooper. We were lucky to have as advisers Bruce Forsyth, a neutron diffractionist and expert on magnetic materials, and Mike Hart, a very eminent X-ray diffraction expert. Indeed it was Bruce Forsyth who coined the name, ‘**XMaS**’, for the beamline. At the time, the acronym stood for **X**-ray **M**agnetic **S**cattering, given that the scientific program for the beamline was heavily biased towards studies on magnetic materials. The name has stuck, and fortunately remains accurate today even though the beamline presently supports a far broader scientific remit with studies now encompassing a more general ‘Materials’ theme, so **X**-ray **M**aterials **S**cience we have now become.

XMaS celebrated 20 years of user science in 2017 [4] with continuation funding secured from EPSRC at largely 5-year intervals; 1997–2002, 2002–2007, 2007–2012, 2012–2018 being latterly categorized as an EPSRC Mid-Range Facility. Recently funding has been obtained for the period 2018–2023 as a National Research Facility. We have had the support of an excellent on-site

Figure 1. The XMaS beamline. (a) The diffractometer in a vertical scattering geometry with the polarization analyser attached. (b) A view of the experimental hutch showing the diffractometer configured for WAXS studies. (Online version in colour.)

team at all times. Joining the initial team, Paul Thompson helped install and commission the original components with Simon Brown and they have been developing sample environments ever since, ably supported by John Kervin who provides additional technical support. During the initial operations, Laurence Bouchenoire and Jonathan Wilmschurst were PhD students, with Laurence then joining Anne Stunault as a beamline scientist, a role ably filled at various times by Danny Mannix, Peter Normile and currently Oier Bikondoa and Didier Wermeille. We have also benefited from the expertise and help of Matt Longfield, Gemma Newby and Sean McMitchell who have been our postdoctoral researchers.

Over the 20 years of operation, the beamline has existed in a form that the originators and early users would recognize (figure 1). The key components—the monochromator, mirror and diffractometer—have remained unchanged. The very significant incremental changes that have been made are largely in terms of the variety of sample environments and beam-conditioning elements. For example, the beamline now provides several different magnets; a 200 mT electromagnet (which can be housed in a vacuum chamber), a 1.5 T electromagnet and a 4 T superconducting warm bore magnet. Non-ambient temperatures are provided by a suite of ^4He and ^3He cryostats allowing experiments to be performed down to 1 K [5]. A cryo-furnace increases the operational temperature to 700 K. Electric fields up to several kV mm^{-1} are also available, fully incorporated into the cryostats and control software. There is the provision of various two-dimensional area detectors as well as polarization and beam conditioning elements (filters, phase retarders, micro adjustable slits, etc.) as well as access to wider ESRF resources. A wide range of sample environments for *in situ* and *in operando* studies have been designed and are available for users on both the synchrotron beamline and off-line laboratories. There is also a provision for handling radioactive samples, a useful legacy from the first experiments performed on the beamline. Underpinning this expansion of capability is an active programme of commercialization of our in-house designed equipment [6], for example, the accurate and reproducible x - y - z positioner for cryostats and sample holders [7]: overall XMaS holds a dozen licences for instrumentation sold to synchrotrons around the world.

Although the original design specification was for a diffraction beamline, both small (SAXS) and wide angle X-ray scattering (WAXS) along with grazing incidence (GI) capabilities have been added to the operational portfolio. The lower operational photon energy limit has been reduced to 2.1 keV [8] which has resulted in an upsurge of spectroscopy studies. As a result of our bespoke sample environments, our capabilities and capacity have continually evolved and

Figure 2. A breakdown of scientific research area covering the last operations period (2012–2018). (Online version in colour.)

expanded allowing a much broader range of scientific challenges to be tackled than is normally found on a single SR beamline (figure 2). The resulting user community is very multidisciplinary, spanning physics, chemistry and materials with additional activities across the life sciences and healthcare areas. XMaS has used this breadth of experiences to ensure that the best practices of one community are shared, adapted and implemented by others, to the benefit of all. Since 2012, when XMaS became a mid-range facility, the beamline has hosted over 800 independent user visits from 90 identifiable UK user groups. Of these, around 55% of user visits are from either students (40%) or post-doctoral researchers (15%). New users account for around 40% of the total user visits each year. XMaS also facilitates international collaborations with approximately 25% of visits being researchers coming from outside the UK.

