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| **Institution:** | **University of Warwick** |
| **Unit of Assessment:** | **UoA B9 Physics** |
| **Title of case study:** | **XMaS: development of innovative X-ray instrumentation for synchrotron radiation facilities** |

## 1. Summary of the impact

XMaS is a dedicated materials science beamline at the European Synchrotron Radiation Facility (ESRF). It develops and disseminates novel instrumentation and sample environments that allow new experiments which support emerging technologies. By commercialising the intellectual property through licenses to companies economic impact is derived directly. Further economic impact is facilitated through knowledge transfer by trained people and expertise in new processes, which enhances the capability, capacity and efficiency of other central facilities. Public interest and awareness are engendered through individual research projects being highlighted in the media and through people's skills and experience being utilised in a broad range of sectors.

## 2. Underpinning research

In the late 1980s there was a strong community interested in actinide magnetism. This, coupled with the recently discovered phenomena of x-ray resonant magnetic scattering (XRMS), high-lighted a need for an x-ray synchrotron beamline facility to investigate the magnetic structure and associated phenomena in single crystals. To maximise its impact we identified that the higher flux and overall beam characteristics of a third generation synchrotron radiation (SR) source were required*;* so the beamline was built at the European Synchrotron Radiation Facility (ESRF).

EPSRC funds [6] were awarded to Prof. M.J. Cooper (University of Warwick) & Prof. W.G. Stirling (University of Keele, and later Liverpool) to design (1991-94), build and commission (1994-97) a flexible beamline that exploited an operational energy window between 3 and 20 keV covering the actinide resonances. The beamline was fully commissioned in 1997 and consisted of a non-magnetic six circle diffractometer with a standard suite of sample environments and detectors that were suitable to exploit the high flux and beam polarisation needed to study magnetic materials. The design of the optical components (mirrors and monochromator), as well as any air-paths, was optimised for low energy XRMS operations to facilitate studies on actinides [1]. The polarisation of the incident beam is a critical parameter for the study of ferromagnetic materials, and it was evaluated during the early build phase. These measurements showed that the design and beamline characteristics were suitable for studies of a wide range of magnetic materials [2]. On completion of the commissioning period, EPSRC awarded further funds for operations which have been concurrent from 1997 [6].

Due to the simple, robust and flexible design of the original beamline, the user community expanded rapidly to include new scientific areas and challenges beyond actinide magnetism. These experiments used the beamline in different ways and highlighted areas that could be optimised and made more efficient through better control of the sample position and slit defined beam paths. Off-the shelf components could not be found to resolve these issues and the team therefore researched and designed new infrastructure. Examples include the development of "tube slits" for reducing background noise and the XYZ cryostat mount for highly accurate and efficient sample positioning [3] during "beam-on" conditions. New sample environments such as magnetic fields (1 T electromagnet and 4 T superconducting magnet), a wet chemical cell and the ability to apply electric fields at low temperatures (1 K) were also designed to fit onto the diffractometer. These developments were completing during Phase II of the project (2002-2007).

Whilst the original beamline was designed for lower than usual energies (3 keV instead of >5 keV), Phase III (2007-12) investigated how to further lower the operational energy to 2.3 keV. This would both enable new magnetic materials to be studied and extend our reach to new user communities working on soft/biological and catalytic materials. This involved changes to the instrument hardware, a redesign of the monochromator, and designing new evacuated sample environments. For the first time, attenuators compatible with UHV operation had to be developed to overcome absorption problems [4]. New methods for controlling the incoming polarisation of the beam, back compatible with previous developments, were also developed [5].

Although originally envisaged as a probe of actinide magnetism, the continual developments throughout the XMaS project now mean that it is a facility for the study of a broad range of material science. This collaborative project is on-going, having been refunded recently as a Mid-Range Facility by EPSRC [6] to allow continuation of research on new capabilities and projects.

Warwick Researchers associated with XMaS development:
*Project Directors*, Professor M Cooper (from 1991), Dr T Hase (from 2007);
*Research Staff*, Bowyer (1992-95), Laundy (1992-1995), Brown (1993- ), Thompson (1993- ), Paul (1995-2005), Bouchenoire (2001- ), Newby (2007-09), Bikondoa (2009- ).

## 3. References to the research


Publications: (Principal Warwick authors in bold)

1. **D.F. Paul, M.J. Cooper** and W.G. Stirling, *Design of an x-ray beamline on a bending magnet at the ESRF for magnetic and high resolution diffraction*, Rev. Sci. Instrum. **66**, 1741 (1995) DOI: 10.1063/1.1145836
2. **D. Laundy,** S. Brown, **M.J. Cooper**, **D.F. Paul**, D. Bowyer, **P. Thompson** and W.G. Stirling *Magnetism in nickel and Synchrotron Beam Polarization studied by X-ray diffraction*, J. Synchrotron Rad. 5, 1235-9, (1998). DOI: 10.1107/S0909049597019420
3. S.D. Brown, **L. Bouchenoire**, D. Bowyer, J. Kervin, D. Laundy, M.J. Longfield, D. Mannix, **D.F. Paul**, A. Stunault, P. Thompson, **M.J. Cooper** and W.G. Stirling, *The XMaS beamline at ESRF: instrumental developments and high resolution diffraction studies,* J. Synch. Rad. 8 1172 (2001) DOI: 10.1107/S0909049501015394
4. P.B.J. Thompson, S.D. Brown, L. Bouchenoire, **D.F. Paul**, D. Mannix, P.S. Normile, **O. Bikondoa**, **T.P.A. Hase**, J. Kervin , C.A. Lucas and **M.J. Cooper**, *Optimizing the XMaS beamline for Low Energy Operations to maximize benefits from the ESRF Upgrade Program*, AIP Conf. Proc. 1234, 407 (2010). DOI: 10.1063/1.3463226
5. L. Bouchenoire, **R.J.H. Morris** and **T.P.A. Hase**, *A silicon <111> phase retarder for producing circularly polarized x-rays in the 2.1-3 keV energy range*, Appl. Phys. Lett. 101, 064107, (2012). DOI: 10.1063/1.4740080

Grants/awards:

6. Total EPSRC commitment - £20 M
   Phase I (build) GR/G02338/01, GR/J79263/01, (Jan 1991 to Nov 1997)
   Phase I (operation) GR/L39698/01, GR/M21706/01, GR/R14989/01 (Sept 1997 to Sept 2002)
   Phase II (operation) GR/R87420/01, (Sept 2002 to Sept 2007)
   Phase III (operation) EP/F000375/1 and EP/F000766/1, (Oct 2007 to Sept 2012)
   Phase IV – £6.2 M Mid-Range Facility Grant (Sept 2012 to Sept. 2017)


## 4. Details of the impact

The XMaS project provides opportunities that underpin the UK material science community through novel research infrastructure. It delivers economic and commercial impacts, stimulates public discourse on science, transfers knowledge to a broad range of society, facilitates new experimental techniques, and provides training to UK and international stake holders.

A significant impact of the XMaS project has been through the commercialisation of intellectual property, generated as a result of developments made in the specific research infrastructure to implement new experimental advances, to produce products of generic applicability to synchrotron-based infrastructures. To date, 9 commercial licences have been generated (shared between Liverpool and Warwick). We hold agreements with the *National Physics Laboratory* (NPL) and *American Magnetics*, and we have formed a close partnership with *Huber Diffraktionstechnik GmbH & Co* [7], who are world leaders in supplying specialist x-ray equipment with an annual turnover of ca. €8 M. In the REF period, sales of licensed XMaS instruments to customers in the US, Europe, and the Far East have produced a turn-over of €750 k for *Huber*. They write:

> "We have benefitted enormously through the XMaS collaboration through which we have been able to add to our product range and ensure that we stay at the forefront of the worldwide market in x-ray instrumentation. This benefit is not just from the additional new Huber instruments that we have developed in partnership with the XMaS team but also through the increased market share this partnership brings to Huber…. With the XMaS products, we were able to sell complete integrated systems… to meet the future needs of our customers." [7]

An article in the EPSRC newsletter, *Connect*, describes the impact of the XMaS instrumentation as 'big business' highlighting that "research can have impact through innovative instrumentation as well as science" [8]. By adopting the XMaS-developed research infrastructure, SR facilities world-wide can enhance their capabilities, extend their reach and run experiments more efficiently.

> "If these XMaS designed slits were not available commercially we would have had to develop them ourselves" [9].

As an example, our XYZ-cryostat mount [10] enables the sample to be repositioned remotely, aligning its position on a micron scale. At the US Advanced Photon Source

> "use of the XYZ cryostat mounts saves ~1 day of beamtime per experiment … which equates to $9072/day ... directly saving the APS facility $1.5 M per annum" [11].

Since 2008, eleven XYZ-cryostat mounts have been sold globally. Including the 7 sold previously, and used throughout the REF impact period, we can extrapolate a total annual saving of ~ $4 M to the sector.

Since the XMaS project has been running, several new central facilities have been built or up-graded, including DIAMOND, APS and the LNLS in Brazil. Through knowledge transfer and direct interaction of staff between these facilities, we have enabled projects to be completed more efficiently and cost effectively. This was typified by the deliberate sequential appointment of a post-doctoral researcher (Gemma Newby) to work on XMaS for two years before transferring to DIAMOND in 2009.

> "This saved us a year of development time [and] cost saving equivalent to ~£40 k " [9].

Another example of exchange of processes is the installation of an electric-field capability, developed by XMaS with NPL, at DIAMOND [12]. More recently, in 2013, the Swiss Light Source and the APS have implemented our methods for controlling the incident polarisation, which has

> "pioneered the use of thin etched silicon…the fast switching piezo stage…rapid acquisition of high quality data" [11].