2. XMaS's expanding scientific portfolio

XMaS has a long history of engaging with recurrent users as well as encouraging new users and this allows us to continually expand our scientific remit. The efforts to extend the operational energy range into the tender regime (2.1–5 keV) opened up new user communities (particularly within chemistry and catalysis) interested in spectroscopic and scattering experiments at the S, Cl and P K edges [9], as well as potential opportunities for resonant X-ray diffraction studies at the Ru and Pd L edges as well as the Ir M edges [10]. Being open to such developments is at the heart of the operational ethos of the beamline. We continue to expand our capabilities through development of new metrologies and techniques including simultaneous measurements of several data channels at ever-increasing rates (currently several 100 kHz) [11,12] and extending the polarization control using phase retarders operating as a quarter-wave plate [13]. A newly designed (GI)-SAXS and reflectometry (XRR) system has been commissioned and provides enhanced and more efficient operations together with simultaneous WAXS measurements [14]. We have also recently used the white beam capabilities to open up new experimental paradigms [15]. As expected the early work on the beamline concentrated on condensed matter and magnetic systems exploiting resonant scattering [16], with studies on magnetic ordering in the rare earths [17] as well as the spin and charge structure in complex single crystals [18] being relatively common. The experimental conditions were well suited for the study of magnetic [19,20] and soft polymeric [21,22] thin films in grazing incidence [23]. X-ray fluorescence and energy dispersive diffraction were used to characterize breast tissue [24]. In this period, a suite of new environmental cells were developed including an electrochemical cell allowing electrical potentials to be applied to surfaces leading to developments in surface diffraction which exploited the capabilities of the diffractometer [25] and initiated studies of metal/electrolyte interfaces [26].

These early studies were consolidated in the following operational cycles with research activities distributed amongst the condensed matter, surfaces and interfaces, soft matter, healthcare and latterly energy and catalysis themes. Between 2002 and 2012, developments in sample environments were coupled with new detectors enabling the facility to expand its operational capabilities. The beamline's polarization analysis and access to low temperatures ensured that studies on the magnetic structures in single crystals [27–29] and thin films [30] continued, supported by advances in the metrology of resonant scattering [31] and absorption [32]. The high resolution in both conventional and grazing incidence scattering enabled structural studies of thin films to be continued [33] and the new two-dimensional detectors kick-started SAXS experiments from a range of materials including studies of the nano-roughening of nanoparticle colloidal arrays [34], the ordering of hairy-rod polyfluorenes [35], liquid crystals [36,37] and the crystalline orientation of enamel in a tooth crown [38]. Temporally resolved experiments in environmentally controllable cells enabled self-assembly of nanofilms [39,40] and the crystallization and segregation dynamics in solar cells [41] to be studied. New activities on preventing corrosion of heritage artefacts commenced using combinations of WAXS and spectroscopy [42]. Surface electrochemical studies expanded into several new areas; measurements of the liquid side of the interface [43], incorporation of temperature control into the design of the electrochemical cells [44] and new measurements of surface resonant X-ray scattering to obtain spectroscopic information on atoms at the electrochemical interface selected by the diffraction conditions [45].

In the last operational period, the research themes crystallized into three broad thematic areas; materials for energy, functional materials as well as healthcare and therapeutics.

(a) Materials for energy

Catalysis, chemical reaction dynamics, energy storage, fuel cell technologies, materials for energy applications, nuclear fission and decommissioning and photonic materials. Research in this thematic area covers important topics including photovoltaics, battery materials and catalysis. Experiments have used grazing incidence X-ray diffraction to investigate the effect of nanoparticles on the crystallization and surface morphology of commercially important polymer blends [46] as well as how surfaces evolve under ionic exchange [47]. A large growth area has been in the studies of photovoltaics including exploration of material growth and synthesis [48–50]. In partnership with a new-user community (based mostly in chemistry and including partners in the Catalysis Hub at Harwell) we have developed *in situ* fluorescence spectroscopy (XAFS) capabilities [51] using the low X-ray energies and newly developed sample environments to study catalysts and ionic liquids [52,53].

(b) Functional materials

Polymer materials, magnetism and magnetic materials, electronic structure, spintronics, superconductivity, surface science and supramolecular chemistry. The research in this theme is associated with the physical sciences and new functional materials which underpin a plethora of emerging technologies, ranging from mobile phones to computer displays. Studies of these important materials will continue to be carried out using combinations of XAS/WAXS/XRR and diffraction [54] in sample environments optimized for *in-operando* conditions (temperature, humidity, applied fields, etc.) [55]. Increasingly, experiments use multiple data channels to monitor the functional properties simultaneously with the X-ray data. Thus, exploring the structure and physical properties of materials remains a key research area in which the high magnetic and electric fields coupled with our low temperature environments can be exploited to study multiferroics [56,57] and polar materials more generally [58,59]. Fundamental condensed matter physics will always be a strong research area with studies of the Dzyaloshinskii–Moriya interaction in single crystals and thin films coming to the fore [60–62]. Experiments exploring the magnetic order in PrPtAl provided the first clear experimental observation of