Commercial organisations making use of our facilities include the (US) National Standard Laboratories of NIST, the NPL, the Swiss national laboratory EMPA, as well as St. Bartholomew's Hospital, Mary Rose Trust, Rijksmuseum, Amsterdam and the Academy of Fine Arts, Vienna.

XMaS has also developed sample environments that allow materials to be studied under a range of atmospheric (gaseous or liquid) conditions. These metrologies are well suited for cultural heritage and anthropological studies where

> "XMaS is making a significant contribution to preservation of the heritage of the EU…extending the lifetime and our understanding of beautiful artworks and artefacts" [13].

Two leading museums (Rijksmuseum, Amsterdam; Academy of Fine Arts, Vienna) use the unique capabilities of XMaS to provide "deeper insights" and develop new strategies to prevent atmos-pheric corrosion, and thus preserve their collections for future generations [14]. Other examples, which have also entered the public domain, include corrosion studies of Tudor cannon balls from the wreck of the Mary Rose, highlighted in *The Sunday Telegraph* and on-line [15]; as well as how teeth enamel can be used to trace migratory patterns of early humans [16]. These types of study:

> "ensure that the huge contribution heritage tourism makes to the GDP of EU countries will continue" [13].

As a major facility we communicate, disseminate and transfer knowledge and ideas to a wide community which includes both industry and the public: we circulate an annual newsletter world-wide, hold open user meetings, and present at events such as the *Technology World* conference, organised by the DTI and attended by over 1,000 global companies. In conjunction with commercial partners such as NPL and DIAMOND, we co-host, sponsor and run specialist meetings and workshops for both academic and industrial researchers. One outcome of these meetings was a new web resource detailing metrology standards for electrical measurement hosted by NPL [17]. As a result of this collaboration XMaS and NPL became partners in a major EU project that brings together the premier EU metrology labs with commercial partners such as IBM [12]. Furthermore, some 3,000 members of the public visit the ESRF annually (50% students, 50% general public or industrial visitors). The on-site XMaS staff conduct tours and highlight the importance and relevance of science conducted in this large-scale European facility.

The XMaS project is committed to train the next generation of material scientists with specialist expertise in advanced synchrotron radiation techniques. Since 2008, more than 50 post-doctoral researchers have been trained. 23 have since drawn on their research in positions at central facilities around the world, including 10 who have taken up roles at the home of synchrotron science in the UK (DIAMOND). As noted by the Institute of Science and Technology in Art director

> "only institutions such as XMaS enable young researchers to be trained in synchrotron techniques by experienced scientists … extend their own scientific network…, and to be one brick in the wall of Europe's scientific ability to compete with the US and Asia" [14].

The diverse range of people trained on XMaS - undergraduate and postgraduate students, post-doctoral researchers, international visitors - have been able to transferred their knowledge and experience to their current roles These span industry, other central facilities (as mentioned above) and education in schools. We highlight four examples: Stuart Bland is an editor at *Materials Today*, which is a monthly open-access journal presenting material science research in an accessible way; Alex Pym works at Kromek, an SME developing digital colour x-ray imaging in County Durham; Rachael Houchin is a physics teacher at the Royal Grammar School, Newcastle who brings her XMaS experience into the classroom, especially an ability to develop engaging experimental demonstrations; Mark Gallagher is a project scientist at IDT, which is an advanced engineering company specialising in beamline components:

> "My training on XMaS gave me invaluable insight as a synchrotron user to the importance of precision, reliability and stability of experimental instrumentation to the end goal of a synchrotron experiment. It is difficult to envisage a more appropriate experience for my present position and has added great value to my role within the company" [18].

**5. Sources to corroborate the impact**


7. Letter from Managing Director, Huber Diffraktionstechnik GmbH & Co. KG;
8. EPSRC Connect 77, p5 (2010) "Innovative Instrumentation for Synchrotron Source" http://issuu.com/epsrc/docs/connect77/1
9. Letter from Principal Beamline Scientist, DIAMOND Light Source.
10. XYZ-Cryostat Mount offered in Huber product catalogue http://tinyurl.com/jwqjyel
11. Letter from Associate Division Director, Advanced Photon Source, Argonne National Laboratory, USA.
12. Letter from Science Area Leader and Principal Research Scientist at the UK's National Physical Laboratory (NPL)
13. Letter from Head of Conservation & Scientific Research, Rijksmuseum, Amsterdam.
14. Letter from Chair of Institute for Natural Sciences and Technology for the Arts, Academy of Fine Arts Vienna.
15. Mary Rose cannon ball article in *The Sunday Telegraph***Error! Hyperlink reference not valid.** http://tinyurl.com/bls2zyu
16. http://www.wired.co.uk/news/archive/2012-12/19/xmas-teeth-studies and also in *Laboratory News*, Dec. 2012 http://tinyurl.com/matkpbf
17. http://interactive.npl.co.uk/multiferroics/index.php/Main_Page
18. Email from Project Scientist at IDT