'order by disorder' [63] and magnetostriction in MAX phases [64] was observed. Users have also investigated charge density waves in the high-temperature superconductor YBCO [65] and magnetic proximity in thin films [66]. We have a growing user community focused on understanding the properties of soft matter systems [67] including polymers and carbon nanotubes [68,69]. Protecting the environment and the preservation of heritage artefacts remains an important concern for both environmental safe-guards [70] and archaeological preservation [71]. Corrosion is a major source of failure in materials and experiments continue exploring corrosion on an atomic scale using diffraction and spectroscopy.

(c) Healthcare technologies

Biomaterials, tissue engineering and biophysics. The beamline continues to explore dental and bone materials [72,73] where recent experiments have used a model system to mimic how nanoparticles enter cells and their subsequent impact on human anatomy [74]. Studies have explored how Cu ions act as a contraceptive in an intrauterine device (IUD) [75]. There have also been studies of photonic materials that could be used in the next generation of medical imaging detectors [76].

At the time of writing, there have been a total of 400 papers published based on XMaS research activities, with a paper now resulting from every approximately 1.5 experiments. As well as academic output, information for the public is shared through our social media platforms (Twitter: @XMaSBeam and @XMaSSchoolTrip as well as a YouTube channel). In addition, XMaS played a significant role within the EURAMET Nanostrain Project (2013–2016): Novel electronic devices based on the control of strain at the nanoscale [11]. This collaboration brought together EU metrology laboratories along with industrial partners to explore new strain-mediated transistor devices to replace current CMOS technology. The success of the Nanostrain Project resulted in the follow-up EMPIR JRP programme ADVENT: Metrology for advanced energy-saving technology in next-generation electronics applications [77].

In addition to our academic output, XMaS has a dynamic and growing outreach programme. In April 2015, a new outreach activity was initiated to tackle the gender bias in Physics. An annual competition had been run from which we selected 14–16 winners and took them on a life changing trip to visit the European Photon and Neutron (EPN) campus and tour the ESRF and XMaS [78]. The aim remains to encourage young women to consider a career in science, by showing them the career opportunities available to them as well as introducing them to inspirational role models, in an international setting. The students also participate in the excellent ESRF Synchrotron@School programme which gives students the chance to manage their own research project (under suitable guidance), develop communication skills and address their aspirations. The trip itself is supported by additional widening participation events held at both the Universities of Liverpool and Warwick in partnership with our STEM collaborators.

3. XMaS as the UK X-ray materials science facility at the ESRF

A big change will occur during 2019/2020, consequent on the ESRF's upgrade of its magnet structure to a multibend achromat lattice with ultimate brilliance: the extremely brilliant source (EBS) project. The new ESRF lattice increases the number of bending magnets from 2 to 7 between each straight section, allowing stronger focusing and a reduction in the horizontal emittance by roughly a factor of 30. Our current X-ray source will be replaced by a new smaller and brighter 'short bend' magnet with a 0.86 T field and critical energy of 20.5 keV. The source moves upstream by some 2.76 m meaning that the diffractometer (extensively reconditioned and with a refurbished heavy load-bearing detector arm) has had to be moved downstream by an equal amount to preserve the current focusing and minimize aberrations: this in turn has necessitated the extension of the experimental hutch by several metres. The hutch extension also allows additional beam defining and conditioning elements to be incorporated, increasing our operational efficiency and allowing rapid switching between experimental configurations with a concomitant increase in

Figure 3. (a) A comparison of the flux at the sample through a $100 \times 100 \mu\text{m}^2$ aperture for the current and new source assuming realistic parameters for the mirror aberrations and absorption of beamline components. The dashed line corresponds to the current source parameters and the solid line represents the new source parameters. (b) Ray tracing calculation of the beam profile at the focused position. (Online version in colour.)

user throughput. Low wavevector resolution in SAXS experiments will also greatly benefit from the hutch extension as the sample to detector distance can be approximately 4 m. Unfortunately, the source point also results in a beam-path horizontally translated by a few 100 mm at the sample position, necessitating a lateral shift of the entire beamline.

The higher magnetic field of the ‘short-bend’ dipole increases the available flux at high energies by several orders of magnitude. To fully exploit the extended capabilities of the source itself, it will be necessary to upgrade the current optics. First, the current double crystal monochromator is being upgraded to liquid nitrogen cooling to cope with the higher heat load and equipped with newly designed high side load actuators for better operations across a wide range of monochromator angles and more precise fixed exit height control. A new mirror system comprising two interchangeable, meridionally bendable, cylindrical mirrors (coated with chromium and platinum, respectively) will be installed. This double mirror system allows us to increase the working cut-off energy up to 33 keV while having the adequate sagittal and meridional radii to focus the new source onto the sample position (figure 3). The two coatings allow a mirror to be selected avoiding any undesirable absorption edges across the operational energy range. For comparison, the flux through a $100 \times 100 \mu\text{m}^2$ aperture for the current and new system is shown in figure 3a. Ray tracing calculations assuming realistic mirror aberrations show that the new spot size will be significantly smaller in both the horizontal (H) and vertical (V) dimensions ($30 \times 80 \mu\text{m}^2$ [H×V]) than the current focused ($300 \times 600 \mu\text{m}^2$) spot size (figure 3b). As most experiments require a small angular divergence and beam size, the more brilliant source increases usable flux for most users by at least an order of magnitude. Further increases in the signal-to-noise ratio are derived from the reduced background and increased stability of the source.

To further exploit the enhanced capabilities, we will be making efficiency improvements through installing in vacuum beam positioning diagnostics allowing beam defining components (phase retarder, mirrors, slits, etc.) to be configured rapidly, minimizing downtime and ensuring that the beam interacts with the same sample volume across the entire energy range. Upgraded counting chains with new energy dispersive and the latest two-dimensional detectors with the capability to measure at high energies will be incorporated. A new gas handling system for sample environments and ion chambers will also be commissioned.

For the user community, the newly upgraded beamline will enable higher sample throughput, open up studies of weakly scattering systems as well as emergent phenomena more generally. The high energy and brightness will allow new insights into quantum critical behaviour as well as facilitating studies of confinement and proximity in magnetic and superconducting materials.

Advances in simultaneous studies allow the direct correlation of functional properties with structure. Newly combined (GI)-SAXS/WAXS and XRR metrologies will enable structure to be measured across a wide range of length scales simultaneously. The higher brightness enables more systems to be studied *in-operando* and on relevant time-scales across a range of materials including batteries and photovoltaics. The well-defined small beam will facilitate experiments on small samples or on localized regions of larger samples. Structural studies will be more spatially resolved, enabling individual domains and their temporal evolution to be studied under external stimuli. Such activities will provide new insights into phase transitions and the emergence of new properties. The new mirrors will transform studies of new catalysts and materials for green chemistry applications more generally by allowing the same sample volume to be probed across the entire operational energy range and within the same sample environment. Reactions can thus be followed on a site-by-site basis and in real time. The higher energies also facilitate experiments on buried interfaces or in more complex sample environments. Finally, the new lattice will greatly facilitate polarization studies due to the better-defined beam position and more efficient phase retarders.

As the beamline continues to support users into the future, it exists in an evolving materials research landscape that will require continuing development of strategic partnerships. These include the wider EPN campus, including the ESRF and ILL, as well as DIAMOND and the Advanced Photon Source. The latter two facilities undergo upgrades over the next decade. Exciting opportunities for closer collaboration with national and international research institutes and hubs including the Faraday Institute and the Catalysis Hub, which together with DIAMOND and ISIS are located on the Harwell Science and Innovation campus, as well as the Sir Henry Royce Institute will be explored over the next operational period. We are actively looking to engage with Centres for Doctoral Training (CDTs) and welcome opportunities to provide training courses for the next generation of material scientists.

4. Conclusion

An essential deliverable of the XMaS project over the years has been to support UK researchers and provide additional capability through state-of-the-art infrastructure for advanced functional material discovery. XMaS, along with the ESRF and DIAMOND, continues to provide the unique capabilities needed for investigations on the atomic-scale across a range of material systems from the academic and industrial sectors. XMaS has delivered significant scientific impact throughout the course of its operational life. We look forward, with confidence, to the next operational cycle where we can fully exploit the ESRF's new lattice and our new source. Doubling the operational energy range provides new opportunities to tackle the latest scientific challenges and retains the UK's route into the European research landscape by providing additional capability at the EPN.

Data accessibility. Further information and up-to-date operational statistics are available through our website: www.xmas.ac.uk.

Authors' contributions. T.H., M.C. and C.L. are directors of the XMaS facility and O.B., L.B., S.B., P.T. and D.W. are current beamline staff supporting users. All contributed to the writing of this paper.

Competing interests. We declare we have no competing interests.

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